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Conference topics

Beam and plasma sources
Fundamentals of modification processes
Modification of material properties
Coatings deposition
Nanoscience and nanotechnology

INVESTIGATION OF A HIGH VOLTAGE AC PLASMA TORCH OPERATING ON MIXTURES OF METHANE AND OTHER GASES

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Plasma technologies are actively investigated for a wide range of processing substances: plasma gasification of solid fuels [1], reforming of natural gas [2], decomposition of toxic substances [3], and the production of oxide systems. In addition, plasma pyrolysis of organic substances in an inert atmosphere is studied for a long time. In this case, soot, hydrogen and a small amount of acetylene are formed. There are a number of studies devoted to the plasma pyrolysis of methane under the action of a DC arc [4] and an AC arc [5]. In all these cases, a low voltage was applied, which led to a rapid erosion of the electrodes (due to high electric current). Before, high-voltage plasma torches working on oxidizing media (low-voltage with rail electrodes [6], high-voltage with hollow electrodes [7], multigas [8] plasma torches) were investigated. The erosion of the electrodes of such plasma torches is comparable to the erosion of electrodes of DC plasma torches (at the same currents), which leads to a decrease in the erosion products.

A new three-phase AC plasma torch operating on methane and other gases is being investigated. A protective gas (nitrogen, argon, CO2) is supplied to the near-electrode zone, and methane is supplied to the arc zone. At the same time, the arc ignition is facilitated, and the electrode insulators are prevented from coming into contact with the electrically conductive soot. The power source of the plasma torch consists of a high-voltage transformer, current-limiting inductors, reactive power compensators and a system for measuring electrical parameters. During operation of the plasma torch, oscillograms of current and voltage are recorded. The power of the plasma torch and the voltage drop on the arc significantly depend on the total flow rates of plasma-forming gases, as well as on the mass fraction of methane. Increasing the proportion of methane in the plasma-forming mixture significantly increases the power, because hydrogen is formed, which has a large heat capacity and thermal conductivity. The composition and properties of the produced soot were investigated by scanning electron microscopy, IR-spectroscopy and X-ray phase analysis.

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RESEARCH OF IGDANTINE DESTRUCTION UNDER HIGH-CURRENT BEAM OF ELECTRONS WITH ENERGY MORE THAN 1 MW [1]

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Research of the composite materials destruction under extreme pulsed loads is an important task, both from material science applications and development of a fundamental understanding of the formation and propagation of shock waves in materials with a complex internal structure. The problem in this field is concerned with the absence of universal theories and mathematical models that describe such interactions This is especially true of organic materials, in which chemical transformations occur during the action of irradiation. In this paper, we present an experimental study of the interaction of a high-current electron beam with a low-modulus polymer material characterized by high elasticity. As an object of study, samples of igdantine, consisting of gelatin, glycerol and formaldehyde, are used, which is actively used in studying the properties of thin-layered composites [1].

The experiments were carried out on a powerful high-current electron accelerator RS-20, providing a current in the diode gap of up to 100 kA at a peak voltage of up to 1.5 MeV and a total pulse duration of not more than 500 ns. At electron energies of more than 1 MeV, the average range of electrons can exceed 5 mm. In experiments, the through-burning of the sample was found out. On the other hand, the range of the electrons in such sample is almost four times less than its thickness. With pulsed irradiation, volumetric energy release takes place, which has a significant effect on the formation of shock waves [2]. It is also worth noting that, due to the low conductivity of the sample material, some electrons are "stuck" in its thickness and create a very substantial space charge. The accumulation of such volumetric charges leads to electrical breakdowns in the thickness of the sample, traces of which due to the high transparency of the material are also detected. It was also demonstrated that this effect causes the formation in the peripheral regions of the sample of multiple cracks (length ~ 10 mm). Note that when creating a pressure comparable to that achieved in the focal spot of the beam, by means of electric explosion of the foil, the formation of macrocracks was not observed. The features of the destruction of a highly elastic polymer material are discussed in comparison with the results obtained by the action of a powerful electron beam on polymeric materials with a higher pyrolysis temperature (polystyrene, epoxy compositions).

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DEVELOPMENT OF PLASMA DISCHARGE IN SALINE SOLUTION [1]

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There is an interest in pulsed discharges in electrolytes, including in water-salt solutions recently. Biomedical applications, as well as the development of devices for the sterilization of water and liquid aerosols [1] stimulate further research. Attention is paid to applications related to the formation of shock waves in high-current discharges [2, 3], in particular, in relation to the problems of hydro acoustics in seawater [4].

Since electrolytes have a high conductivity in comparison with distilled water, a high-density current flows through the electrolyte near the active electrode at a relatively low voltage. Therefore, the gas cavities can occur already at gap voltage of tens of volts.

When the voltage reaches the critical value, a gas-discharge plasma occurs in some micro cavities near the active electrode. In this case, we can talk about the incomplete breakdown of the gap.

If the initial voltage on the electrodes significantly exceeds the critical value, then in process of development of the discharge gas cavity and a gas discharge plasma closes the gap completely. In this case, we can talk about a complete breakdown of the gap. The discharge passes into a high-current form of combustion. The plasma column completely determines the resistance of the gap.

In this paper, we study the stages of discharge formation in saline solution in cases of both incomplete and complete breakdown.

Studies were carried out in the configuration pin-plane of the electrode gap. Voltage pulses of micro and millisecond duration, amplitude less than 5 kW and maximum current up to several kA were applied to the gap.

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TO THE QUESTION OF INSTABILITY IN THE HALL THRUSTER (ION BEAM SOURCE)

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The balance of electric $\overline{F}_E = e\overline{E}_x$ and magnetic components $\overline{F}_B = e\left[\overline{v}_{ex} \times \overline{B}_z\right]$ of the Lorentz force has long been used in a *vacuum* filter of ion beam velocities, called the Wine filter [1]. A similar balance of forces occurs in the Hall plasma thruster [2]. Here two streams of unlike charged particles carry a single discharge current $\overline{j}_d = \overline{j}_e + \overline{j}_i$. Changing the distribution of the intensity of the E_x field along the axis of the channel x (due to the potential difference U at the ends of the channel and the geometry of the B_z field in it), it is possible to minimize the j_{ex}-projection of the electron current in the zone of crossed E_x and B_z fields [3].

If, at the entrance of an electron into the $E_x \times B_z$ zone, the modules of the Lorentz force correspond to the inequality

$$F_B \geq F_E, \tag{2}$$

then the electron passes from the longitudinal motion along the channel x to the azimuthally drift perpendicular to both fields. Taking $F_B = F_E$, we obtain the equality

$$v_{ex} = \frac{E_x}{B_z}, \tag{3}$$

which becomes a condition for changing the longitudinal velocity v_{ex} of the electron to the drift velocity. The value of (3) is none other than the drift velocity of the electron u_θ, when the Hall parameter $\beta_e = \omega_e \tau_e \geq 40$ (it is established experimentally) and the thruster works steadily.

If the longitudinal velocity of the electrons at the entrance to the engine is greater than the drift velocity

$$v_{ex} > u_\theta = \frac{E_x}{B_z}, \tag{4}$$

then the regime is stable. If

$$v_{ex} < u_\theta = \frac{E_x}{B_z}, \tag{5}$$

then the regime is unstable.

The larger the E_x or the greater the voltage of the thruster U, the greater the v_{ex}, and the thruster works steadily. To increase E_x and U, it is necessary to increase the plasma resistance in the channel, which is achieved by increasing B_z. Therefore, according to inequalities (4) and (5), the high-voltage DAS works more steadily than the comparatively low-voltage SPD. (DAS and SPD — the names of the two types of Hall thruster adopted in Russia).

Unlike the velocity filter in Hall thruster, there is an inhomogeneity of the E_x field. Due to this, a dispersion of the deflection of the electron flux j_{ex} arises in the Hall thruster in the transition to a stable operating mode. As a result of the dispersion, oscillations of the discharge current occur, preventing the thruster from operating at the low voltage.

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EXTENDED ION-PLASMA DEPOSITION SYSTEM [1]

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The paper presents the results of the development of an extended ion-plasma deposition system for the production of thin-film coatings. The principle of operation of this system is based on depositing the target material with gas ions extracted from the plasma of a non-self-sustaining arc discharge [1] with a heated and hollow cathode. The parameters of the plasma generated by the deposition system and the characteristics of the resulting coatings were investigated.

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MODERN ION SOURCES FOR SEMICONDUCTOR IMPLANTATION

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Modern Ion Sources for Semiconductor Implantation including flat panel display are reviewed. Development of a Freeman and Bernars-Whit prolonger lifetime ion sources are discussed [1]. Development of a small anode ion sources reviewed [2]. Microwave ion sources for Ion implantation are reviewed [3]. Negative ion source for high energy tandem implanter is discussed [4]. Ion source for large and very large ribbon ion beam system for flat panel display implantation is described [5] A space charge compensation of ion beam and instability damping is discussed [6,7].

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DISCHARGE WITH A SELF-HEATED HOLLOW CATHODE AND A VAPORIZABLE ANODE IN AN INHOMOGENEOUS MAGNETIC FIELD[1]

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Interest in the use of the method of reactive evaporation for obtaining non-conducting thin films of metal oxides is explained by the possibility of achieving high deposition rates that cannot be obtained by ion sputtering. The formation of an oxide layer on the surface of the sputtered target reduces the rate of sputtering, whereas the accumulation of surface charge and layer breakdowns are the cause of arc formation, which degrades the quality of applied coatings.

In the present work, a discharge with a coaxial self-heated hollow cathode and a water-cooled hollow anode-crucible, which is placed in the magnetic field of a short solenoid, is used to deposit the Al_2O_3 coatings. The distance between the electrodes was 30 cm. The compression of the discharge column in an inhomogeneous magnetic field provides an increase in the power density at the anode and the evaporation rate of the metal from the crucible, and also increases the degree of ionization of the metallic vapor. The supply of oxygen into the near-anode region of the discharge increases the content of ionized and excited oxygen particles in the plasma. As a result, a high density of the ion current from the plasma to the surface of the growing coating is achieved, which ensures the crystallization of high-temperature oxide phases at lower temperatures.

The aim of the research was to study the conditions of stable burning of a discharge with a self-heated hollow cathode and a vaporizable anode in an inhomogeneous magnetic field, to diagnose the plasma discharge parameters, to measure the spatial distributions of the fluxes of charged and neutral particles in the volume, and to determine the parameters of the ion flux, at which the low temperature formation of a nanocrystalline alumina films at a rate of ~ 4 μm/h on a surface of several hundred cm^2 is ensured.

It is demonstrated that the creation of inhomogeneous magnetic field with an induction on the axis up to 20 mT in the anode part of the discharge at currents above 30 A provides high power density at the remotely located anode (up to 120 W/cm^2) and the density of the plasma (10^{12} cm^{-3}), generated in the anode region. The heat output from the anode, measured by the calorimetric method, is about 50% of the total discharge power, which ensures high rates of evaporation of aluminum from the water-cooled crucible in a discharge with a current of ~ 40 A and a burning voltage of ~ 60 V.

The discharge instability resulting from the occurrence of ion-acoustic oscillations of the plasma potential is suppressed upon transition to the mode of a discharge operation with a vaporizable anode in an inhomogeneous magnetic field with currents above 30 A and minimum values of argon flow through the cavity of 10 cm^3/min. In the mode of the metal vaporization, with a total power released at the anode of ~ 1 kW and a power density more than 100 W/cm^2, the size of the liquid bath is several mm, the evaporation rate is constant, splashes and outbursts of liquid metal are absent, and the discharge is stable.

On a stainless steel sample the rate of Al_2O_3 thin films deposition was up to 4 μm/h for a discharge current of 40 A, which is much higher than the rates presently achieved by reactive magnetron sputtering (~0,1 -1 μm/h) .

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POWER SUPPLY FOR LOW-TEMPERATURE PLASMA JET (DESIGN AND RESULTS OF TESTING) [1]

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In our days, the atmospheric-pressure plasma jets is a subject of intensive research due to a variety of applications, such as air purification, chemical synthesis, plasma-assisted technology, and other [1]. The example of system for plasma jet obtaining is the so-called low current non-steady-state plasmatron.

The paper describes the results of the design and testing of the high-voltage DC power supply whit reactive ballast for low-temperature plasma jet applications. In the proposed system, plasma jets is generated by the non-steady-state plasmatron at average glow discharge currents less than 100 mA, and average power consumed by discharge about 100W.

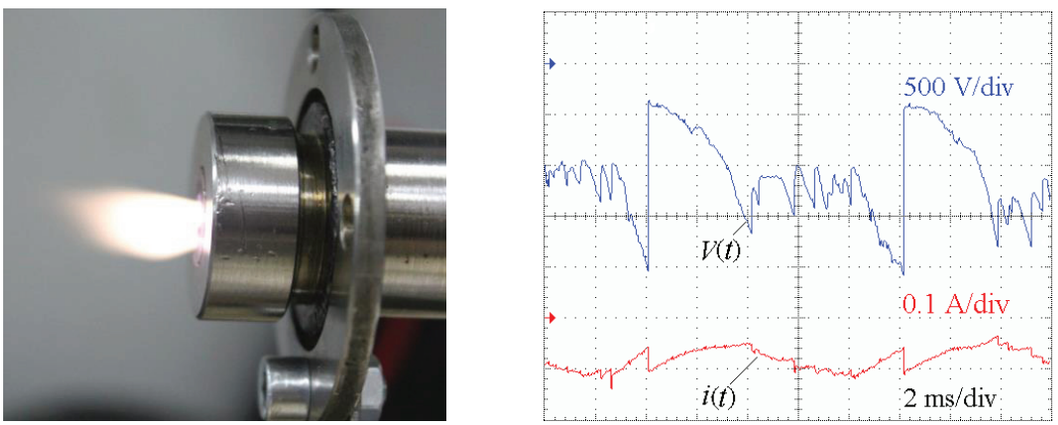

Fig. 1. Photography of plasma torch on the exit of non-steady-state plasmatron and discharge voltage and current waveforms. $V(t)$ – discharge burning voltage, $i(t)$ – discharge current. The gas flow rate G(air) = 0.2 g/s, diameter of the plasmatron nozzle is 5 mm.

One of the significant advantages of the inductive-resistive current limiting method is the system has the properties of short-circuit current limiting and maximum output power limiting without the use of special automation circuits. This is important when supplying a discharge witch is an essentially non-linear load [2]. Another important feature is the practically zero idling consumption power.

Due to a small number of elements, as well as the use of general-purpose components, the circuit proved to be simpler and cheaper than the previously high frequency inverter topology based circuits developed by us.

Testing of the power supply prototype shows that it provides a maximum output voltage up to 6 kV, maximum output power approximately 170 W and maximum discharge current of up to 120 mA. In the optimal operational mode, the power conversion efficiency can reach $\eta \geq 70\%$ at electrical power consumed by discharge of about 100 W. The ripple of the discharge current (Fig. 1) contributes to increasing the degree of non-stationarity of the powered discharge.

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SOME VACUUM-ARC-BASED PLASMA AND ION BEAM TOOLS FOR SURFACE MODIFICATION

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Vacuum arc discharges deliver a copious supply of dense metal plasma, and this kind of plasma formation mechanism has gained wide acceptance as a standard laboratory tool. Plasmas can be formed from virtually all of the solid metals of the Periodic Table as well as carbon, and by admitting a controlled flow of gas into the arc region, hybrid metal/gas plasmas can be formed also. Here we describe four different approaches to material surface modification using the vacuum arc discharge as the means for generation of metal (and carbon) plasma.

Because the metal plasma from a vacuum arc is created as a jet that streams away from the cathode, this plasma source itself, unadorned by any further elaboration, provides a "metal plasma gun" – a tool for generating energetically streaming (10–200 eV directed ion energy) metal plasma. This affords an excellent, and quite simply made, means for the energetic deposition of thin film structures. Considerable effort world-wide has been given to the utilization and exploration of this plasma deposition technique.

High energy beams of metal ions can be formed by embodying the vacuum arc plasma source within an ion source (i.e., an ion beam generator) configuration. Then the metal plasma provides the "feedstock" for the ion beam. The ion beam current can be very high, and currents of up to 20 A have been reported. At the same time, the extraction voltage employed in these kinds of ion sources can be up to around 100 kV, and since the vacuum arc ions are multiply stripped with charge states typically in the range 1+ to 5+, the ion energy of the extracted ion beam is greater than the extraction voltage by the same factor. Ion beam energy can be up to several hundred keV. As a tool for surface modification, the vacuum arc ion source has been used widely for high energy, high dose, metal ion implantation.

Plasma immersion ion implantation (piii or pi^3) is an alternative method of implantation in which the energetic ions are provided by acceleration across the high voltage sheath established around the biased target which is immersed within the plasma. The target is repetitively pulse-biased to high negative voltage. During the pulse-on period energetic implantation occurs, and during the pulse-off period the plasma recovers. This technique can be used with a metal plasma provided from a vacuum arc. In this case, during the pulse-off period there is some deposition of neutral metal plasma on the target, and so the process is a hybrid of metal ion implantation and metal plasma deposition. The method has been called "plasma immersion ion implantation and deposition", or "mepiiid", and has been used for a range of surface modification applications.

In conventional ion implantation, the ions are extracted from a plasma that is held at high positive potential, and the ion energy is determined by the potential drop through which the ions fall in the beam formation electrode system. An alternative approach has been demonstrated in which the plasma and its electronics are held at ground potential and the ion beam is formed and injected energetically into a space maintained at high negative potential. This configuration has been called an “inverted ion source”, and precisely because the plasma source and electronics are at ground potential, it allows substantial savings both technologically and economically, rendering feasible some ion beam implantation applications that might otherwise not be possible for researchers and laboratories of more limited means.

Here we describe these four specific ways of utilizing the vacuum arc plasma for the surface modification of materials – plasma deposition of thin films, metal ion implantation using a vacuum arc ion source, plasma immersion ion implantation and deposition, and implantation using the "inverted ion source" concept. The methods and their hardware are outlined, and examples of their applications presented.

PULSED HIGH-INTENSITY SILICON ION BEAMS FORMATION[1]

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The results of experimental studies on the high-intensity axially symmetric low-energy silicon ion beams formation are presented. The method of ion beam formation is based on plasma-immersion extraction and ion acceleration with their subsequent ballistic focusing in the equipotential drift space [1]. For the generation of silicon plasma, a pulsed vacuum-arc discharge with a current pulse duration of about 250 μs and an amplitude of 100 A was used. To increase conductivity, single-crystal silicon was preliminarily subjected to neutron transmutation doping at the TPU IRT-T research reactor. Extraction and ion acceleration were carried out by a grid electrode in the form of a part of a sphere with a radius of 75 mm. Pulsed bias potential of negative polarity with an amplitude in the range of 0.5–2.0 kV and a pulse duration in the range of 200 μs was applied to the grid electrode immersed into plasma. The influence of amplitude and duration of bias potential pulses on the parameters of the formed ion beams was investigated. It was shown that the efficiency of transportation and focusing of high-intensity pulsed silicon ion beams depends, to a large extent, on the geometric dimensions and structure of the grid focusing electrode, the amplitude-frequency characteristics of the bias generator and the conditions for neutralizing the beam charge in the drift space with its ballistic focusing. Silicon ion beams with a maximum ion current density of the order of 0.4 A/cm^2 at the ion current amplitude of the order of 0.5 A were obtained for the first time.

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ATMOSPHERIC PRESSURE DISCHARGE PLASMA SOURCE FOR BIOCOMPATIBLE POLYMERS TREATMENT [1]

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The parameters of the plasma source of the atmospheric pressure discharge for the modification of biocompatible polymers with a low threshold of resistance to temperature are investigated. The optimal operating conditions were as follows: argon flow rate of – about 1 l/min; discharge voltage magnitude – about 300 V; discharge current magnitude – about 40 mA; pulse duration – 1 - 5 μs; pulse repetition rate – 100 kHz; electron temperature – about 0.3 eV; plasma density – about $5 \cdot 10^{11}$ cm^{-3}.

In these conditions the optical emission spectra of discharge plasma were investigated. The intensity of the lines of excited argon atoms is three orders of magnitude greater than the intensity of the lines corresponding to the second positive nitrogen group. Lines of excited atoms of copper - the material of the electrodes of the discharge system, were not observed.

The dependencies of the temperature vs the flow rate of the argon jet passed through the discharge was also investigated. The temperature range was determined (40-50^{0}C) at which the surface treatment of polymer samples was carried out without their thermal destruction.

The samples of polymeric materials: polylactide, polyvinyl alcohol, composite materials based on polylactide and hydroxyapatite, as well as a lactide-glycolide copolymer were treated with the plasma of atmospheric pressure gas discharge. Dependences of the surface resistance of experimental samples on plasma exposure regimes show that an increase in the total processing time, as well as the average discharge power, leads to a clear decrease in the surface resistance for samples based on a lactide-glycolide copolymer and hydroxyapatite. Also, an increase of discharge average power leads to an increase in the hydrophilicity of polymer surfaces after their direct contact with the plasma of this discharge (fig. 1).

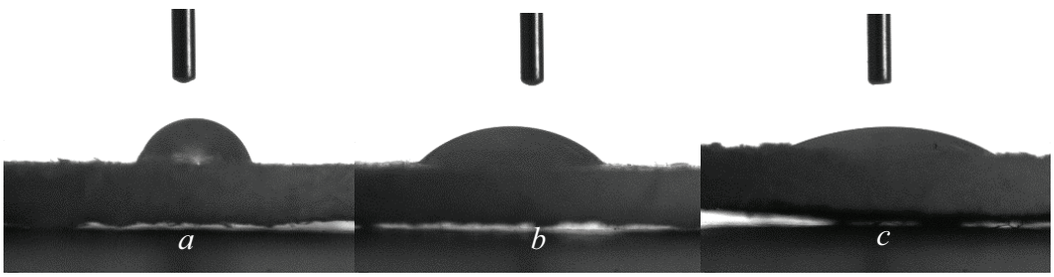

Fig. 1. Photos of water droplets on the surface of polylactide: *a* – original sample and sample treated with heated argon; *b* – after plasma treatment with mean power 1 W; *c* – after plasma treatment with mean power 5 W.

Special attention should be paid to the result showing that the effect of only one argon jet heated to the same temperature as in the discharge, but without the discharge, does not change the surface resistance and wetting conditions of the polymers. Evidently, the modification of polymeric materials occurs as a result of the complex effect of a gas-discharge plasma, involving collisions with their surfaces of heated neutral molecules, charged particles, optical radiation, and the temperature effect contribution is not predominant.

[1] This work was supported by Russian Foundation for Basic Research and Tomsk region Government under Grant No 16-48-700654.

POWERFUL AC ELECTRIC ARC PLASMA TORCHES - ADVANCED DIRECTIONS OF IMPLEMENTATION

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Thermal plasma on the one hand is an instrument for the synthesis or transformation of substances, and on the other, it allows the destruction of complex molecules and compounds, what is useful in plasma processing methods [1-3]. IEE RAS research team has created powerful high-voltage AC electric arc plasma torches [4], with help of which virtually all tasks on waste processing can be solved, including the release of useful products, the destruction of toxic waste, the increase in the depth of processing of heavy oil fractions and associated petroleum gas, as well as efficient gasification of solid fossil fuels. The talk presents AC electric arc plasma torches operating on air up to 500 kW with thermal efficiency up to 93%. Electric arc plasma torches operating on mixtures of plasma-forming media such as steam, carbon dioxide, nitrogen, argon, gaseous and evaporated hydrocarbons (including chlorine and fluorine containing), with a power of 80 to 160 kW with a thermal efficiency of not less than 94% are also presented.

This work was supported by Programs of Fundamental Research of the Presidium of the Russian Academy of Sciences No. 31.

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EMISSION OF THE ELECTRON BEAM FROM A SINGLE CHANNEL OF THE FOREVACUUM PLASMA ELECTRON SOURCE [1]

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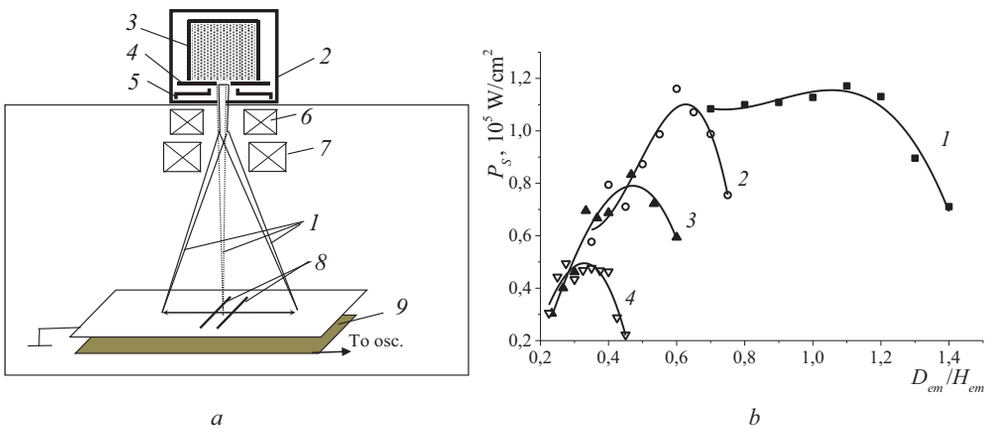
Interest in using electron-beam sources based on electron emission from a hollow cathode plasma for such applications as welding, soldering, cutting, etc., is directly due to their ability to efficiently transfer energy to the local area and a long operating time. One of the variety of the class of plasma sources are the forevacuum plasma sources of electrons [1], operating in the pressure range from one to hundreds of pascals. Due to the characteristic properties of these sources during operation at such pressures, these sources are capable to efficiently treatment of high-temperature dielectric materials and can be used for cutting ceramics and glass [2,3], selective sintering of ceramic powders [4], etc. The improvement in the quality of precision processing by such sources and the widening of the field of their application require an increase in the previously achieved level of the beam power density.

In the studies carried out, an increase in the specific parameters of the electron beam was achieved by optimizing the geometry of the emission channel having a cylindrical shape. The experiments were carried out according to the scheme shown in Fig. 1.a. The obtained dependences of the electron beam power density in the crossover on the ratio of the diameter of emission channel to its length are shown in Fig. 1.b.



a *b*

Fig. 1. Scheme of the experiment (*a)* and dependences of power density P_S of the focused electron beam on the ratio of diameter D_{em} of emission channel to its length H_{em}. *a*: 1 - 1 – electron beam; 2 – forevacuum plasma electron sources; 3 – cathode; 4 – anode; 5 – extractor; 6 – magnetic lens; 7 – magnetic deflection coil; 8 – measuring gap; 9 – current collector plate. b: 1 – H_{em} = 1 mm, 2 – H_{em} = 2 mm, 3 – H_{em} = 3 mm, 4 – H_{em} = 4 mm

These dependences indicate an increase in the beam power density as the ratio of the channel diameter to its length increases. However, exceeding a certain value of the ratio of the channel sizes leads to a sharp decrease in the power density. As the length of the emission channel decreases, the maximum value of the power density increases.

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GENERATION OF THE LOW-ENERGY LARGE-RADIUS QUASI-CONTINUOUS ELECTRON BEAM BY THE FOREVACUUM PLASMA-CATHODE SOURCE BASED ON THE CATHODIC ARC[1]

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The paper describes research of generation of the low-energy (up to 10 keV) large-radius quasi-continuous electron beam by plasma-cathode source based on the cathodic arc in the forevacuum pressure range. The forevacuum electron sources provide direct processing of various dielectric materials due to beam plasma neutralization of the negative charge on the dielectric surfaces. To implement the quasi-continuous mode of electron beam generation, an arc discharge with pulse duration of up to 10 ms has been used to generate emission plasma. The current-voltage characteristics of the plasma-cathode source generating the quasi-continuous electron beam in the forevacuum pressure range (3–15 Pa) are presented. The current-voltage characteristics of the quasi-continuous electron beam source have two distinct regions: sharp growth and saturation, which is typical for electron sources with plasma-cathode. The low-energy electron beam with pulse duration of up to 10 ms at an emission current of up to 15 A and pulse energy up to 1500 J is obtained. The presented cross-sectional energy density distribution of electron beam indicate the need for the use of further steps to form uniform distribution for the surface processing of materials.

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REGISTRATION OF OVER-ACCELERATED ELECTRONS IN A HIGH-CURRENT PICOSECOND ACCELERATOR

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A high-voltage (U = 280 keV) high-current (I = 5 kA) electron accelerator with a beam duration of 200 ps was investigated. The luminescent method showed that the beam diameter in the uniform pinching mode is about a micrometer, therewith the beam contains electrons with an energy exceeding 400 keV. Analysis of the bremsstrahlung X-ray spectrum of the beam electrons showed that in this mode a significant part of the spectrum lies in the energy range> 300 keV, with the energy of quanta in the "tail" of the spectrum exceeding 500 keV.

OPTICAL SPECTRA OF PLASMA OF A PULSED NON-SELF-SUSTAINED HOLLOW CATHODE GLOW DISCHARGE [1]

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The non-self-sustained glow discharge with the hollow cathode allows to reach a plasma density of up to 10^{18} m^{-3} with a high homogeneity of plasma density[1].

It was shown in [2] that after the discharge voltage pulse ends, the plasma relaxation time in a non-self-sustained glow discharge at a low pressure (≈ 1 Pa) has a finite time, which can be tens to hundreds of microseconds. The decaying plasma contains a large number of ionized and excited states. Selecting such a pause time between pulses, at which the plasma does not decay, it is possible to change the density of certain plasma states and use this feature in practice.

In this paper we investigated the emission spectra of a plasma of a non-self-sustained low-pressure glow discharge in a stationary and pulsed combustion regimes. The purpose was to determine the decay time of the plasma, depending on the amplitude of the current in the pulse and the operating pressure.

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INVESTIGATION OF ELECTRIC-ARC PLASMOTRONS FOR MATERIAL PROCESSING

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The design schemes of plasmatrons for material processing include both the jet and melting apparatuses. If it is a question of applying powder coatings, the plasma jet structure in the plasmatron for spraying should ensure stable temperature and velocity distributions for repeatability of the technological process.

If it is necessary to implement intense heating of the metal surface, then the arc discharge is required. The two-jet plasmatron, effective for processing of powder materials, also belongs to the melting type.

The energy, thermal and resource characteristics of arc plasma generators with the power of 10-50 kW, used in many technological processes, including thermal processing of materials: plasma spraying, processing of powder materials, and metal surface treatment, are presented in this study.

Today it is determined that application of powder materials of various technological purposes can be made with the help of the arc plasmatron with an interelectrode insert. It provides stable parameters of power, pulsation components, and location of the near-electrode section of arc at the anode. The use of reducing and neutral gaseous media provides the required enthalpy of the plasma flow for processing and spraying the powders onto hard surfaces. The advantages of the proposed plasmatron are compared with the previously used ones.

Peculiarity of the design scheme of the two-jet plasmatron is that a significant part of the arc column is located outside the electric-discharge chamber and its effect on the processed material is very effective. When working on argon, nitrogen, and hydrogen, the electrode assemblies are made of thermionic metal with fixed electrode spots. If the plasma-forming gas is air or steam, the copper tubular electrodes are used.

The design schemes of plasmatrons, current-voltage characteristics of the arc and methods of their calculation are presented.

THE POTENTIAL DISTRIBUTION IN TWO-ELECTRODE GAS-FILLED GAP IN WEAK AND STRONG ELECTRIC FIELD

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To obtain current carriers in gas environment, sources of charged particles, such as electrons or ions, of different constructions, are often used. In some cases, it is necessary to know how the electric field intensity in the interval between the electrodes, where the current carriers are introduced, depends from performance of the source.

The presence of a charged particle source can be simulated by introducing of some emitter surface. Therefore, to solve the task of the distribution of the electric field, it is necessary to solve the Poisson equation for the two – electrode gap, in which one of the electrodes is an emitter, and the other is a collector of charged particles.

Equations for calculation of electric field intensity in an arbitrary cross-section of a plane-parallel interelectrode gap (including on surfaces of electrodes) taking into account a volume charge are received.

For "weak" electric field:

$$\frac{dU}{dx} = \left(\beta_{01}^2 + \frac{9}{4}\frac{x}{L}\frac{j}{j_0}\right)^{1/2}\frac{U_a}{L}.$$

For “strong” electric field:

$$\frac{dU}{dx} = \left[\beta_{02}^{3/2} + \left(\frac{5}{3}\right)^{3/2}\frac{x}{L}\frac{j}{j_0}\right]^{2/3}\frac{U_a}{L},$$

where U - is the potential of space, x - is the coordinate, L - is the distance between electrodes, U_a - is the applied voltage, j - is the current density, j_0 - is the current density that would flow between electrodes at unlimited emission, β_{01}, β_{02} - are functions depending from the kind and pressure of the gas and from the volume charge.

For weak electric fields the magnitude β_{01} we find from the equation:

$$\left(\beta_0^2 + \frac{9}{4}\frac{j}{j_0}\right)^{3/2} - \beta_0^3 = \frac{27}{8}\frac{j}{j_0}.$$

For strong electric fields the magnitude β_{02} we find from the equation:

$$\left(\frac{5\sqrt{5}}{3\sqrt{3}}\frac{j}{j_0} + \beta_0^{3/2}\right)^{5/3} - \beta_0^3 = \frac{25\sqrt{5}}{9\sqrt{3}}\frac{j}{j_0}.$$

Numerical data of β_0 (j/j_0) for strong and weak fields can be found from presented curves in figure 1:

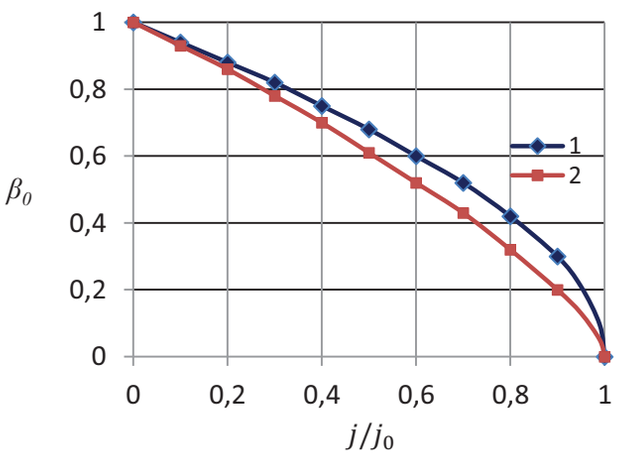

Fig. 1. For determine of the electric field intensity on the surface of emitter: curve 1 – for a weak field, curve 2-for a strong field.

The obtained expressions, unlike the known ones, allow to calculate without data on the emission capacity of the source and the value of the thermal speed of the current carriers.

INCREASE OF THE UNIFORMITY OF THE PLASMA DENSITY DISTRIBUTION IN A NON-SELF-SUSTAINED GLOW DISCHARGE BY CHANGE OF THE SHAPE OF A MESH EMISSION ELECTRODE [1]

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At present time the generation of a homogeneous volumetric plasma is an actual scientific and technical task. A series of studies on the method of plasma generation in a non-self-sustained glow discharge with a hollow cathode at a low pressure (0.4 to 2) Pa and a discharge voltage of 50 to 300 V were implemented, in which additional electrons are injected from the plasma emitter [1]. When using a mesh electrode, it is possible to disperse the beam of the injected electrons and change the distribution of the plasma density in the hollow cathode [2]. The purpose of this work was to determine the influence of the shape of the mesh emission electrode on the azimuthal and radial distribution of the plasma density in a non-self-sustained low-pressure glow discharge with a hollow cathode when an injected electron current is up to several tens of amperes.

To determine the influence of the shape of the mesh emission electrode on the distribution of the plasma concentration, six configurations were used, shown in fig. 1. For these forms, the radial and azimuthal distributions of the plasma density were measured by a single cylindrical probe and a flat probe with a guard ring, respectively.

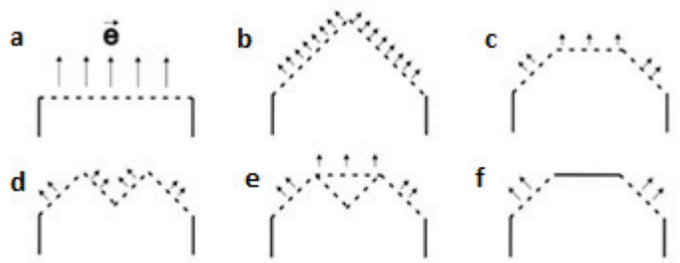

Figure 1 − Forms of mesh emission electrode.

Based on the measurement results, the inhomogeneity coefficients of the azimuthal and radial distributions of the ion current density arriving at the probes which are equal to the ratio of the maximum deviation of the ion current density from the average value of the ion current to this average value. The table shows the obtained values of the inhomogeneity coefficients for six forms of the grid emission electrode.

The electrode configuration (according to Figure 1)	a	b	c	d	e	f
Current recall. discharge IA	32	34	39	32	35	43
The coefficient of inhomogeneity of the azimuthal distribution k, %	32	43	55	46	49	48
The inhomogeneity coefficient of the radial distribution k, %	40	26	24	24	44	24

According to the set of parameters, the optimal form of the mesh electrode is the shape of the emission electrode in the form of a truncated cone with a concave central part.

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ENERGY SPECTRUM OF THE ELECTRON BEAM OUTPUTTED INTO THE ATMOSPHERE USING AN ELECTRON ACCELERATOR WITH A MESH PLASMA CATHODE[1]

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Using the advantage of most electron sources with mesh (layer) stabilization of the emission (cathode) plasma boundary associated with the possibility of a slightly dependent change in the parameters of the generated electron beam from each other (electron energy, beam current amplitude, duration and pulse repetition rate) in an electron accelerator with such a wide-aperture (750 × 150 mm) cathode it is demonstrated the principal possibility of predicting the energy spectrum of an electron beam outputted into the atmosphere through the thin metal foil. The energy spectrums obtained at different accelerating voltages (100 ÷ 160 kV) were reconstructed on the basis of the experimentally obtained attenuation curves of an electron beam as it passed through thin metal foils by the Tikhonov regularization method for solving an ill-posed problem for the Fredholm integral equation with minimal a priori assumptions. It is shown that under the experimental conditions the form of the energy spectrum slightly depends on the working pressure of the gas in the accelerating gap of the accelerator, and the expansion of the spectrum is primarily due to the scattering of the beam in the output foil and air. It is shown that a controlled change in the accelerating voltage during a current pulse of a beam outputted into the atmosphere leads to a predictable expansion of its energy spectrum and displacement of its maximum in the region of lower energies, which, first of all, can be useful for specific applications of such accelerators, both for scientific and technological purposes.

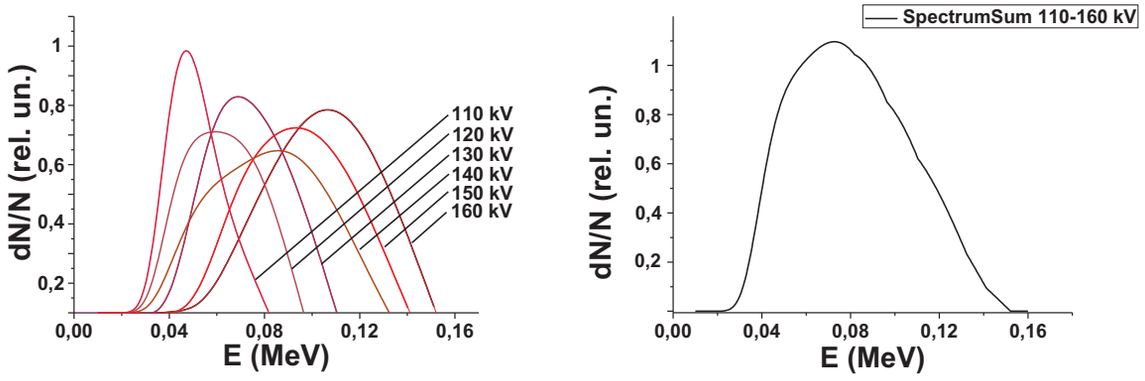

Fig. 1. Energy spectrums of an electron beam outputted into the atmosphere, taken at different accelerating voltages. Experimental conditions: AMg-2n foil with thickness of 30 μm, the distance from the output foil to the collector of the foil-filter is 15 mm.

Fig. 2. The total energy spectrum of the electron beam outputted into the atmosphere when the accelerating voltage varies from 160 kV to 110 kV for the duration of the beam current pulse.

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A HOMOGENEOUS EMISSION PLASMA GENERATION IN A DISCHARGE SYSTEM WITH AN EXTENDED HOLLOW CATHODE[1]

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For a discharge system with an extended hollow cathode used in the forevacuum plasma source of a ribbon electron beam [1], the influence of the geometry of the cathode cavity and the type of plasma-forming gas on the homogeneity of the plasma density distribution in the region of the emission boundary is studied. It is shown that the geometric parameters of the discharge gap of the electron source exert the greatest influence on the emission plasma density distribution. With the optimal geometry of the discharge system, a range of discharge current, pressure, and gas type is determined, which ensures that the distribution of the plasma concentration is not more than 20% of the mean value.

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EXPERIMENTAL INVESTIGATION OF POWERFUL WIDE BAND RADIATION INTERACTION WITH THE CONDENSED MATTERS [1]

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There are many experimental and theoretical publications on powerful laser radiation interaction (changing target aggregate state) with matter. Generally, these are lasers of the visible and infrared range, where laser photon energy is less than the target atoms ionization potential. There are practically no works on ionizing (with photon energy there is higher than the target atoms ionization potential) laser radiation interaction with the condensed matters.

We present the results of powerful (i.e. ionizing) wide band (with photon energy from visible to VUV) radiation interaction with the condensed matters. We used a source with a power up to 10^7 W/cm^2 and processes duration $10^{-6} – 10^{-5}$ s.

Processes on thermonuclear plants walls and heat-shielding layers of aircraft ablation, processes in plasma accelerators and electrodynamic devices, in technological (photolithography, structural surface modification of construction materials) and photochemical processes are accompanied with interaction of ionizing wideband radiation with matter.

As a radiation source, it was used the magnetoplasma compressor (MPC) discharge in gas [1]. The gas-discharge camera was pumped out before each discharge up to the pressure ≤ 1 Pa and filled with gas (Ne, Xe, Ar, air) up to pressure $p=2*10^2 – 10^5$ Pa. Capacitor storage device (10 μF, 28 kV) was switched with MPC electrodes a controlled discharger; full energy deposition into the discharge was ~ 70% of the stored energy, of which 40% – is invested in the first half-period of the discharge current, current maximum ~ 150 κA and duration of half cycle ~ 5 μs.

As a targets we used rectangular samples 8 – 10 mm thick. Width of targets was 30 mm. Targets separately or some at once were placed parallel to MPC axis at distances of 30-40 mm. As targets plates from aluminum, titan, copper, PTFE and thin films of bismuth and aluminum were used. We have registered abnormally low gasdynamic evaporation mode threshold.

At schlieren and interferograph photos it is visible that the gasdynamic evaporation mode (the mode of the plasma piston) is implemented: there is a shock wave in gas, contact border between gas and vapors plasma. There is received thermodynamic parameters distribution on height in vapours over the target. The maximum temperature is reached not at contact border (from a radiation source), and in "plasma piston".

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FEATURES OF DEVELOPMENT OF A PULSE DISCHARGE IN A SOLINE SOLUTION AT DIFFERENT VOLTAGE POLARITY ON ACTIVE ELECTRODE [1]

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Currently, the discharges in electrolytes are widely used in medicine, biology, echolocation, etc. [1, 2]. The peculiarity of these discharges is that gas cavities occur near the active electrode at low voltages applied to the gap [3]. Further increasing the voltage leads to the ignition of the discharge in the cavities and subsequent spread of the plasma to the opposite electrode. At the final stage, the high-conducting channel closes the gap, i.e., a breakdown occurs.

In this paper, we consider the processes occurring in a three-percent NaCl solution in water in the geometry of the active electrode (pin) and the return electrode (plane). The pulse voltage applied to the gap is of micro and millisecond duration, while the amplitude of the voltage is 0.3–5 kV. The maximum currents in the electrolyte are at the level of a few kA. There is the set of data on the process of formation, development and degradation of gas cavities at different polarity of the applied voltage pulse. The authors analyze the physical mechanism of the occurrence of cavities. Such data as CCD camera photos, the gap luminescence, and spectral measurements in the process of gas-discharge plasma formation in cavities are presented. We discuss the shape of the discharges and the process of the breakdown development. It is shown that the occurrence of cavities and plasma ignition in them have a significant effect on the current passage.

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FORMATION OF SHOCK WAVES AT DIFFERENT STAGES OF DISCHARGE DEVELOPMENT IN SALINE SOLUTION [1]

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Currently, the discharges in electrolytes are widely used in medicine, biology, echolocation, etc. [1, 2]. The peculiarity of such discharges is that gas cavities occur near the active electrode already at low voltages applied to the gap [3]. Further increasing the voltage leads to the ignition of the discharges in the cavities and subsequent spreading of the plasma to the opposite electrode. Practically, here there is an incomplete breakdown. At the final stage, the high-conducting channel closes the gap, i.e. there is a completed breakdown.

The paper deals with the processes occurring in 3% NaCl solution in water in geometry active electrode (pin) – inverse electrode (plane). The duration of the pulse voltage applied to the interval lies in the micro and millisecond time interval, and the amplitude is 0.8–5 kV. At the same time, the maximum currents through the electrolyte are at the level of hundreds of amperes or units of kiloamperes. A set of experimental data on the process of formation, development and degradation of gas cavities is presented. The physical mechanism of the occurrence of cavities is analyzed. The data on the process of gas-discharge plasma formation (CCD camera photos, gap emission spectra) are presented. The forms of discharge combustion and the process of breakdown development are discussed. It is shown that the occurrence of cavities and plasma ignition in them has a significant effect on the current passage.

The technique of measuring shock waves in an electrolyte is described. The results of experiments on the study of the formation of shock waves at different stages of discharge development are presented. It is shown that shock waves of high intensity appear already at the stage of incomplete breakdown in the saline solution. There is a discussion of this effect.

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GAS-DISCHARGE STARTING OF HOLLOW CATHODE OPERATION IN SELF-HEATING MODE[1]

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The conditions of gas-discharge starting of all-metal (Mo) massive (75 g) hollow cathode with small (5 mm) cavity diameter and thick (5 mm) wall in electrode system with limited (<100 sccm) gas flow through the cathode cavity and distantly (35 cm) placed anode were determined. The difficulty of gas-discharge starting realization in such system is caused by the complexity of glow discharge ignition in cathode cavity of low diameter that is usually provided by using special systems of ignition [1], as well as by the necessity to minimize local arc attachments that arise during the process of cathode heating and may cause intensive starting erosion of the cathode.

Usage of gas-discharge starting of cathodes-compensators of plasma engines where impregnated emission insertions are used and the current does not exceed 5 A at cathode temperatures 1000ºC is known [2]. It was reported that gas-discharge starting of such cathodes takes 2-5 s. There was also information about development of the method of gas-discharge starting of massive all-metal cathodes of vacuum plasmatrons having practically any design and mass, diameter under 60 mm and operation current several kA during 40-100 s [3].

In the system under investigation, the operation currents reach 50 A while the cathode temperature may exceed 2000ºC, which fact complicates the starting as compared with [2] where the cathodes are accelerated up to operating parameters in arc mode. As compared with [3], the starting is impeded by small diameter d of the hollow cathode in which steady burning of glow discharge is possible only if thickness of cathode layer is $l<d/2$, that causes the necessity to rise the glow discharge current at the initial stage of heating till values at which cathode spots initiation is very likely.

The purpose of our study was to optimize the conditions of gap breakdown with distantly placed anode at small gas flow through the cavity, ignition of glow discharge in cathode cavity of small diameter and heating of the cathode till temperature providing the required thermionic emission of the cathode, in the glow discharge mode without any local cathode attachments arising.

The reduction of static breakdown voltage of electrode gap till values under 1 kV at argon flow 60 sccm was provided by usage of cylindrical hollow igniting electrode with apertures in face diaphragms that was installed on the outlet of cathode aperture. The development of discharge to the main anode occurred when voltage was supplied to the main gap from a power source providing direct current >0.4 A. Voltage pulses of 40 kHz frequency were fed to the gap in parallel. The source provided smooth growth of pulse voltage and discharge current during 5-10 s. The cathode was heated in pulse-periodic mode at regulated pulse amplitude 10-30 A. Selection of the mode with definite current density on the cathode at predetermined pulse duration provided not only heating but also training of the cathode that reduced the possibility of arcing.

The most lasting process is the starting of a new cathode, as the heterogeneity of emission properties of its surface caused by inclusions, surface films and microextrusions leads to increased tendency of the cathode to arcing. The process of initial starting of the cathode may last for several minutes. Cathode starting after cold contact with the atmosphere is easier and takes ~0.5-1.5 min. Cathode heating up to the mode with pulse current 25 A in standard conditions takes only 15-20 s.

Thus, using pulse-periodic mode of discharge burning to minimize the possibility of arising of local arc attachments allows to heat a hollow cathode at high average power ~ 1 kW with low start erosion of the cathode.

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A NOVEL ANODE-LAYER PLASMA THRUSTERS FOR MATERIALS MODIFICATION[1]

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We report a new plasma and ion sources development for materials modification. The plasma sources are adaptation of an ion thruster technology and are based on an Institute of Physics (NAS of Ukraine) toroidal plasma optic device. The plasma optic devices or (in other words) plasma lens is a well-explored tool for focusing high-current, large area, energetic, heavy ion beams. It provides a convenient, affordable and quick way of carrying out particularly high-dose ion implantation. The anode layer thruster was used in a wide-aperture plasma optical system for producing and transporting intense electron beams [1].

An important advantage of anode-layer thruster is a high probability of ionization, which makes it possible to create a plasma with a high concentration at relatively low flow rates of the working gas, and that makes the thruster attractive to use in high-efficiency plasma generators and ion sources. Other important advantage is the presence of permanent magnets that do not need power supply.

Figure 1 shows the cross sections of the two plasma thruster geometries (toroidal and radial), that were used in experimental studies.

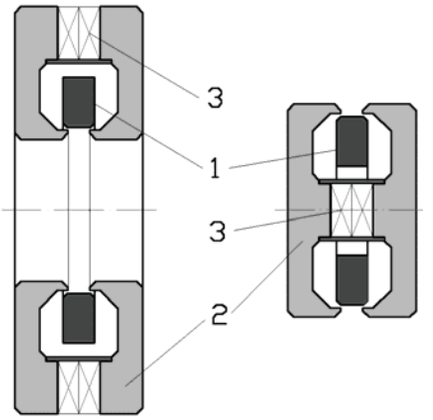

Fig. 1. The two geometries of anode-layer plasma thruster: left – toroidal geometry, right – 360°radial geometry.

It is known that plasma thruster provides three discharge modes: (1) a low-current mode with a collimated ion beam at relatively low pressures and currents of several tens of milliamperes (ion generation mode); (2) a high-current plasma mode at higher pressures and currents from several amperes (plasma generation mode); and (3) an arc mode with a cathode spot. The latter mode is an undesirable discharge mode, since a large droplet fraction is contained in the plasma. The first mode is used in ion sources, while the second mode can be used to generate a dense gas plasma. The toroidal ion source generates a collimated ion beam converging to the axis, and radial source forms an ion beam diverging in a 306°. Both ion sources can be used to coat outboard and inner surfaces of cylinders by simply moving the source.

The volt-ampere characteristics of the anode-layer thruster in different modes have been studied in detail as well as the plasma density, floating potential, and plasma electron temperature for plasma mode.

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GENERATION OF MONATOMIC, MOLECULAR AND TRIATOMIC HIDROGEN ISOTOPE ION BEAMS USING HOLLOW CATHODE DISCHARGE [1]

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A reflective discharge with a hollow cathode in crossed ExB fields with hydrogen and deuterium used as a working gases was studied in a wide range of operating parameters: the discharge current from 0.0002 to 40 A and the working pressure from $2 \cdot 10^{-5}$ to $5 \cdot 10^{-4}$ Torr. Their effect on the ion species fractions in the extracted beam, along with the discharge gap configuration effect, was examined using time-of-flight methodic. The effect of discharge gap geometry and magnetic field configuration, along with the effect of external discharge parameters, on the plasma ion composition were studied. It is shown that fractions of monatomic, diatomic and triatomic ions of hydrogen and deuterium strongly depend on these parameters, and maximal fractions of each of three components are reached at certain combination of the parameters. At the pulsed discharge current of 40 A and working pressure of $2{,}2 \cdot 10^{-5}$ Torr, the fraction of protons is 80 % in the discharge system with plasma volume of 6 cm^3. At the steady-state discharge current of 40 mA and working pressure of $4 \cdot 10^{-4}$ Torr, the fraction of H_3^+ ions is 90 % in the discharge system with plasma volume of 24 cm^3, and the ion beam current is 1.25 mA with the ion energy of 11 keV. The discharge parameters which provide maximal fractions of D^+, D_2^+ and D_3^+ are also determined.

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METHOD OF DIAGNOSTICS FOR THE LOW-TEMPERATURE PLASMA JET [1]

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Currently, the plasma jets/torches based on atmospheric-pressure discharges attract considerable attention. In most case, the systems for obtaining plasma jets include of specially configured electrodes allowing the gas to flow through the discharge plasma region. The well-known examples of such systems is a coaxial plasmatron, where, due to a gas flow, the discharge background weakly ionized gas forms the plasma jet inside the nozzle and plasma torch at the exit of plasmatron [1].

The goal of the paper is to estimate the parameters of the plasma jet generated by the low-current non-steady state plasmatron. In the experiments, the discharge in air powered from DC power supply that connected to the gap via a coaxial cable and ballast resistor R_b. In the above conditions, an average discharge current can be varied up to 0.1 A, and the discharge burns in a glow form with the occasional transitions from glow to spark [2].

The proposed electrodes configuration of improved system for plasma jet diagnostics contain two additional grids *4* and *5*, as shown on Fig. 1. When a positive potential V_4 is applied, a non-self-sustained currents of negative charged particles (i_4 and i_5) flowing through the plasma jet volume and diagnostic electrodes *4* and *5* are measured.

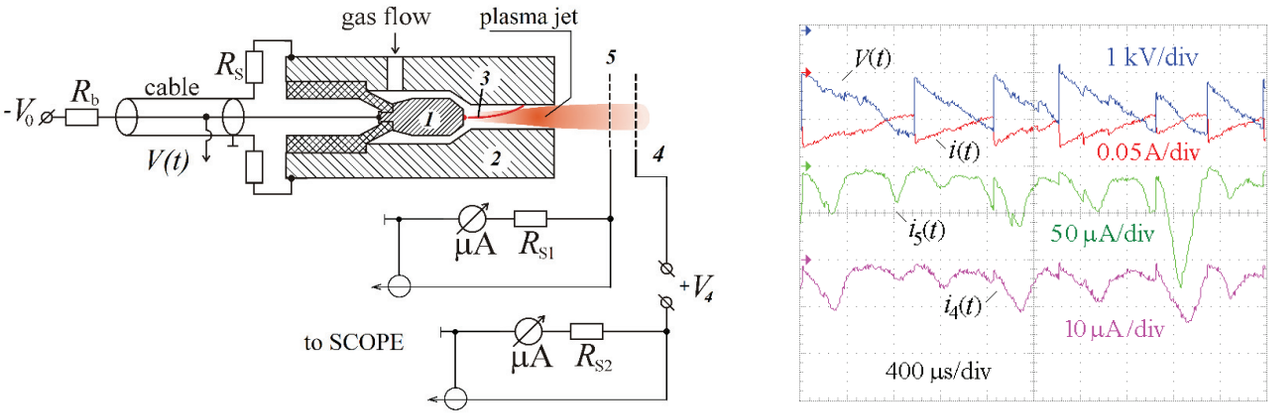

Fig. 1. Simplified electrical circuit of the non-steady state plasmatron with improved diagnostic system for the low-temperature plasma jet and typical voltage and current waveforms. *1* – cathode, *2* – anode/nozzle, *3* – discharge plasma region, *4* – measuring grid, *5* – grounded grid. The gas flow rate G(air) = 0.15 g/s, diameter and length of the plasmatron nozzle is 20 mm and 5 mm respectively. Voltage of DC power supplies: $V_0 \le 5$kV, $V_4 \le 3$kV. R_S – discharge current shunt, R_{S1} – shunt for measuring a current trough plasma jet volume in nozzle, R_{S2} – shunt for measuring a current trough the plasma torch volume, $R_b = 42$ kΩ, $V(t)$ – discharge burning voltage, $i(t)$ – discharge current, $i_4(t)$ – current trough plasma jet in plasmatron nozzle, $i_5(t)$ – current of trough the plasma torch volume.

The main idea of the proposed method is that, based on experimental data, it is possible to calculate the electric field strength, current density and the charged particles flux density in the volume of a plasma jet. The density of charged particles and ion drift velocity has been estimated using of suggestion for average negative ions mobility $\mu = 1$ cm^2/V·s and electrical field in diagnostic system $E_{45} = 2$ kV/cm. In this case, the charged particles density in plasma jet volume has been estimated a level of $n \approx 10^{10}$ cm^{-3}.

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DBD AS A TOOL FOR INITIATING AND STIMULATING OF CHEMICAL REACTIONS[1]

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To effectively use the dielectric barrier discharge (DBD) as a tool, it is necessary to understand the physics of the process, and also what chemical reactions are activated and stimulated by a DBD at various discharge parameters. For this purpose an electrophysical installation was created. It consists of a power supply system [1] and a plasma-chemical reactor (PCR) with a coaxial geometry of the electrodes (gap is 1.5 mm). At this installation a number of experiments were carried out, during which the natural gas was treated by DBD (93.8 % vol. – CH4, 3.7 % vol. – C2H6, 1.1 % vol. – C3H8 and other impurities in small quantities). During the experiments the air flow rate varied from 0.4 to 18 l/min. To the outer electrode were applied rectangular unipolar voltage pulses (15 kV, 60 us, 4 kHz). The gas composition at the PCR outlet was measured in two ways – using a gas chromatograph and using a quadrupole mass spectrometer.

As a result of experiments, the multichannel mode of the discharge was realized in the PCR. The average power consumption is 75 W, the energy deposited in the plasma in one pulse is 18.8 mJ.

Modeling of discharge processes and chemical transformations in a gas with using a 1-dimensional approximation was also carried out. In Fig. 1 shows the dependence of the concentration on the number of pulses. The dotted line corresponds to the experimental curve, dashed - the results of modeling. The results showed good agreement. Thus, it can be concluded that the most significant chemical processes that were initiated by the discharge were included in the model.

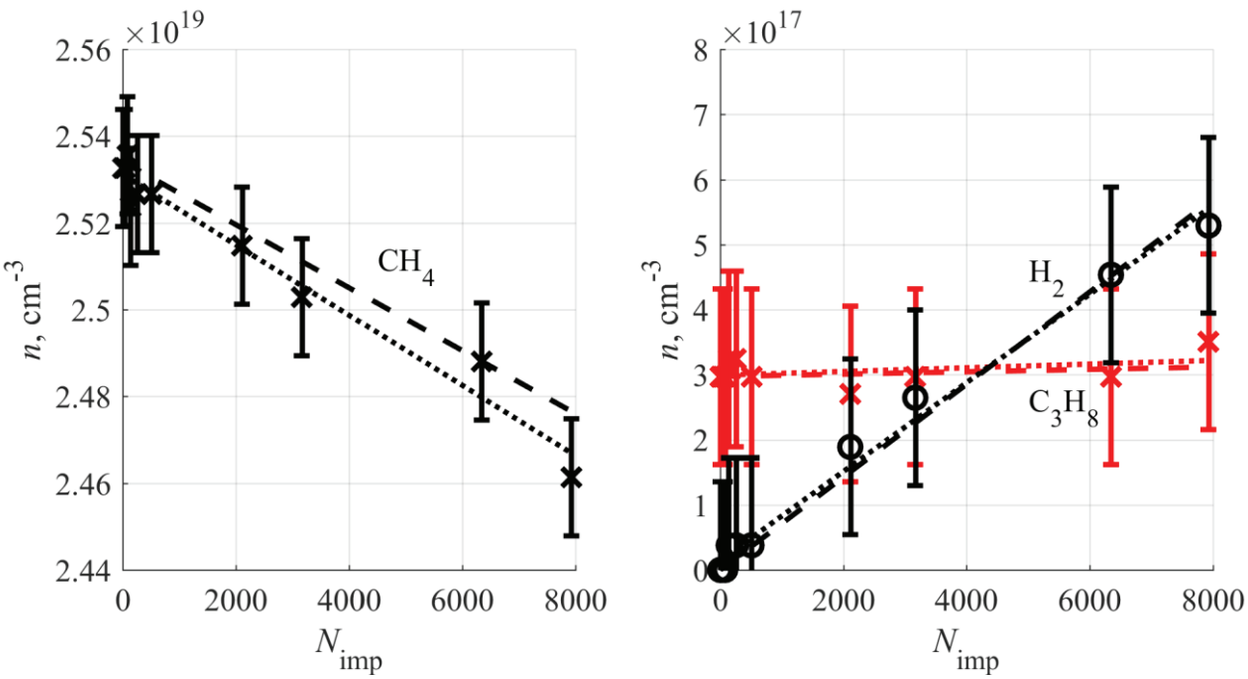

Fig. 1. Dependence of the concentrations of CH4, H2 and C3H8 on the number of applied discharge pulses

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INVESTIGATION OF MULTISTAGE ELECTRODE SYSTEMS FOR INCREASING ELECTROHYDRODYNAMIC FLOW VELOCITY[1]

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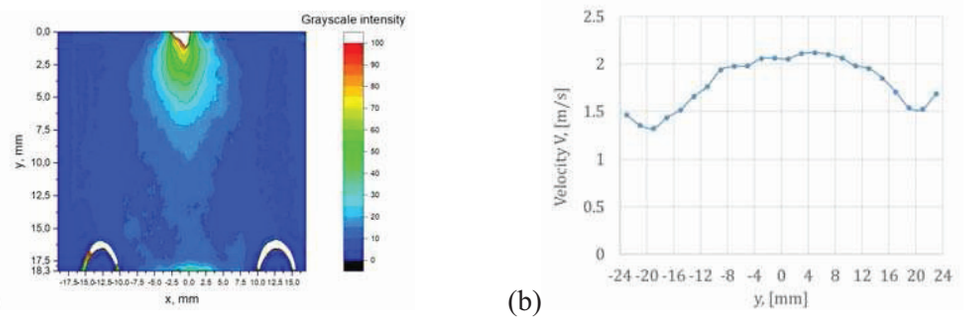
One of the possible applications EHD flow formation systems are ionic atmospheric engines [1]. Their advantage is the absence of noise, the need to have a fuel on board, high efficiency at high altitudes [2, 3]. One of the effective types of such systems is based on the geometry of an asymmetric capacitor (Pic 1a). A typical scheme of this device consists of two conductive parts. The first one is presented in the form of a thin wire or point, called the emitter or the corona electrode. The second (collector) is made in the form of a wire mesh, tube or foil skirt. High voltage is applied to the corona electrode, which results in ionization and electric field energy transfer to the ions, which leads to the appearance of an EHD flow. In this paper, we consider the effect of geometric parameters and design features of the electrode systems on thrust characteristics and efficiency in atmospheric pressure air, with the goal of creating an optimal electrode and accelerating sections design. Thrust parameter is represented by magnitude of a generated thrust, and the efficiency is evaluated by magnitude of thrust-to-power ratio.



(a) (b)

Fig. 1. Photo of gas glow of the electrode system in a mixture of air with argon (a) and the profile of the air flow at the outlet (b)

During the study, several systems were considered, including nichrome and cooper wires with diameters of 0.02, 0.05, 0.08 and 0.1 mm and a series of collectors: one or two parallel aluminum tubes, a steel grid with a cell size of S = 2x2 mm2 and drop shape collector. Voltage was supplied from Plazon and varied from 0 to 30 kV. The height between the electrodes was changed from 5 to 50 mm. Changing the weight of the structure due to the creation of a thrust was measured by Ohaus Scout Pro scales.

Its known, that the thrust of electrode systems is proportional to the distance between the collector and the emitter [3]. In practice, this dependence is not fully implemented. An increase in the length of acceleration zone leads to a linear increase in thrust. With its growth, the increase in thrust slows down. At distance about 20-30 mm the thrust and velocity is maximal (pic 1b). At low current values the systems are more effective. At small emitter-collector distances, all the types of electrode systems have shown approximately equal characteristics. With increasing distance, a system with a negative corona is more efficient than a device with a positive corona similar in configuration. This is due to the difference in the composition of ions and their mobilities in the air for different polarities. Using an emitter of smaller diameter, the thrust and efficiency gain reached 10%, which is expected from the point of view of the estimation of the losses for corona formation. The results of the study can be used in other fields: pumping gas-discharge lasers [4], plasma sources for ion cloud acceleration systems [4, 5], cooling systems [6] etc.

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PARAMETERS OF THE BEAM PLASMA NEAR THE ISOLATED COLLECTOR

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Plasma generation by electron beam is an alternative to the electrode, RF and microwave discharge. This plasma can be used for different applications such as plasma chemistry, ion etching, coating deposition, sterilization [1]. One of the problems arising in the creation of beam plasma is an increase in the energy efficiency of the method. It can be partially solved by the use of secondary electrons emitted by the beam collector. The contribution of secondary electrons to the ionization of a gas depends both on their quantity and energy. The former is determined by the secondary emission coefficient, the second - by the potential difference between the plasma and the collector. The purpose of this paper is to establish the dependence of the beam plasma concentration on the combination of the collector-gas material, if collector is isolated.

The experiments were carried out on an installation (Fig. 1), which includes a chamber 1 pumped out by a mechanical fore-vacuum pump, a plasma electron source 2 with a focusing system 3, an isolated collector 4. A beam 5 produced a plasma whose parameters were measured by a probe 6 with a beam-shielding shield. Stainless steel, steel 3, copper, titanium, aluminum were used as a collector materials. Working gases: argon, oxygen, nitrogen. Pressure p is controlled by the gas inlet to electron source.

As a result of the experiments it was established that the energy of the primary electron beam, as well as the material of the collector, play a decisive role in establishing the potential of an isolated collector. Influence of the gas kind manifests itself to a lesser extent. As a rule, collector potential grows monotonically with accelerating voltage U_a (Fig. 2). Plasma concentration has a maximum at collector potential 200-300 V. Since the collector potential determines the energy of the secondary electrons, this correspondence makes it possible to explain the presence of a maximum by the contribution of secondary electrons to ionization [2], since the ionization cross section is maximal for electrons with energy of 100-200 V.

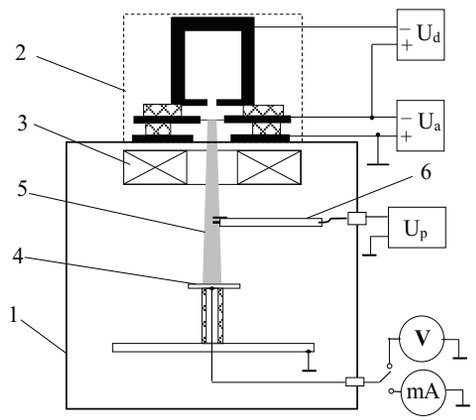

Fig. 1. The scheme of the experimental setup.

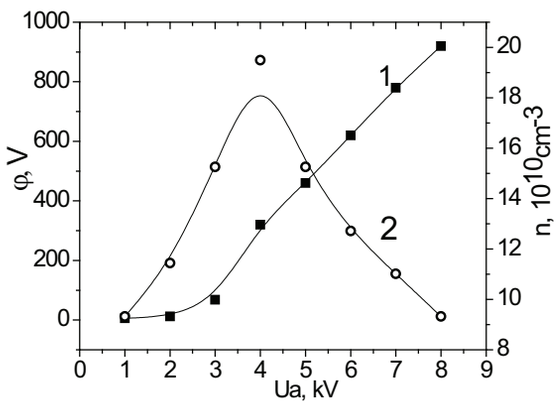

Fig. 2. Potential φ (1) of the collector and plasma concentration n (2) as a function of the accelerating voltage U_a. Collector – stailess steel, gas – argon, p=1,5 Pa, beam current 5 mA.

The obtained results show that the efficiency of the electron beam for plasma generation can be substantially increased by using an isolated collector.

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ANGULAR AND ENERGY DISTRIBUTIONS OF IONS IN PLASMA VACUUM ARC WITH ZIRCONIUM CATHODE SATURATED BY DEUTHERIUM[1]

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Experimental studies on the measurement of the angular and energy distributions of ions in a vacuum arc plasma with a zirconium cathode saturated with deuterium are presented. These experiments are a continuation of our earlier studies [1,2] in which we studied the mass-charge composition of an arc plasma generated in an ion source of a vacuum arc MevvaV.Ru with a zirconium cathode saturated with deuterium. It was shown that the angular distribution of deuterium ions is much wider than the distribution of zirconium ions. In this case, the distribution of zirconium ions, in the case of a deuterium-saturated cathode, coincides with the distribution for a pure zirconium cathode.

Measurements of the velocities of deuterium and zirconium ions were carried out by the method of fast "breaking" the arc current, when a high-speed ignitron switch shunted the arc, and the ion velocity was estimated by delaying the reaction to the "breakage" of the ion current of plasma fractions. This time delay occurred when ions moved from the cathode to the emission electrode. As a result, it was shown that the velocities of zirconium ions of all charge states are equal, and the velocity of deuterium ions is almost twice as high as that of zirconium velocities are. The energy of the deuterium in the vacuum arc plasma was about several eV, and energy of zirconium ions were about 90 eV. The results of studies are discussed.

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OPTICAL EMISSION SPECTROSCOPY STUDY OF THE INFLUENCE OF THE LOW-ENERGY ELECTRON BEAM PARAMETERS ON THE CONTENT OF NEUTRAL ATOMIC NITROGEN IN THE BEAM PLASMA [1]

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The influence of plasma generation mode (continuous and pulse-periodic), electron beam parameters (current 2 – 14 A, energy 100 – 300 eV) and the pressure of nitrogen-argon gas mixture (0,3 – 3 mTorr) upon relative content of neutral atomic nitrogen in beam plasma was experimentally studied. Direct comparison of relative content of neutral atomic nitrogen in beam plasma and the plasma of discharge with self-heated plasma cathode was also done. All else things being equal, the maximum content of atomic nitrogen is observed in the plasma of high-current electron beam in pulse-periodic mode of discharge burning in the environment of nitrogen-argon gas mixture. In the plasma of discharge, the atomic nitrogen content is 4-5 times lower than those in beam plasma (Fig. 1). Using of pulse-periodic mode leads to growth of the relative content of atomic nitrogen by 20% (Fig. 2). The rise of pressure, accelerating voltage and discharge current also lead to increase of the share of atomic nitrogen, at that gas mixture pressure and average value of beam (discharge) current exert the strongest influence. Correlation of the relative content of atoms (IN^*/IN_2) and molecular ions (IN_2^+/IN_2) of nitrogen (Fig. 1) confirms (as well as [1]) that dissociative recombination of molecular nitrogen ions with low-energy plasma electrons is the most possible reaction of atomic nitrogen formation, and molecular ions formation takes place, in its turn, preferably due to direct ionization by electron impact.

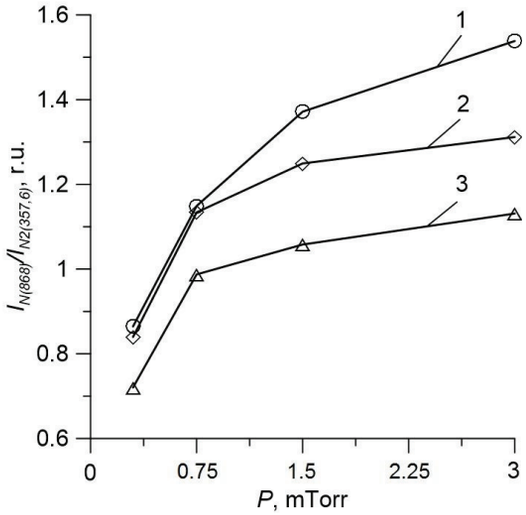

Fig. 1. Dependencies of the relation between intensities IN^*/IN_2 and IN_2^+/IN_2 in the spectra of direct discharge plasma (60 A) and electron beam (10 A, 200 V) on nitrogen pressure.

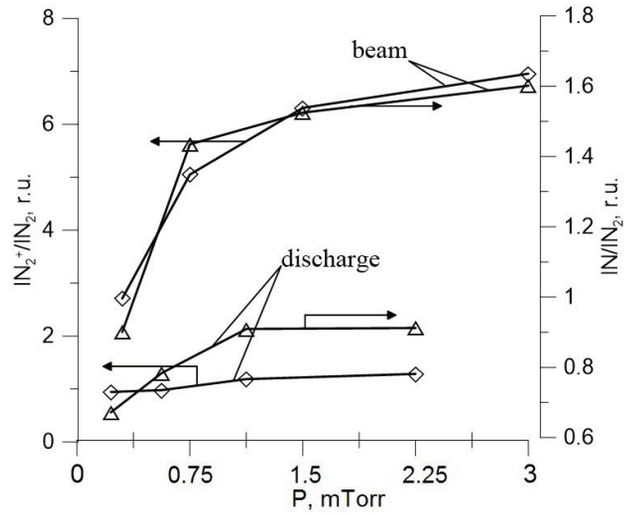

Fig. 2. Dependencies of the relation between intensities IN^*/IN_2 in the spectra of electron beam plasma on the pressure in vacuum chamber in pulse-periodic (1) and continuous (2, 3) modes in the environment of nitrogen (3) and nitrogen-argon gas mixture (1, 2).

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OPERATION OF HIGH-VOLTAGE AC PLASMA TORCHES WITH CHANGING PRESSURE

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Many chemical processes occur at elevated pressures. The implementation of plasma processes in such technologies requires the development of reliable electric arc plasma torches operating at pressures other than atmospheric pressure. This leads to a reduction in the reactor volume and a reduction in the number of compressors. Especially relevant is excess pressure plasma torches in the gasification of solid organic substances [1], reforming of natural gas [2] and processing of organochlorine compounds [3]. At present, AC atmospheric pressure plasma torches operating in air [4], steam (mixed with other gases) have been developed [5]. The increase in pressure after the plasma torch leads to its unstable operation, which significantly reduces the life time of electrodes and the technological parameters of the plasmachemical process.

The report presents results of studies of air three-phase high-voltage AC plasma torches under pressure changes. The plasma torch is three separate discharge channels with a tangential feed of the plasma-forming gas and three copper rod electrodes. The power of the plasma torches reached 70 kW, the range of the excess pressure variation from 0 to 4 atmospheres, the idling voltage of the power supply was 10 kV.

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INVESTIGATION OF AVERAGE CHARGE STATE OF COPPER IONS IN THE LOW-CURRENT VACUUM ARC PLASMA AT DIFFERENT VALUES OF DISCHARGE CURRENT

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The dependence of the average charge state of copper ions on the arc current value was investigated. The experiment was carried out in the range of discharge currents from 1.8 to 40 A. As opposed to the previous study [1], quasi-rectangle current pulses formed by a LC-line were used. Therefore the estimation of the actual current value was more accurate. Pulse duration was about 2 μs. The pulsed electrostatic gate was used to exclude from analysis the plasma flux formed at the ignition mode of the discharge. The charge composition of ions was obtained via the Thomson spectrometer with automated recording and processing of spectrograms.

There was obtained the dependence of average charge state of copper ions on the value of discharge current, as shown in Fig.1. The average charge of copper ions significantly decreased with the arc current decreasing from the tens of Amperes to the minimum spot current for copper. And there was shown that the arc current value, and therefore the quantity of simultaneously operating emission centers is the one of the basic factors influencing on the charge composition of the vacuum arc plasma.

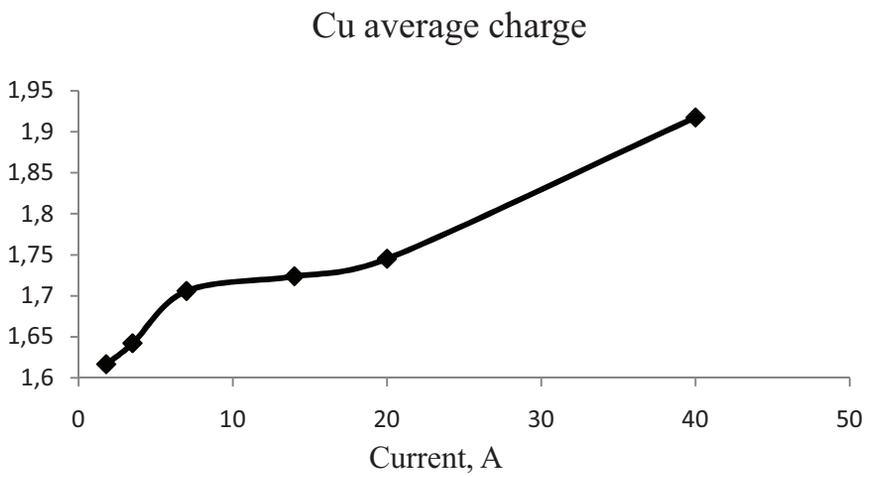

Fig. 1. The average charge of copper ions depending on arc discharge current

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INVESTIGATION OF THE MASS-CHARGE COMPOSITION OF ION FLUX FROM THE PLASMA OF THE VACUUM ARC DISCHARGE ON CUCR CATHODE

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Mass-charge composition of ion flux from the CuCr cathode of low current vacuum arc was obtained via the Thomson spectrometer with automated recording and processing of spectrograms. The microsecond arc was ignited in high vacuum conditions between the CuCr cathode and the copper anode. The arc current was varied in the range from units to tens of amperes.

There were obtained data on charge composition at different values of the discharge current for copper and chromium ions. Few differences between the dependences of these charge compositions on the arc current were revealed.

THE CUTTING EDGE SHARPENING BY FAST NEUTRAL ARGON ATOMS[1]

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A professionally sharpened samurai sword can cut a silk handkerchief that falls on its blade. Before the sword whetting, both faces of its blade are smoothed out with the most hard and dense hone stone. The smaller the grains of the stone, the sharper is the cutting edge and the more durable is the whetting results. For reduction of the grain to the minimum possible atomic size, it was proposed in the present work to whet the blade by etching its faces with fast neutral argon atoms.

The left figure is a schematic of the knife blade etching with a homogeneous converging beam of fast neutral atoms. The beam is produced by a curved grid immersed in a uniform plasma and negatively biased to 5 kV [1]. The plasma is generated by the glow discharge with electron confinement in a vacuum chamber playing the role of the discharge hollow cathode [2]. Argon ions are accelerated in the positive space charge sheath between the plasma and the convex cylindrical surface of the grid. They fly through the grid holes and are decelerated in the positive space charge sheath between the plasma and the concave surface of the grid.

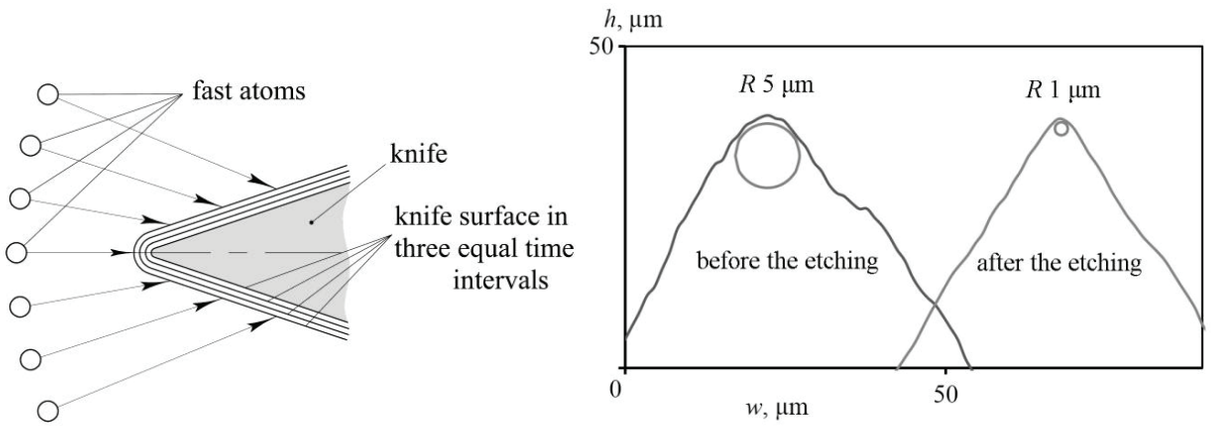

Fig. 1. Schematic of the knife etching (left) and profiles of the knife cutting edge (right)

With an increase in the argon pressure from 0.1 to 1 Pa, the fraction of ions that passed through the grid and turned into fast neutral atoms as a result of the charge exchange collisions approaches 100%. They sputter the cutting edge and the blade faces with the same rate, which leads to a reduction in the cutting edge radius R.

After processing a ceramic knife made of zirconium dioxide for one hour, its cutting edge radius decreased from the initial value of 5 μm to R = 1 μm, and the lateral faces of the blade were noticeably leveled. Attempts to further reduce the radius of the cutting edge by etching the knife for another one hour failed. Apparently, the radius R of the cutting edge is limited by the size of the knife grains. To reduce the cutting edge radius down to R = 0.1 μm, the size of the knife grains should not exceed 0.01 μm. It means that the knife should be made of a nanomaterial.

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SURFACE STRENGTHENING IN PLASMA OF NON-SELF-SUSTAINED GLOW DISCHARGE WITH ELECTROSTATIC CONFINEMENT OF ELECTRONS[1]

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Ion nitriding, plasma immersion ion implantation and synthesis of wear-resistant coatings are widely used for the surface strengthening of various machine parts. In order to use all these ion-plasma processing methods, it is needed to fill the process vacuum chamber with a homogeneous plasma at the working gas pressure ranging from 0.01 to 1.0 Pa. For the plasma production are often used the vacuum arc discharge and the magnetron discharge. In both cases, the plasma is non-uniform and contains particles of the cathode material – metal ions and atoms, as well as metal droplets in the case of the vacuum arc. Another gas discharge form widely used for the ion-plasma processing is the RF discharge.

It was shown in [1] that glow discharge with electrostatic confinement of electrons can fill any vacuum chamber with a dense and homogeneous plasma at the discharge voltage of 400–500 V and the gas pressure p = 0.01–1.0 Pa. So it can be used at p = 0.01–0.1 Pa for the plasma immersion ion implantation and at p = 0.1–1.0 Pa for the coating synthesis and the ion nitriding. Despite the high homogeneity of the discharge plasma heating of the product in the latter case under a negative bias voltage by accelerated from the plasma ions results in an intensive etching of its sharp edges. While nitriding a cutting tool it leads to the cutting edge material removal and the tool blunting. Another drawback of the discharge is contamination of the plasma with impurities sputtered from the chamber walls bombarded by ions with the energy of 400–500 eV. To solve the problem it was proposed in the present work to use for the plasma generation the glow discharges sustained by broad beams of accelerated ions or electrons injected into the vacuum chamber.

In the first case a source of fast neutral atoms and molecules is used [2]. The ions accelerated in the sheath between the plasma emitter of the source and its single grid enter through the grid holes the vacuum chamber. At the nitrogen pressure p = 0.2–1.0 Pa due to charge exchange collisions with gas molecules the ions with energy of 1–4 keV turn into fast neutral molecules with the same kinetic energy and ionize the gas. To prevent the fast molecule beam from sputtering the opposite wall of the chamber the latter is covered with a set 0.5-mm-thick titanium plates parallel to the beam axis.

At a voltage U □ 10 V applied to the anode placed inside the chamber all electrons produced inside the chamber are switched to the anode circuit. Growth of the discharge voltage to U □ 300 V results in self-sustained discharge, which does not expire after the beam source is switched off. In non-self-sustained discharge at U = 10–20 V the chamber walls are not sputtered by the ions and heating of the tools by homogeneous neutral beam instead of the ions accelerated from the plasma does not lead to the cutting edge blunting.

In the second case a grid is used as a part of the chamber wall and, simultaneously, as the anode of an auxiliary hollow cathode glow discharge. All electrons produced inside the hollow cathode enter through the grid holes the sheath near the chamber wall. They are accelerated in the sheath and start their oscillating movement through the plasma together with electrons emitted by the chamber walls. Increase in the number of fast electrons entering the plasma results in a substantial decrease in the discharge voltage down to 50–60V and growth of the molecular gas dissociation degree. It allows an effective ion nitriding of the products without their contamination with impurities from the chamber walls.

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STUDY OF PLASMA PARAMETERS AND OPTICAL EMISSION IN DC, HIPIMS AND HYBRID DC + HIPIMS MODES OF MAGNETRON SPUTTERING [1]

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Direct current (DC) magnetron sputtering is the most basic and inexpensive type of magnetron sputtering. But during DC magnetron sputtering the plasma concentration is low (about 10^9 cm^{-3}), and its ionic component is represented mainly by the ions of the working gas (Ar^+). In high-power impulse magnetron sputtering (HiPIMS) high peak voltages (up to 3000 volts) and high peak power densities (up to 3000 W/cm^2) are used at low duty cycles to give an average power similar to DC magnetron sputtering (e.g. 3 W/cm^2) and the plasma density could be as high as 10^{12} cm^{-3} [1]. However, a significant disadvantage of HiPIMS is the reduction in the deposition rate of coatings compared to DC sputtering. The reason for this is the attraction of the ionized sputtered material back to the cathode [2]. Various methods have been attempted to improve deposition rate, such as, hybrid secondary plasma source, pulse waveform modulation. Another commonly utilized approach is hybrid technology, such as hybrid DC + HiPIMS co-sputtering processes [3].

The aim of this work was to study the plasma parameters and optical emission during hybrid high-current magnetron discharge, in which high-current pulses are superimposed on a direct current. The plasma parameters and spectroscopic properties in different modes of magnetron sputtering have been investigated. DC, HiPIMS and hybrid mode (DC + HiPIMS) of the magnetron sputtering were realized at an average power of 2 kW with a planar magnetron sputtering system with a Cu target.

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MICROWAVE DISCHARGE OF REDUCED PRESSURE TO MODIFY THE CRYSTAL SURFACE

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The characteristics of the discharge in argon excited by microwave radiation from a magnetron with a power of 800 W and wavelength λ = 12.2 cm were investigated. The discharge burned in a working chamber made of quartz glass with a working gas pressure in the range of 6-30 Pa. Emission spectra were recorded through the chamber wall using an AvaSpec-2048 fiber optic spectrometer in three sections of the chamber located at different distances from the emitter. It is shown that the plasma concentration decreases sharply with distance from the microwave emitter, and the temperature calculated from the emission spectrum in the LTE model varies insignificantly and lies in the range Te = 1.5 - 1.8 eV. The numerical simulation through the Comsol package gave Te = 2.6 eV. Samples of the LiF crystal placed in the chamber acquire a coloration characteristic of the F2 and F3 + centers. Using thermo luminescent detectors on the basis of LiF, a very high level of radiation in the volume was also detected , so that the absorbed dose of the TLD near the emitter was about 1300 R in 2 seconds, and when detector was removed from the emitter, fell to 250 R in 2 seconds. The presented results show that this device can be used to modify the surface of solids, for example, to create luminescent layers in alkali-halide crystals.

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GENERATION OF HIGH CHARGE STATE HEAVY METAL ION BEAMS[1]

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High charge state metal ion beams are used in fundamental nuclear and atomic physics, also for applied problems, such as surface modification by an ion beam. The metal ions of plasma produced by the vacuum arc, as a rule, have a charge state from 1+ to 3+. The actual problem is to obtain ions with high charge states, since at a fixed accelerating voltage this leads to a substantial increase in the energy of the ion beam. The charge states of the ions can be substantially increased in the case of a vacuum arc discharge with a short pulse duration. The use of such a discharge with a pulse duration less than 10 μs and a current up to 10 kA makes it possible to obtain ion beams with a current of several amperes with a mean ion charge state about 10+. This paper describes design of the discharge system and some results of the high charge state heavy metal ions beams generation.

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VOLT-AMPERE CHARACTERISTIC OF TWO-ELECTRODE GAP AT HIGH PRESSURE AND AN ARBITRARY EMISSION OF CURRENT CARRIERS

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In some cases, as the ion source used three-electrode systems, in which the discharge is ignited between the electrode and the grid, and the ions are extracted using the electric field of the collector in the gap grid – collector. Considering the grid electrode as an equivalent emitter, it is possible to calculate the volt-ampere characteristic of a two-electrode gap at arbitrary emission of current carriers moving in the mobility mode.

For the case of arbitrary emission of current carriers in the interval between electrodes, it is known that the electric field intensity on the emitter surface can be calculated using the equation:

$$E_0 = \frac{\beta_0\left(\frac{j}{j_0}\right)U_a}{L}, \tag{2}$$

where $\beta_0\,(j/j_0)$ – is a dimensionless function describing the degree of shielding of the emission surface by a volumetric charge, $j -$ is the current density flowing between the electrodes of the gap, U_a – a is the voltage between the electrodes of the gap.

For the case of arbitrary emission of current carriers is also known:

$$E_0 = \frac{\bar{c}}{4b\left(\frac{j_e}{j_0} - 1\right)}, \tag{3}$$

where $\bar{c}$ - is the mean arithmetic value of the thermal velocity, j_e - is the emission current density, b - is the mobility coefficient of charged particles.

Equating the expressions (2) and (3), we obtain an equation that allows us to calculate the desired volt-amperes characteristic. This equation is convenient to present in the form of

$$\frac{j_e}{j_0} = \varphi\left(\frac{j}{j_0}, A\right), \tag{4}$$

where

$$\varphi\left(\frac{j}{j_0}, A\right) = \left[1 + \frac{A}{\beta_0\left(\frac{j}{j_0}\right)}\right]\frac{j}{j_0}, \qquad A = \frac{\bar{c}L}{4bU_a}.$$

Equation (4) allows to find j depending on L, U_a and $j_e$as a result of graphical or numerical analysis. The value of j_e in the considered conditions is found through the current density in the ion source and the optical transparency of the grid electrode. On the fig. 1 presents the calculated and experimental volt-ampere characteristics.

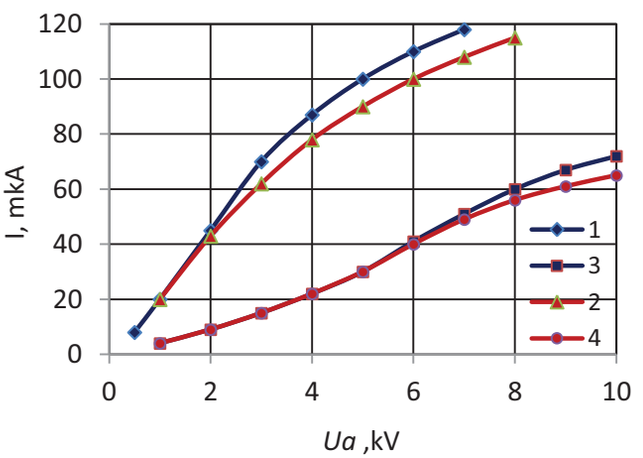

Fig. 1. Design and experimental volt-ampere characteristics: 1, 2-calculation and experiment at L=3 mm, j_e=160 μa; 3, 4 – calculation and experiment at L=11 mm, j_e =160 μa.

SOME NON-VACUUM APPLICATIONS OF GUNS WITH A PLASMA EMITTER

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The paper presents a description of electron-beam equipment for transportation of the focused beam to air. The equipment contains an electron gun with a plasma emitter and a system of gas-dynamic windows that create a pressure drop from value 10^{-4} Torr to an air pressure of 760 Torr. The electron beam energy is 120 keV. Measurements of the beam diameter into air at working distances of 5-15 mm have been performed.

It is shown that guns with a plasma emitter can be effectively used to produce nanopowders of metal oxides (zinc oxide in our case) in the atmosphere. The size of nanoparticles in the resulting powders does not exceed 100 nm.

The results of non-vacuum production of carbide coatings with good heat-, wear- and corrosion resistance are presented. A particular feature of the method of creating coatings is the use of SHS-process followed by electron-beam surfacing.

The possibility of using the equipment for realization of electron-beam welding of joints of different complexity is shown. The results of two types of welded joints are presented: aluminum parts and copper parts. In addition, the possibility of using an electron beam for welding electrodes of industrial batteries was demonstrated.

FEATURES OF THE FORMATION OF HIGH-INTENSITY BEAMS OF ALUMINUM IONS[1]

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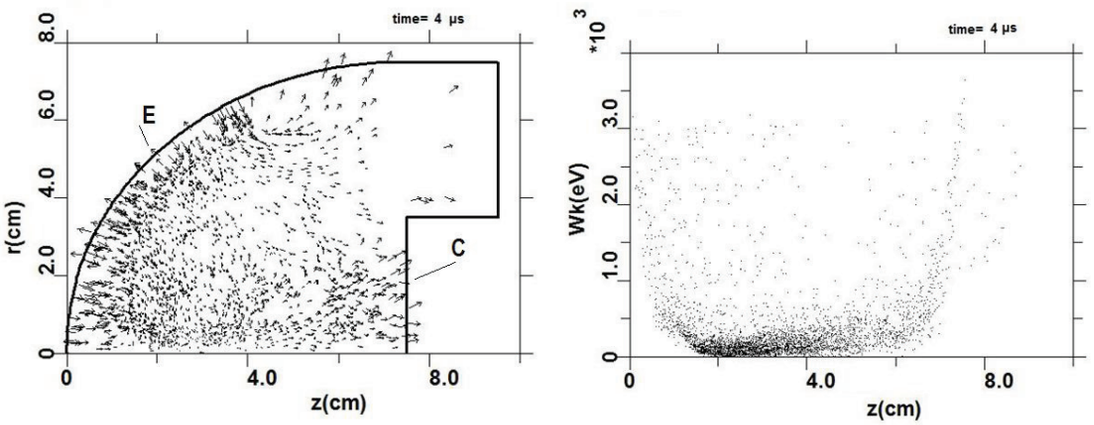
A hybrid system of the formation of ion beams, combining the features of the traditional grid ion extractors and the plasma-immersion method of ions extraction with their subsequent ballistic focusing, is a promising source of high-intensity beams of metal, gas and mixed ions for high-intensity implantation of low-energy ions [1].

In this paper the plasma-immersion formation and transportation of a high-intensity beam of aluminum ions under ballistic focusing conditions is studied experimentally and theoretically with the use of numerical simulation (PIC code KARAT). The influence of metal plasma parameters, amplitude-frequency characteristics of the negative bias potential, gas pressure in the experimental chamber and preliminary injection of plasma into the transport region on the formation of an ion beam and its characteristics on the collector are carried out.

It is shown the focusing of ion beam is accompanied with the formation of inhomogeneous potential well, which defines the movement of charged particles of plasma (ions and electrons) and under certain conditions (determined by the geometry of the drift space, plasma parameters, amplitude and frequency of the negative bias potential) to the formation of a virtual anode. Fig. 1 shows a configuration portrait of beam ions and their energy distribution under conditions of a significant deficit of plasma electrons at time t = 4 μs; pulse durations of τ = 8 μs, average plasma density $n = 4 \cdot 10^9$ cm $^{-3}$.



a b

Fig.1. Configuration portrait of the ion beam (a) and energy distribution (b); E - grid electrode, C – collector

Numerical simulation and experimental studies make it possible to optimize the conditions for the formation of focused ion beams with respect to high-speed deep ion doping of materials.

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OBTAINING OF METAL ION BEAMS BY HEATING METHODS IN AN ECR SOURCE OF DC-60 CYCLOTRON[1]

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Accelerating complex DC-60 (in operation from 2006) is a unique electro-physical unit for accelerating charged ions in the range from lithium to xenon. Over the past 11 years of cyclotron operation, numerous works have been carried out in full on the development of methods for obtaining gas ions at the ECR source. To solve this problem, a number of ion beams were obtained at the ECR source and the modes of ion acceleration were successfully worked out for the following: ^{4}He, ^{12,13}C, ^{14,15}N, ^{16,17}O, ^{20,22}Ne, ^{32}S, ^{40}Ar, ^{84}Kr and ^{132}Xe in the energy range 0.4 - 1.75 MeV/nucleon [1].

The next stage to expand the types of accelerated ions used at cyclotron is the development of methods for heating the working substance to create the required vapor pressure and introducing it into the ECR source. In this paper we will consider two methods for obtaining ion beams: direct injection and evaporation from a crucible.

Direct injection is one of the methods for obtaining solid substances, realized by introducing a container with solid into the plasma of the ECR source. The control of substance vapor pressure is ensured by the depth of entry of the container into the plasma. Direct injection is most suitable for refractory materials, such as bismuth, niobium, tantalum, tungsten, etc.

Also, there is a method to obtain ions from ECR source named *evaporation from the crucible.* It is proposed to heat the working substance in the crucible using a micro-furnace. The adjustment of working vapors of substance in this method is carried out by changing the heating power of the crucible. This method works well with fusible materials, such as lithium, magnesium, calcium, etc.

Works were carried out to obtain beams of lithium, magnesium, calcium ions by direct injection and evaporation from the crucible. Table 1 shows the obtained intensities of ion beams after optimization of ECR source operation.

Table 1 - Intensities of ion beams in the ECR source, μA.

Ion type	Ion charging								
	1+	2+	3+	4+	5+	6+	7+	8+	9+
^{6}Li	37	20							
^{7}Li	520	45							
^{24}Mg			60	81	79	98			
^{31}P				40	61	35	16		
^{40}Ca					135	100	72	53	30

Therefore, ion beams were first obtained by two methods of heating a working medium with high intensities, on the ECR source of the DC-60 cyclotron, which were successfully injected into the cyclotron and accelerated.

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ONLINE METHOD OF DIAGNOSTICS OF PHYSICAL QUANTITIES ON THE ACCELERATOR DC-60

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In various accelerator systems is often necessary to analyze the dynamics of time-varying values (current, voltage, pressure, etc.) at the cyclotron DC-60. In this regard, for the monitoring of parameters of some physical quantities of the cyclotron developed online diagnostics based on SCADA systems.

Based on the computer system of the accelerator DC-60 implemented an automated workstation that allows without interference in the software complex of the accelerator and it's quickly interrogate the array of necessary data. Tracking changes in physical quantities in real time allows quickly to influence on the processes occurring during irradiation of targets, thus visualizing deviations from the specified values of all systems. For example, it is necessary to analyze static values when irradiating a polymer film on a cyclotron DC-60. The dynamics of these values changing over time (current, voltage, pressure, etc.) in various accelerator systems leads to deterioration in the quality of work. In this regard, for monitoring parameters of the systems of the cyclotron to the irradiation process has been developed the online diagnostics parameters of the cyclotron-based SCADA systems. Using the OPC-server ModBus RTU/ASCII /TCP, which allowed visualization the interesting parameters of the cyclotron.

This system allows collecting and storing the database with the indication of the polled values in trends while maintaining the important parameters of the accelerator DC-60.

Figure 1 shows the dynamic characteristics of physical quantities when setting the accelerator to output accelerated ions in the transport path and deviation of these values from the norm. On trends appear manifest deviation from the set values of the electrostatic deflector system that requires correction and operant treatment of cyclotron personnel.

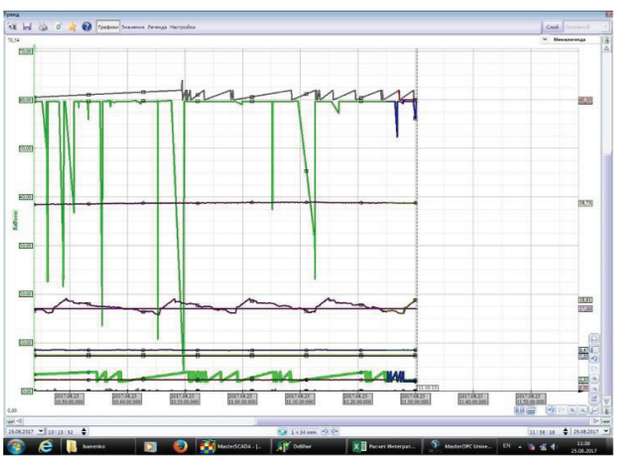

Fig. 1. Dynamic characteristics of physical quantities of the accelerator DC-60

The system allows to include data on the accelerator state in the polling cycle and to adjust its parameters depending on the requirements of the experiment directly during measurements. Provide the highest quality beam of accelerated ions and observe the dynamic processes in real time.

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MASS-SPECTROMETER WITH EXB FILTER FOR BEAM AND PLASMA DIAGNOSTICS[1]

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Corpuscular plasma diagnostics is a unique source of information about fluxes of charged particles in technological plasma facilities [1, 2]. Among topical research areas of gas discharges in magnetron sputtering systems, variously configured plasma chemical reactors, plasma immersion ion implantation devices, etc., studying ionic species abundance and measuring fluxes of different ions and their energy distributions are of a considerable interest. In present contribution, we investigate the characteristics of a compact modular mass-spectrometer based on E×B probe (or Wien filter) [3]. The results of numerical simulations of ion trajectories in the analyzer as well as the experimental results of mass spectrometer application for ion content diagnostics in a number of beam and plasma facilities are presented. It is shown that the resolution of the filter is sufficient to perform quantitative measurements of fluxes of different ionic species produced in plasma-material treatment processes.

The E×B probe under investigation was 12 cm long; the magnetic field was 0.63 T on the major axis of the ion optics, and the voltage between electrodes was up to 1 kV. The experiments were carried out in a special diagnostic ion beam facility with an electron impact ion source (ion energy up to 3 keV, energy spread < 10 eV), in an inductively coupled plasma reactor, and in a cusped magnetron plasma source. A sample mass spectrum recorded from the ion source is shown in Fig. 1.

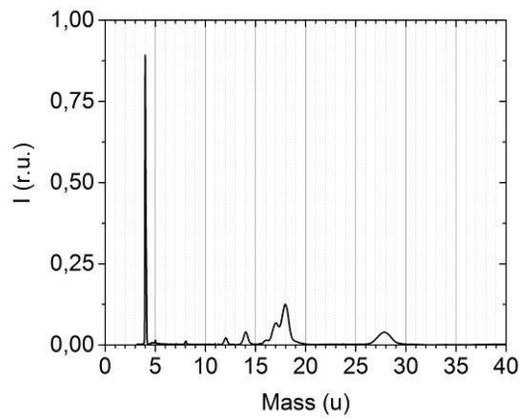

Fig. 1. Mass spectrum of an ion beam from He ion source

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INFLUENCE OF COMBUSTION CONDITIONS ON THE TEMPERATURE DISTRIBUTION ON THE SURFACE OF EXTENDED METAL PRODUCTS HEATED IN A PLASMA OF GLOW DISCHARGE WITH HOLLOW CATHODE [1]

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Ion modification of the surface layers of metal products by ion-plasma methods is widely known, and these methods have been used for a long time in industry to produce products with modified surfaces. These methods are well studied and successfully applied to the treatment of external surfaces. Such treatment requires the generation of low-temperature plasma by means of discharges of various types in the working volume of the vacuum chamber and the placement of processed products in this plasma. For the treatment of the inner walls of elongated products, the choice of discharge is limited by a glow discharge, while the workpiece is a cathode and participates in the generation of a gas-discharge plasma. Existing schemes for treating the internal surfaces of elongated cylindrical products [1, 2] assume the presence of a fibriform electrode as an anode placed along the axis of the workpiece. Such systems are unable to process curved long cavities. In this work was designed and manufactured a system in which the anode was located locally from one end of the cavity, which removes the limitations in the curvature of the processed articles, and for the uniform generation of plasma from the opposite end of the cavity, additional electrons were injected from the auxiliary discharge plasma. Thus the ignition and maintenance of the self-supporting glow discharge with a hollow cathode was implemented.

In this discharge system, the cavity of the workpiece was the cathode of a non-self-sustained glow discharge, and the anode was a specially introduced tungsten electrode. On the side of the anode, a plasma-forming gas was fed. At the opposite end of the cavity was located an emission grid that, together with the outer surface of the product, was the anode of an auxiliary arc discharge generated by a plasma source with a combined heated and hollow cathode [3]. Electrons from the auxiliary discharge plasma are closed to the outer surface of the pipeline and flying through the grid, enter the cathode layer of the glow discharge, where they are accelerated to an energy corresponding to the burning voltage of the main discharge. Since the walls of the cavity are the cathode of the main discharge and form an electrostatic trap, the accelerated electrons, falling into the cavity of the cylinder, begin to oscillate and lose their energy in inelastic collisions with the molecules of the plasma-forming gas. Thus, the effect of a hollow cathode is realized, which contributes to an increase in the uniformity of plasma generation along the entire length of the extended curvilinear cavity.

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PECULIARITIES OF COMBUSTION OF A NON-SELF-SUSTAINED LOW-PRESSURE GLOW DISCHARGE WITH HOLLOW CATHODE THE EMITTING SURFACE OF WHICH IS COVERED BY TITANIUM NITRIDE [1]

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Recent studies have shown that the processes of nitriding steels and titanium are most effective at an ion current density of about 4 mA / cm^2 or more [1]. To obtain such high values of the ion current density on large areas of products, it is required to obtain a discharge current in the same chamber volume of more than 100 A. An increase in the discharge current can be achieved by injecting of additional electrons [2]. If in the case of a self-sustained discharge with a hollow cathode the cathode current is equal to the sum of the ion current and the electron current produced as a result of secondary ion-electron emission (γ-electrons), then in the case of a non-self-sustained combustion regime, a term equal to the current of the emitted electrons is added. These electrons are emitted into the plasma of a glow discharge, accelerated in the near-cathode potential drop, oscillate inside the hollow cathode and ionize the gas. This makes it possible to obtain discharge currents of several hundred amperes with the possibility of independently adjusting the combustion voltage of the discharge. In a non-self-sustained glow discharge, injected electrons, like γ-electrons, are primary, but unlike γ-electrons, their electron emission current density is 2 to 3 orders of magnitude higher than the emission current density of gamma-electrons, since electrons are emitted from a small area emission window. As a result, a local increase in the density of plasma concentration is created in the direction of their emission. At discharge currents of 100 A, the maximum and minimum values of the ion current density at the azimuthal density distribution of the ion saturation current can differ several times. In view of this, the question remains of the possibility of increasing the fraction of electrons emitted from the surface of the hollow cathode as a result of secondary ion-electron emission processes.

In this paper we investigated the possibility of increasing the current of primary γ-electrons obtained on the surface of a hollow cathode coated with titanium nitride, and also the possibility of reducing the influence of electron injection discharge from a small emission area on the uniformity of the plasma density distribution.

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INFLUENCE OF THE CONDITIONS OF COMBUSTION OF THE EMITTER DISCHARGE ON THE HOMOGENEITY OF DISTRIBUTION OF PLASMA DENSITY IN A NON-SELF-SUSTAINED GLOW DISCHARGE WITH HOLLOW CATHODE [1]

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To decrease the working pressure in a glow discharge with a hollow cathode up to $5 \cdot 10^{-3}$ Pa allows external injection of electrons [1]. In addition, it becomes possible to independently adjust the current, discharge voltage and operating pressure, which is important for practical applications. At relatively low burning voltages of such a non-self-sustaining glow discharge (up to 300 V) and relatively high (above 10 A) currents, the current of a glow discharge increases linearly with increasing current of injected electrons [2]. In such a two-stage discharge system, in the case of current oscillations of the injected electrons coming from the first stage, the current oscillations of the glow discharge (the second stage) will also be observed. Since the first stage serves as the source of electrons, which should be accelerated mainly in the near-cathode fall of the potential of the main glow discharge, it is advisable to use in the first stage a discharge with a low combustion voltage. In this case, the energy efficiency of the two-stage system as a whole increases. Such requirements correspond, for example, to an arc discharge with a cold hollow cathode [3], which was used in [2,4]. The configuration of this plasma source with two magnetic field coils is presented in [5], which allows controlling the motion of the cathode spot along the inner surface of a cylindrical cathode. This increases the life of the cathode. However, when the cathode spot moves, fluctuations of the arcing voltage of the arc discharge occur. If such a configuration is used to generate a plasma from which electrons are emitted into a glow discharge, then the parameters of the injected electron beam change when the arc discharge voltage fluctuates.

In this paper we investigated the influence of fluctuations of the discharge voltage of an auxiliary arc discharge on the fluctuations of the current of the main glow discharge and the uniformity of the distribution of the plasma concentration in a non-self-sustained glow discharge with a hollow cathode.

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VIBROACOUSTIC DIAGNOSIS OF THE SURFACE ALLOYING PROCESS [1]

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Studies of the processes of surface alloying of materials using concentrated energy flows have shown the opportunity of creating wear-resistant surface layers using electron beam technology. However, the instability of the parameters of the pulse electron beam and the process of its interaction with the processed material leads to quite significant random changes that occur spontaneously, regardless of the control system.

Experiments have shown that in this situation the acoustic c emission method can provide significant assistance. Registration of vibroacoustic signal allows monitoring, , stabilizing and optimizing the treatment.

The process of surface alloying was studied on the installation "RITM-SP"[1]. The working table of the unit was replaced with a nitride sample of steel 08Cr17Ti. A number of plates were coated by magnetron sputtering by Nb70Hf22Ti8 i8 alloy coating, which was subsequently used as a material for surface alloying. Irradiation causes dissociation of iron nitrides. The released nitrogen atoms enter into an exothermic chemical reaction with the coating material to form a refractory nitride phase based on Nb and Hf.

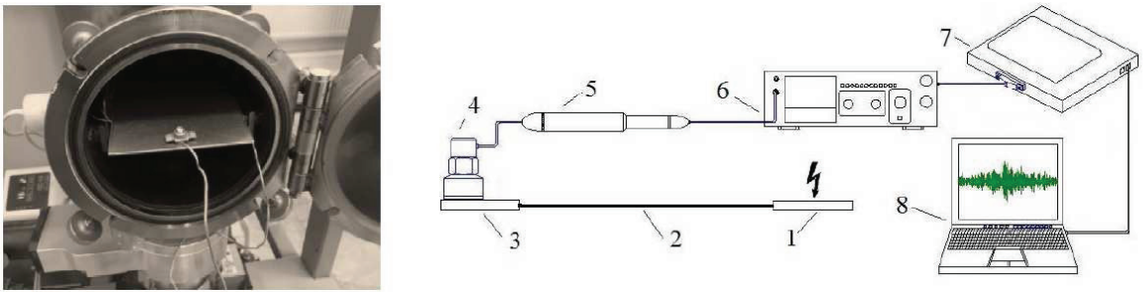

Fig. 1. Circuit channel for recording vibroacoustic signals: 1-test sample; 2-wire waveguide; 3-receiving plate; 4-accelerometer KD-35 with magnet; 5-preamp preamp PM-3; PM 6-amplifier VSHV-003; 7-ADC E440; 8-recording computer

To register the vibroacoustic signal, the plate was connected to the accelerometer using a waveguide (Fig.1). To obtain information about the process the recorded vibroacoustic signal was subjected to time and frequency analysis. The spectra of vibration signals obtained for different time intervals from the moment of pulse occurrence and the records of effective signal values in different frequency bands were compared.

There is a sharp increase of the amplitude of the vibroacoustic signal at high values of the charging voltage, which, is associated with the intensification of the evaporation process. This helps to find rational modes of irradiation for electron-beam beam surface alloying, assuming on the one hand the maximum possible power supply, limited by the possibility of evaporation of the film with alloying components on the other.

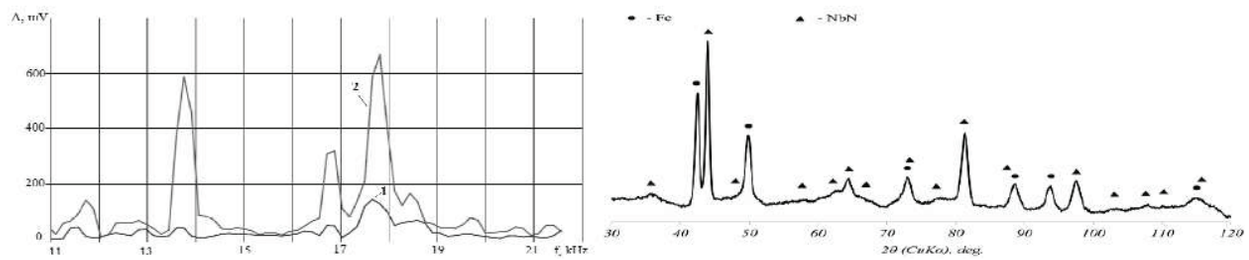

Fig.2. Comparison of signal vibroacoustic spectra in the frequency range of 11-22 kHz for the interval up to 10 msec (graph 1) and for the interval in the region of 15 msec (graph 2), diffraction pattern from the sample.

For a vibroacoustic signal with an irradiated nitrided plate coated with a alloying coating, after the 10th msec, there is a sharp increase in amplitude, which indicates the emergence of a process with high vibroacoustic activity. It can be argued that such activity is associated with the formation of nitride phase as a result of exothermic chemical reaction, which is in good agreement with the data of x-ray analysis (Fig 2).

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DIAGNOSTICS OF GAS CLUSTER ION BEAM FOR MATERIALS TREATMENT[1]

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In last decade it has been shown that the gas cluster ion beams (GCIB) can serve a unique tool for modern technological applications: surface polishing of the various materials down to subnanometer roughness, low-temperature formation of thin films, ultrashallow implantation etc. [1, 2]. For materials treatment the accelerated cluster ion beams with sizes from dozens up to thousands of particles (atom or molecule) per cluster and with energy of up to 30 keV are required.

The nature of the interaction of cluster ions with the solid is determined by their kinetic energy and sizes. It is known that cluster beams formed from a supersonic gas jet have a very broad size distribution, which is usually characterized by the mean cluster size [3]. The mass composition of the cluster beam depends on the parameters of the gas source (nozzle geometry, stagnation pressure and temperature) and can be estimated using empirical relationship [4]. However, the mass spectrum of the cluster ion beam can differ significantly from an initial neutral cluster beam due to cluster fragmentation upon their ionization. Therefore, it is necessary to control the mass composition of the cluster ion beam upon materials processing. To measure the mass spectra, time-of-flight diagnostics is used, when the cluster ion beam is modulated after it acceleration. This requires the use of additional electrodes and imposes restrictions on the measurement modes [5].

In this work, the original time-of-flight diagnostic technique based on modulation of ion flux at the stage of beam formation was used. As a source of the intense flux of neutral clusters, the supersonic gas jets of easily condensable Ar and weakly condensable N_2 after conical nozzles are utilized in experiments. Mass spectra of cluster ion beam at various stagnation pressure and ionization conditions are obtained. To determine the correct mean cluster sizes the measured distributions were approximated by lognormal distribution functions.

To determine the sputtering yields of the non-size-selected cluster ion beam, it was proposed to use the effective mean cluster size N_{eff}, which is determined from the convolution of the mass distribution function and gas cluster sputtering yields equation. It was demonstrated that the sputtering yields of SiO_2 for size-selected and non-size-selected argon cluster ion beams are similar by using the effective mean cluster size N_{eff}.

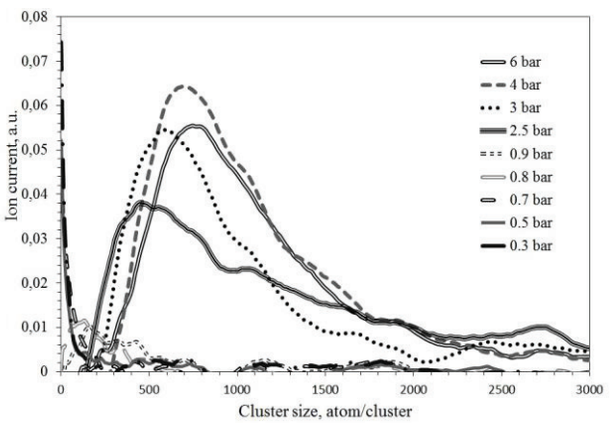

Fig. 1. Mass spectrum of Ar cluster ion beam at different stagnation pressures.

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OXIDATION RESISTANCE OF TITANIUM TREATED BY COMPRESSION PLASMA FLOWS

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Surface materials modification is a really important field of the modern materials science. The most widespread approach of the materials surfaces modification is connected to the influence of concentrated energy flows which are presented by electron beams, ion beams, lasers and plasma flows. A set of different structural transformations occur in the modified layers after the treatment. As a rule, the most applicable modes of the influences allow to improve the physical properties of the treated materials. For example, high cooling rate of the sub-surface layer after high-energy flows impact provides growth of the dispersed gains and, as a consequence, the mechanical properties such as hardness, tensile strength and others increase.

There are a lot practically important devices and constructions which work at elevated temperatures. So, the used materials for these ones should possess not only high physical properties, but and save their stable values at high temperatures. In the case when the material is suppose to use in air atmosphere, additional problem connected to high-temperature oxidation with oxides, oxinitrides, oxicarbides and others phases formation takes place. So, the physical properties of the material will degrade faster.

In the present work the problem of titanium oxidation resistance enhancement is solved. It is proposed to use compression plasma flows generated by a magnetoplasma compressor. The pulse duration of the plasma flow was about 100 μs and the absorbed energy density was chosen as a value that is enough for the titanium surface layer melting. After the treatment of titanium by compression plasma flows the samples were annealed in air atmosphere at the temperatures from 100 to 900 ºC. The change of surface morphology, microstructure and phase composition was revealed in the experiments.

It was found that compression plasma flows impact on the titanium samples results in dispersion structure formation and α-Ti(N) solid solution formation that influences on the oxidation process in the surface of titanium. During the oxidation the nitrogen-contained solid solution transforms into the α-Ti(N,O) solid solution gradually and the lattice parameters of the hexagonal close-packed structure increase. At 700 ºC the rutile modification of the titanium oxide TiO_2 starts growth, after annealing at 900 ºC the whole surface layer analized by X-ray diffraction method contains only rutile phase. At the same time oxide phase of titanium starts to form at 600 ºC in the case of titanium without plasma treatment.

To increase oxidation resistance it was proposed to from Ti-based alloys (Nb-Ti, Zr-Ti and Nb-Zr-Ti alloys) after combined treatment including the metal (Nb, Zr, Nb/Zr) deposition and the following compression plasma flows impact. It was shown that formation of binary and ternary Ti-based alloys with compression plasma flows provides increasing the temperature of oxide growth beginning and oxidation resistance of Ti phase. The mechanisms of oxidation stability treated titanium alloys are discussed in the work.

PLASMA-SURFACE INTERACTION: ION FLUX ON EMISSIVE SURFACE WITH DEBYE-SCALE EROSION TRENCHES

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The surface wall morphology in plasma devices can evolve with time due to a dynamic plasma-wall interaction that leads to drastic change of all plasma characteristics. For example, in Hall effect thrusters, plasma - wall interactions have been found to play a key role in the thruster operation and performance [1–2]. In Ref.[2], radially-symmetric surface modulations, at a larger characteristic length scale than the plasma sheath thickness were observed in the discharge plasma for an extensive time operation. The subject of this study is the plasma interaction with the emissive surface with a Debye length scale grooved topology. In 2D3V Particle-in-cell Monte Carlo collision simulations are compared with the experiment [3]. The discharge operation and secondary electron emission from the hBN plate are controlled by an electron beam from heated cathode. A change of the energy of beam electrons initiates the transition between different types of the wall sheath near an emissive plate with planar and grooved surfaces.

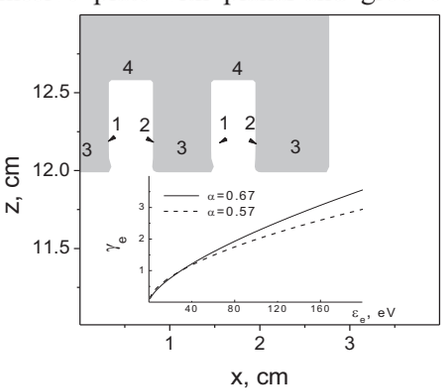

Figure 1. *Grooves geometry in simulation. 1- 4 show different fragments of grooves for calculation of floating potential. Insert shows secondary electron emission coefficient for hBN as function of electron energy.*

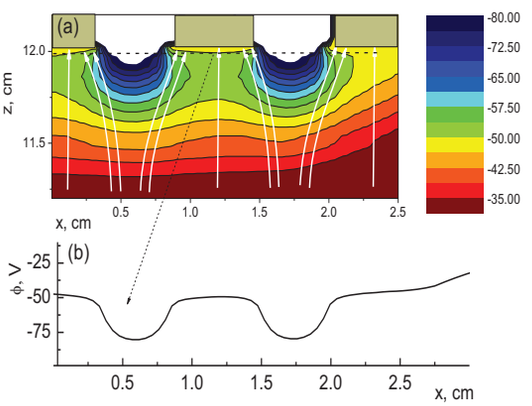

Figure 2*. (a) Spatial electrical potential distribution and (b) potential profile along a dashed line shown in (a), U=-150 V. White arrows show schematically the low energy electron trajectories.*

To understand the mechanism of sheath formation over the surface with complex topology in discharge plasma we use the model from Ref. [4]. The Boltzmann equations are solved to find the distribution functions for electrons and ions together with the Poisson equation for electrical potential. In Fig. 1, a part of calculation domain (x=0 is the axis of symmetry) with the grooves of 5 mm wide and 5 mm depth is shown. The beam electrons are emitted from the cathode surface and accelerated within the cathode sheath to the direction of grooved plate. These energetic electrons provide the secondary electron emission coefficient for hBN plate. The potential in Fig. 2 demonstrates a non-monotonic distribution with a strong gradient over x along the grooved surface. The potential distribution acts as a focusing lens on the low energy electrons and ion currents and redirects the electron current to the front surface and the ion current inside to the trenches. Both measured and calculated data exhibit a rapid transition from developed to collapsed sheath types at some critical voltage U_{cr}. The potential drop over the sheath diminishes approximately in 3-5 times for planar and grooved plates during transition, but it happens at very different voltages.

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PROGRESS IN HIGH INTENSITY, LOW ION ENERGY IMPLANTATION METHOD DEVELOPMENT[1]

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The report presents new results on the development of equipment and method of high-intensity implantation of metal ions, gases and semiconductors of low energy providing long rang effect of dopants penetration. The results of research and development of plasma-immersion systems for the ion beam formation with a current density of tens and hundreds of mA/cm^2, beam current exceeding 1 A with kilovolt accelerating voltages using different ion extraction and beam ballistic focusing systems are shown. A comparative analysis of various systems, their advantages and disadvantages depending on the purpose are presented.

The report presents the results of experimental studies of high-intensity implantation of aluminum, titanium and nitrogen ions into various structural materials. The possibility of ion doping of materials at depths of several tens and hundreds of micrometers is demonstrated. Data on changes in the elemental composition, microstructure and properties of various materials depending on the ion current density in the range from 10 to 500 mA/cm^2, ion energy, temperature implantation regime and irradiation fluence in the range from 10^{18} to 10^{22} ion/cm^2 are presented. The question of manifestation of the shock wave mechanism of the mass transfer of the introduced dopant at high-intensity ion implantation is discussed.

Based on the obtained experimental data analysis and the numerical simulation results the directions of further studies within the framework of the development of the physical and technological fundamentals of high-intensity implantation method of low-energy metal, semiconductor and gas ions are discussed.

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MODELING OF THE PHYSICOCHEMICAL INTERACTION OF REACTING COMPONENTS IN A MOLTEN POOL DURING LASER DEPOSITION OF POWDER

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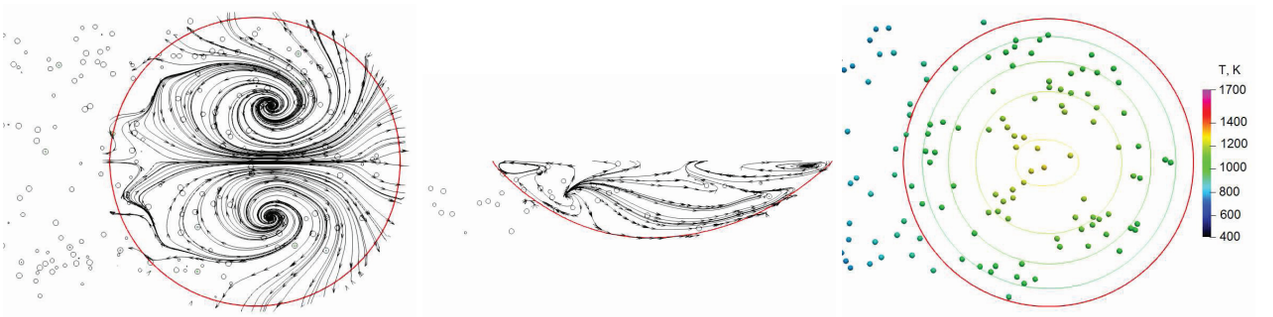
The problems of creating models of physicochemical, heat and mass transfer processes are discussed when using heterogeneous materials forming chemical compounds in gas-powder laser cladding. The local action of laser beam initiates metallurgical processes in the molten pool, which can be accompanied by interphase and chemical interaction between the reacting metallic (metal-metal) and nonmetallic (metal-non-metal) elements in solid or liquid states. The chemical composition of the resulting product depends on many parameters, the main of which are the rates of local heating and cooling. Mathematical modeling of the thermal state of the object and calculation of the phase composition (compounds of chemical elements) of final products is an actual problem for prediction the conditions for obtaining high-quality manufacture.

In the works of the authors [1-3], problems of mathematical modeling of self-propagating high-temperature synthesis (SHS) flowing in a mixture of reacting metal powders are discussed. Based on the analysis of state diagrams of binary systems, schemes for the formation and decomposition of intermetallic phases are proposed and a heterogeneous model of high-temperature synthesis of intermetallic compounds is constructed. In this paper, we describe the processes of heat and mass transfer in the presence of a two-component mixture of reactants in a molten pool induced by laser. Based on the heterogeneous SHS model [3], a model of interphase and chemical interaction between components has been developed, using the example of the nickel-aluminum (Ni-Al) system, which can form intermediate crystalline intermetallic phases. The aim of this work is to create a mathematical model that predicts the rate of exothermic reactions in particles added to the melt of the more refractory components of the powder mixture during laser cladding and also calculates an additional temperature increase in the molten pool, depending on the degree of chemical interaction between the reacting components. In Fig. 1 shows the current lines of liquid aluminum (a, b) and the temperature of the melt and single Ni particles (c) in the transverse and longitudinal sections of the molten pool induced by laser.



a b c

Fig. 1. Current lines in the molten pool of aluminum with particles layered with nickel aluminides.

Double particle radii are shown that characterize the linear dimensions of intermediate intermetallic phases formed on the surface of particles. The beam is scanned from left to right at a speed of 1 m/min, a power of 5 kW, a spot radius of 1 mm, a particle diameter of 20 μm, a flow particle rate of 0.05 g/min.

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BLISTERING FORMATION IN STAINLESS STEEL C12Cr18Ni10Ti AND PURE METALS MO AND W INDUCED BY LOW-ENERGY ALPHA-PARTICLES BOMBARDMENT[1]

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Structural materials of thermonuclear facilities, and first of all materials of the first wall and plasma face materials, should to possess high radiation resistance - they must remain operable under the influence of high temperatures and high fluxes of neutrons and charged particles [1]. Such materials include stainless steels, as the materials of the first wall, as well as molybdenum and tungsten, as possible materials of the divertor plates and plasma face materials. One of the important problems for such materials is the degradation of properties under the action of alpha particles, in particular, helium swelling, blistering and flaking. This work present results of study blisters formation and accumulation of helium in the structural steel 12Cr18Ni10Ti, molybdenum and tungsten under irradiation with low-energy alpha particles.

To study the changes in the surface structure under irradiation with low-energy alpha particles, samples of 12Cr18Ni10Ti steel, molybdenum and tungsten were prepared. The samples of steel, molybdenum and tungsten were plates with dimensions of 20 mm×2 mm and a thickness of 1 mm. Also samples of molybdenum, cut from a foil of 120 μm in thickness, 10 mm × 10 mm in size, were made. Irradiation was carried out on the low-energy channel of the accelerator DC-60 (source ECR channel) by double-charged helium ions with energy 22.5 keV/charge, i.e. the total energy of He ion was 45 keV. The fluence of the alpha particles was 1×10^{18} cm^{-2}, the irradiation temperature did not exceed 200 °C. The projected range of alpha particles with such energy, according to the calculations by SRIM program in steel, Mo and W is about 100 nm.

Surface structure studies were performed using a JEOL JSM-7500F scanning electron microscope equipped with an INCA Energy energy-dispersive microanalysis system to monitor the elemental composition of the samples. After investigations of the surface structure, were performed studies of helium accumulation in irradiated samples by using thermal desorption spectroscopy method.

The results of the studies are following:

Regardless of the type of crystal lattice and the type of material the irradiation with low-energy alpha-particles to high doses (> 5×10^{17} cm^{-2}) leads to the appearance of blisters which are helium filled pores on the exposed surface, see fig. 1 a,в,c. Despite the fact that blistering is observed for all studied materials, it has been shown that on the steel, in addition to blistering, there is also a flaking, i.e. significant part of gas-filled pores are open on the surface. A similar picture - flaking is also observed for tungsten, while for molybdenum there is practically no flaking, only single pores opened on the surface are observed.

Studies of the He release by the thermal desorption method have shown that for steel, molybdenum and tungsten, the first helium yield peak is observed in the low-temperature region (200-300 0C). This peak is associated with the release of helium from the blisters - helium filled pores located in the near-surface region. The amount of helium desorbed in this temperature range does not exceed 25% of total implanted helium. At higher temperatures, the number of helium yield peaks is different for different materials and is associated with the migration of complexes and single helium atoms to the surface.

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ION-BEAM NITRIDING OF STEEL 40X WITH A HIGH-INTENSITY ION BEAM[1]

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Modification of materials properties by the method of plasma-immersion ion implantation of nitrogen makes it possible to obtain a nitrided surface layer having high hardness and wear resistance, corrosion resistance [1, 2]. In work [3], the possibility of plasma-immersion formation of high-intensity repetitively - pulsed beams of low-energy ions with a current density in the range10^{-2}–1 A/cm^2 was first demonstrated. It is promising to use such beams for nitriding metals. The current density for high-intensity implantation is one of the main factors affecting the depth of nitrogen diffusion. The upper limit of accumulation of the implanted impurity is limited to 20 ... 40 atomic %, which corresponds to the range of fluences 10^{20} ... 10^{21} m^{-2}.

In this paper, we provide the results of experimental and theoretical studies of the regularities and features of the modification of steel 40X with high-intensity beams of low-energy nitrogen ions. The theoretical studies carried out are related to modeling a) of the temperature fields of the irradiated sample by the ion beam and heat transfer through the sample holder; b) of a diffusion-kinetic processes of mass transfer of implanted dopants in the sample, taking into account the sputtering of its surface by an ion beam, the formation of phases and radiation-stimulated diffusion of nitrogen into volume. In terms of nitriding parameters the differential equations for the thicknesses ε and γ 'of the layers are obtained.

The possibility of plasma-immersion formation of a high-intensity beam of nitrogen ions with a current of 0.6 A at ion energy of 1.2 keV is shown experimentally and numerically. Nonuniform distribution of the ion current density along the beam cross section was used to study the influence of the ion current density in the range from 0.1 A/cm^2 to 0.5 A/cm^2 on the formation of the crater by ion sputtering, on the depth of nitriding and change in the microstructure and properties of the modified layers on one sample. The paper presents the study of the effect of high-intensity, ultra-high-fluence implantation of low-energy nitrogen ions at temperatures of steel 40X samples in the range of 450-650°C. It is established, that the width of the nitrided layer, during 60 minutes, depends both on the temperature implantation regime and on the ion current density, the maximum depth of nitrogen penetration of 180 μm was observed at a target treatment temperature of 500° C.

The results of numerical simulation agree with the experiments and allow predicting the structure of surface layers when the geometry of the system and parameters of a high-intensity low-energy beam of nitrogen ions change.

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MECHANISMS OF SURFACE FORMATION UNDER PROCESSING BY COMPRESSION PLASMA FLOWS[1]

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The impact by intensive plasma flows with power density of 10^6 – 10^9 W/cm^2 is one of the most important material processing problem which is widely used to improve physical and chemical surface properties. It results in material hardening, roughness reducing, improving adhesion and mass transfer of substances for systems with covering; and also it is used for metal alloying.

It was shown in [1, 2] that under the action of compression plasma flows on matter, a wave-like relief of the surface is formed on the target surface (wavelength of 200–1000 μm). Thus, the wave-like relief of the surface is not always observed and is determined by the thermodynamic properties of the substance. The mechanisms of formation of the surface relief of the target under the action of compression plasma flows, which explain all the observed phenomena are now not detected.

In this work, the action of compression plasma flows on a metal target is studied theoretically in order to analyze the change in the relief of the treated material for the case of various treatment modes.

It is theoretically established that an inhomogeneous velocity field formed in the melt during its radial motion over the surface of the target leads to an increase in the elastic energy in the melt, which can be accompanied by the decomposition of the melt into separate fragments. If the elastic energy is sufficient to form a new surface, then we will observe the fragmentation of the melt. The decomposition of the melt will not be observed if the time during which the elastic energy necessary for decay is stored is less than the characteristic hydrodynamic energy. The hydrodynamic time is defined as the ratio of the characteristic spatial scale of the formed fragment to the sound speed - c_s.

The results of the experiment and numerical calculations were compared. The thinner ring breaks down into smaller fragments with the same difference in the velocities of the outer part of the melt and the inner one. As the velocity difference increases, the fragments become more elongated. This is in good agreement with the experimental data [1, 2].

Thus, theoretically, the phenomenon of the formation of a wave-like relief of the surface observed in the experiment was explained.

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SIMULATION OF SURFACE PROCESES ON SILICON IN $CF_4/O_2/H_2$ PLASMAS [1]

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The processing of thin films by the active particles in low-temperature plasma is widely used in microelectronic device production. Such particles are formed in the RF-discharge zone by the dissociation of parent gases. As for etching of silicon films they are usually pure gases CF_4, SF_6 or binary gas mixtures with O_2, H_2 and etc. However due to the complex multichannel nature of formation of active particles in glow discharge a surface phenomena at the RF-electrodes and wafer surface are insufficient understood. To provide a good optimization of etching process it is quite essential to study the surface phenomena. Some results obtained for silicon etching in $CF_4/O_2/H_2$ plasmas have shown that to obtain adequate results it is necessary to use a detail plasma-chemical kinetics with precise description of heat and mass transfer [1, 2].

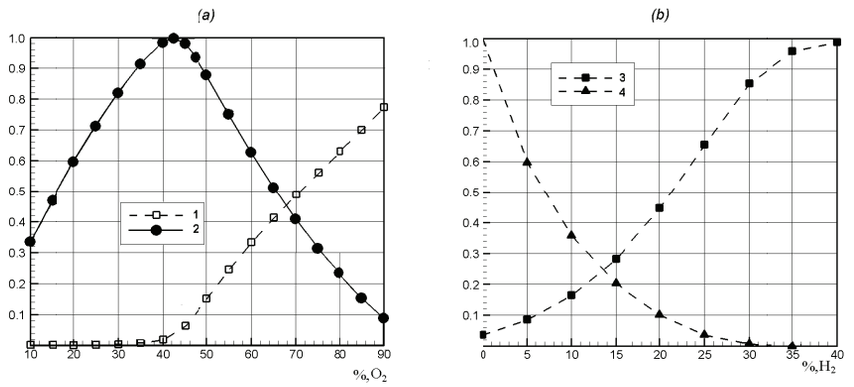

Fig.1. The fractions of silicon surface covered by adsorbed oxygen atoms O (1) and normalized etching rate (2) versus inlet O_2 addition in CF_4/O_2 mixture (a); the fractions of silicon surface covered by adsorbed radicals CF_2 (3) and normalized etching rate (4) versus inlet H_2 addition in CF_4/H_2 mixture (b). Parameters: p = 0.2 tor, Q = 200 cm^3/min.

In the frame of hydrodynamic approach the surface phenomena on silicon in $CF_4/O_2/H_2$ plasma was simulated. In calculations was used 2D mathematical model based on the equations of multicomponent physical-chemical hydrodynamics. The model of plasma-chemical kinetics in CF_4/O_2 mixture contained 16 gas-phase reactions of dissociation and recombination processes and 8 heterogeneous reactions on the wafer, which included 12 products - F, F_2, CF_2, CF_3, CF_4, C_2F_6, O, O_2, CO, CO_2, COF, COF_2 [1]. The model of plasma-chemical kinetics in CF_4/H_2 mixture involved 28 gas-phase and 6 heterogeneous reactions, which contained 11 components - F, F_2, CF_2, CF_3, CF_4, C_2F_6, H, H_2, HF, CHF_3, CH_2F_2 [2].

In CF_4/O_2 system the oxygen atoms replace fluorine atoms in CF_x radicals which set free the additional active particles that allows to increase the etching rate in several times. Starting with 40% of O_2 it is appeared the intensive passivation of silicon surface by oxygen atoms (see Fig.1, a). The fractions of silicon surface covered by radicals CF_2, CF_3 are less than 0.01 because of intensive reactions of CF_x radicals with atomic oxygen. The fluorine concentration in CF_4/H_2 system is considerably lower because of its consumption in the reaction with hydrogen to form HF. This system is characterized by higher coverage of silicon surface by CF_2 radicals compared to the CF_4/O_2 system (see Fig.1, b). On the wafer surface it is formed the adsorption layer of CF_2, which at 40 % of H_2 completely covers a silicon surface and stops the etching process. Starting with 40 % of H_2 the all silicon surface becomes passive because of intensive adsorption of radicals CF_2, CF_3. The addition of O_2 or H_2 component in CF_4 plasma allows efficiently to control an etching rate in the large range of parameters.

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FEATURES OF PLASTICITY NUCLEATION IN DEFORMED VANADIUM CRYSTALLITE UNDER IRRADIATION [1]

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Molecular dynamics simulation of the defect structure nucleation in deformed vanadium crystallites under irradiation was carried out taking into account their internal structure. The crystallites studied contained grain boundaries of different types. Periodic boundary conditions were simulated in two directions, and a shear load was applied in the third direction. Many-body potentials were used to describe the interatomic interaction in vanadium.

In the work, atomic mechanisms for the nucleation of plasticity in simulated crystallites under shear deformation were revealed. The features of the structural changes in the grain boundary region were studied. The behavior of crystallites deformed to values close to the limit of elasticity under irradiation was simulated by the generation of atomic displacement cascades. The energy of the primary knocked atoms, which generated atomic displacement cascades, was varied from 5 to 10 keV. It was shown that the grain boundaries prevent the propagation of atomic displacement cascades and accumulate a large fraction of radiation defects in their region. It was found that atomic displacement cascades in elastically deformed crystallites can lead not only to the formation of radiation defects, but also cause plasticity. The dependence of the threshold energy of the primary knocked atom on the magnitude of the deformation at which plasticity is generated in the crystallite is calculated. It is shown that the nucleation of plasticity in crystallite is associated with shock waves. These waves are formed in the region of atomic displacement cascade development. Their generation is due to the high-speed thermal expansion of the region in which atomic displacement cascade develops.

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FORMATION OF HOLLOW CERAMIC PARTICLES IN PLASMA FLOW[1]

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Powders consisting of hollow microspheres with a diameter of tens of microns have found a wide application in various industries [1]. It is necessary to develop the adequate mathematical models describing the processes of motion, heat, melt of the precursor particles, and also the formation of the morphology of hollow particles to optimize the production of hollow microspheres [2-4]. In the manufacturing practice, the various technologies for obtaining the ceramic hollow microspheres are known [5-8].

The results of mathematical modeling of the formation of hollow spheres during the heating and melting of porous silica particles in a low-temperature plasma flow are presented.

The regularities of the evolution of the diameter and thickness of shell of the microsphere in the range of the precursor particle diameter of (50-150) μm and their porosity of (0.2-0.6) on the base of the proposed model of the process that takes into account the partial encapsulation of gas have been obtained and analyzed. To describe the formation of hollow microspheres in a one-dimensional flow of high-temperature gas, the following processes are considered: particle dynamics, heating and melting of particles, and the formation of hollow microspheres.

At modeling the processes the following assumptions are used:

- gas flow is one-dimensional, its temperatures and velocity are specified, they are constant throughout the length of the high-temperature impact zone;
- the pressure in the gas flow is constant and equals to atmospheric pressure;
- the porous particle of the precursor has shape close to spherical;
- at heating the porous particle it is assumed that the instantaneous temperature equalization in the volume of the particle occurs.

It is assumed that the process of formation of hollow microspheres occurs in four stages: heating of the particle from the initial temperature to the melting temperature of silica; formation of the primary outer shell; formation of the final shell; amorphization of the shell at cooling the particles after escaping the high-temperature zone.

In contrast to the known models, the proposed physical and mathematical model of the formation of hollow microspheres at heating and melting the porous silica particles (precursor) in the flow of low-temperature plasma takes into account the partial encapsulation of the gas during the formation of the shell from the molten metal. The results of the calculations has shown that the value of the outer and inner diameter of the liquid film, its thickness are mainly determined during the melting process and slightly change with further heating. It has been shown that values of the final particle diameter are very different in cases of open and closed pores. It has been established that for a small number of closed pores the value of the final particle diameter insignificantly differs from the initial diameter of the precursor particle.

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IDENTIFICATION OF STRUCTURAL MODIFICATION OF PT EQUIVALENT AFTER NEUTRON AND ION IRRADIATION BY FIELD ION MICROSCOPY

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Investigating interaction mechanisms of accelerated particles with matter and studying the atomic rearrangement and, as a consequence, formation of crystal lattice defects and changing the phase state of the material are important tasks in radiation physics of solids.

This work is devoted to studying the spatial distribution of radiation damages, particularly vacancies and their complexes in the bulk fcc metals exposed to different irradiation (by neutron and ion beams).

Imitation of the neutron irradiation of high fluence with the help of positively charged ion beams allows one to solve the problem of analog simulation of radiation generated on one setup when replacing it by the radiation generated on another setup.

The main objective of this work was to establish the adequacy of the effects of different forms of radiation affecting on one and the same material (Pt) when analyzing radiation damage of the same type. For this purpose, using the methods of field ion microscopy (FIM), we have studied radiation defects on an atomically clean surface and within a subsurface volume of platinum that are created as a result of neutron and ion_beam bombardment ($E > 0.1$ MeV and $E = 30$ KeV, respectively).

FIM allows precise studying to be carried out of the changes in the real crystal lattice structure of metals and alloys occurring as a result of irradiation on the atomic scale. At the same time, this method allows one to analyze the structure in the bulk of the sample by means of consecutive removal of surface atoms by the electric field.

It is shown that the interaction of fast neutrons (E > 0, 1 MeV, F = 6,7 • 10^{21} m^{-2}, F = 3,5 • 10^{22} m^{-2}) with platinum leads to certain radiation damages in the volume of Pt. Such defects occur under irradiation by beams of Ar^+ ions (E = 30 keV, F = 6.7 • 10^{20} m^{-2}). The latter are observed at a depth of about 1.5–2 nm under the irradiated surface of Pt. Thus, we have carried out modeling of neutron impact with matter when replacing the neutron beam by an ion beam that causes the same radiation damage in the bulk of the material.

REGULARITIES OF IMPLANTATION Al, Cr ATOMS OF TWO-COMPONENT FILMS TO POLYCRYSTALLINE SUBSTRUCTION FROM ZIRCONIA UNDER IRRADIATION OF IONS OF Ar AND Xe WITH ENERGY TO 10 keV

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Modification of the surface of the substrate occurs as a result of irradiation by a beam of ions onto the substrate. On the substrate vacuum-deposited one or several layers of films, the atoms of which are supposed to be implanted. To increase the efficiency of the implantation of atoms from sputtered films, it is necessary to reduce their sputtering speed and at the same time to strive to increase the depth of penetration into the substrate, for example, due to radiation-stimulated processes and physicochemical interaction [1-3].

The purpose of this work was to study the effect of energy and radiation dose on the efficiency of modifying the near-surface layer of a polycrystalline zirconium substrate with atoms of two-component films, which were introduced by the method of ionic mixing under the influence of Ar + and Xe + ion beams with a broad energy spectrum.

The surfaces of the samples were modified on the ILUR-03 setup [4] with a discharge chamber that allowed us to irradiate cylindrical samples using a radial beam of ions with a wide energy spectrum of 0.5–5.0 keV (mean energy ⟨E⟩ ≈ 3.0 keV) to a dose of (5−10) × 10^{18} ion cm−2, and three magnetrons that allow the deposition of thin films of metals in an inert atmosphere (the residual gas pressure is no lower than 10−4 Pa). Two-layer films were sprayed onto the substrate to perform two-component doping, each layer being sputtered with one kind of atoms.

It is shown that the penetration depth X_m is linearly dependent on the radiation dose in the range of values Φ=(0.5–10)10^{18} ion/cm^2, and the distribution of embedded atoms films $C(x)$ has multiple maxima.The first maximum corresponds to the depth of the run the ions with an mean energy of $x(<E>)$. The second maximum is in the range of the ions with the maximum energy x (E_{max}), and the appearance of the third and subsequent maxima is due to radiation-stimulated processes, including the distribution of mechanical stresses by depth, by the interaction of interstitial atoms with each other and by the atoms of the substrate, by the presence and distribution of point defects).

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OPERATION PARAMETRS OF MAGNETRON DIODE FOR HIGH-RATE DEPOSITON OF ALUMINUM FILMS[1]

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Magnetron sputtering system is a versatile technique for thin films deposition. But, there is a crucial drawback which consists in low-deposition rates in comparing with resistive and arc evaporation. Thus, the different approaches are applied to increase of films deposition productivity. The most perspective way is based on the combination of sputtering and sublimation (evaporation) of metal target [1-3]. However, it can not be used for Al target sputtering due to low melting temperature (933.3 K) and high ability to form intermetallic compound with other metals. So, the other way to increase of deposition rate of Al films should be considered. Moreover, Al films are intensively used as thermal-control and radio-technical coatings and high attention is devoted to improve their functional characteristics [4]. In this case, the purity of deposited films should be increased that is not a simple task for sputtering method, whereas inert gases are usually used to form and sustain a plasma discharge.

In our work we investigated the operation parameters of magnetron diodes with 90 mm-Al target to obtain the deposition mode of aluminium films with high rates. Special for this, the minimum operation pressure necessary for stable sputtering of the aluminum target was determined by variation of the electric supply circuit and their parameters (discharge power averaged over pulse period and pulse parameters) and by changing of geometry of the sputtering system (magnetic field configuration and the quantity of sputtered cathodes). Moreover, thermal fluxes reached the substrate were calculated that needs to prevent over-heating of aluminum target and possible melting in the case of the use of the magnetron diode with indirect cooling of the metal target. Subsequently, optical-emission spectroscopy was used to identify plasma discharge characteristics and the dependence between operation parameters of the magnetron diode and composition of discharge plasma. Then, absolute deposition rates of Al films were measured and specific deposition rates (averaged on discharge power and target area) were calculated.

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THERMODYNAMIC DISTURBANCES CONTROL DURING FLOW OF VISCOUS FLUID THROUGH A PIPELINE WITH A CONICAL SECTION

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The study of processes occurring during gas flow in complex configuration pipelines is of definite scientific and practical interest. So, in 2014 a full-scale experiment was conducted by specialists of "Giprogaz-center". It was found that during the filling of main gas pipeline sections the anomalous dead-end branches heating at the crane nodes took place. The heating of the gas took place when a conical (confuser) section was used in the dead-end branch.

In this paper it is planned to analyze the thermodynamic characteristics of the gas flow in a pipeline with a confuser section and also to study the influence of the confuser geometry on thermodynamic characteristics for further evaluation of the effect of their change on the contribution to thermal processes.

The problem of nonisothermal motion of a gas in a channel with a confuser section was considered. The formulation of the problem is stationary.

Calculations were made in the software package Ansys Fluent. It was used various modifications of k-ε (where k and ε are the kinetic energy and the dissipation of the turbulence energy, respectively) of the turbulence model (k-ε standard, k-ε RNG, k-ε realizable) as one of the most popular on the present day and included in all packages that are used in the calculation of hydrodynamics.

The pipe configuration can be described as followed: a diameter of the inlet section is 0.2 m, the length of the confusor section is 1 meter. Diameters of the outlet section are various and depend on the tangent β (from 2/200 to 8/200) of the angle of inclination of the surface of the confusion section to the pipeline axis.

Numerical experiments was based on Kawamura's research data. The interval of Reynolds number (Re) was from 1 up to 2. The difference between wall and fluid temperatures was 5 ^{o}C.

The pipeline model is shown in the figure 1.

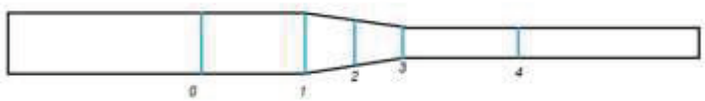

Fig. 1 Pipeline with a confuser section. 0, 1, 2, 3, 4 – investigated cross-sections.

Some thermodynamic flow parameters were calculated.

Such parameters as the flow velocity and its pulsation components, the scale of turbulence, the kinetic energy of turbulence are calculated with different accuracy. It can be noticed that the standard and RNG models have relatively similar results, which differs from the results of the realizable model.

It was also decided to calculate the Nusselt number (Nu) values and compare the obtained result with the data of known experiments in order to obtain an understanding of turbulence models predictable possibilities in case of thermal parameters calculating.

But it necessary to notice that it has been found by calculations that at larger inclinations angles of the generatrix of the confuser surface to the flow axis (β> 8/200), the two-parameter models made an appreciable error in the calculation of the flow structure and its integral parameters (Nusselt's criterion, hydraulic resistance) due to the dominance of the anisotropy mechanisms. This circumstance allow to say that it should be made some corrections of models are used in this investigation when predicting the vortex instability in channels of complex geometry with (or without) accounting of heat exchange, to make this models better in predicting of the anisotropic properties of turbulent flows.

THE WAVE MECHANISM OF HEAT TRANSFER IN FINITE SAMPLES IRRADIATED BY SHORT PULSE LASERS

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In the works [1,2] the solution of hyperbolic heat conduction equation with zero boundary conditions for the flow under the condition of a short pulsed laser beam disturbance on the sample was considered. It was assumed that the temperature gradient is associated with the heat flux in the form of Fourier law, which does not take into account the inertia of the heat transfer. In the framework of extended irreversible thermodynamics the flow is determined by the relaxation Cattaneo equation, and parabolic boundary conditions are not suitable. In contrast to [1] in this work, we first solved the one-dimensional hyperbolic equation for the flow on a finite interval. Next, using the equation of energy conservation, we proceed to determine the distribution of the temperature field inside the sample. The heat source function has been selected in the form of [2]. Thermophysical characteristics of the medium were assumed to be constant. To simplify the solution of the problem we introduced dimensionless time t/τ_p, coordinate $x/(a\cdot\tau_p)^{1/2}$, as well as dimensionless flow and temperature (here τ_p is the relaxation time of the heat flow, a – is thermal diffusivity). The analytical solution of the generalized heat flow equation is derived by using Green's function method. The results of modeling the depth distribution of dimensionless heat flux $J(x)$ and dimensionless temperature θ for different observation times are presented in Fig.1. Several series of peaks indicate the propagation (t = 0.2, 0.85, 1.0) and reflection (t = 1.1, 1.65) of temperature wave.

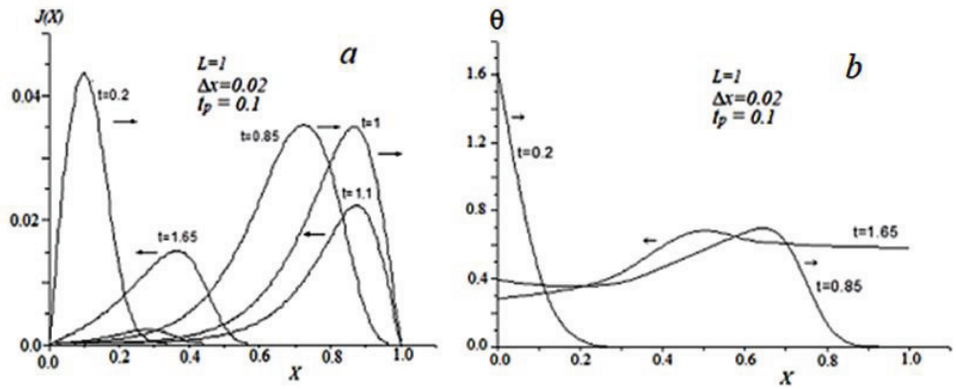

Fig. 1. The distribution in depth of the sample: *a* - the dimensionless heat flux *J(x)*, *b* - the dimensionless temperature θ for different observation times *t*. *L* – the sample thickness; Δ*x*, *tp* - parameters of a laser beam

The temperature distributions calculated in this paper are compared with the results of [1]. The character of change of temperature fields qualitatively and quantitatively different from the behavior in [1] at times comparable to the time of relaxation of the heat flow.

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STUDY OF THE SURFACE RELIEF, STRUCTURE AND PHASE COMPOSITION OF THE SILUMIN COMPOSITE LAYER OBTAINED BY THE METHOD OF ELECTRIC EXPLOSION ALLOYING BY AL-Y_2O_3 SYSTEM [1]

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The promising methods permitting to harden and protect the surface by the modification of its properties are the concentrated flows of energy such as the electron beam [1], laser radiation [2] and plasma effects [3].

The purpose of the research is the study of the surface relief, structure and phase composition of the silumin composite surface layer obtained by the method of electric explosion alloying by Al-Y_2O_3 system.

The electric explosion alloying was done using the laboratory charge-pulsed electric explosion unit EVU 60/10.

The test material was the alloy of aluminium with silicon $AlSi_{10}CuMg_2Ni$. The chemical composition of the sample under test with the main elements of Al – 84.88% and Si – 11.10% was determined by the method of X – ray spectrum analysis with the energy dispersion detector of micro X – ray spectrum analysis INCAx – act. The main alloying elements are Cu – 2.19%, Ni – 0.092%, Mg – 0.58%.

The test samples had the dimensions of 20×20×20 mm^3 and they were oriented perpendicular to the axis of the plasma jet.

The surface treatment of silumin samples was done according to two optimal regimes differing in the discharge voltage and the masses of the powder weighed samples.

The analysis of experimental results shows that the top layer in the samples after electric explosion alloying is a high porous one independent of the regime of treatment. In the statistical analysis of the image the maximum of the pores' depth was detected with the values of the order of 1500 nm for the regime 1 and 500 nm for the regime 2. It should be noted that the surface of the samples treated according to the regime 2 is characterized by the less quantity and pores' depth than in the case of the treatment according to the regime 1. It may be explained by the fact that the particles of the plasma jet distributed more uniformly having less energy by reason of the larger mass of the weighed sample of the sprayed powder and the less energy effect.

By TEM methods of thin foils it is determined that the electric explosion alloying is accompanied by the high speed cooling of the modified layer with the result that the structure of the cellular crystallization of aluminium is formed in the top layer. The size of the crystallization cells varies within 200 – 450 nm. The interlayers of the second phase locate along the boundaries of the cells. By the methods of micro – X – spectrum analysis it is determined that the interlayers are formed by the atoms of silicon and yttrium.

It should be noted that the concentration of oxygen atoms is low that may be indirectly indicative of the absence of the oxide phase at the boundaries of the crystallization cells. The cell dimentions vary within 150 – 300 nm. The microdiffraction analysis showed that the cells were the solid solution based on alumiinium. The particles of the second phase are revealed in the volume of the cells by the dark field analysis method; the dimensions of the cells vary within the units of nanometers.

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ION NITRIDING WITH DIFFERENT TEMPERATURE OF MARTENSITIC AND AUSTENITIC STEELS AFTER SPD

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Nowadays, new structural materials with high mechanical properties and technological characteristics are being developed for machine parts and mechanisms [1]. One of the promising directions is severe plastic deformation (SPD) [2]. Despite the high physical and mechanical properties of structural materials after SPD, their surface is subject to wear. Ion nitriding is often used to improve the wear resistance of machine parts [3,4]. However, ion nitriding is carried out at high temperatures (550-650° C) and long process time (up to 36 hours). Long exposure at high temperature leads to degradation of structure resulting from the SPD [1]. Therefore, to prevent grain growth, ion nitriding should be carried out at low temperatures (350-450° C).

The aim of this work is to investigate the influence of ion nitriding temperature on the structure, phase composition and mechanical properties of the surface layer of martensitic and austenitic steels after SPD.

SPD of steel samples was carried out by the method of plastic deformation by torsion at a temperature of 300° C under a pressure of P = 6 GPa, with a number of turns n = 10. Low-temperature ion nitriding was carried out in the vacuum setup ELU-5M. The processing time was 4 hours at a temperature of 450-550 ° C. As a working gas, a mixture of argon and hydrogen was used at a pressure of 150 Pa.

As a result of the study, it was found that ion nitriding at 450° C for 4 hours makes it possible to increase the surface microhardness of SPD samples to 1.25 for steel 13Kh11N2V2MF-Sh and 1.15 times for 12Kh18N10T steel. Increasing the treatment temperature to 500 ° C leads to increasing of growth kinetics of a nitrided layer and decreasing of core material hardness. It has been proved that the thickness of diffusion layer formed after 4 hours of ion nitriding at 450° C is larger on SPD steels, as compared to steels with a coarse-grained structure. So, the thickness of the hardened layer is 80 microns and 150 microns for 12Kh18N10T steel, 25 microns and 65 microns for 13Kh11N2V2MF-Sh steel, respectively for the coarse-grained and SPD states.

It was found that after 4 hours of ion nitriding at 550° C nitride layer that consists of ε and γ'-phases is absent on surface of all samples, both coarse grained and SPD.

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FORMATION OF IRON BORIDES FROM REACTION DAUBS UNDER THE INFLUENCE OF AN ELECTRON BEAM [1]

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This work is devoted to the development of new methods for the formation of hardening coatings based on iron borides. The main idea of the work is to modeling the synthesis of iron borides from reactive daubs on carbon steels.

One of the main tasks is to determine the optimum conditions for synthesis, namely, the formation temperature at different pressures in the chamber. It is known that at a pressure of 10^5 Pa, the interaction of Fe2O3 with various borating components (B2O3, B4C, B) begins at temperatures of 1500-1600 K, and at a pressure of 10^{-2}-10^{-3} Pa, the temperature decreases to 800 K (Fig. 1a). A sequence of chemical transformations occurring in the synthesis of borides, namely, "oxides → carbides → borides lower → higher borides" was established. Thermal properties and the nature of dissociation of borides Fe2B and FeB were modelled, depending on the total pressure in the system. Using the Terra program, thermodynamic modeling was performed in the range 373 - 1873 K for the total system pressure in the range of 10^5 -10^{-3} Pa. In the present study, we have investigated the formation of borides at a pressure of 10^{-3} Pa. This allowed synthesizing the reinforcing layer on the surface of steel St3 without melting the sample.

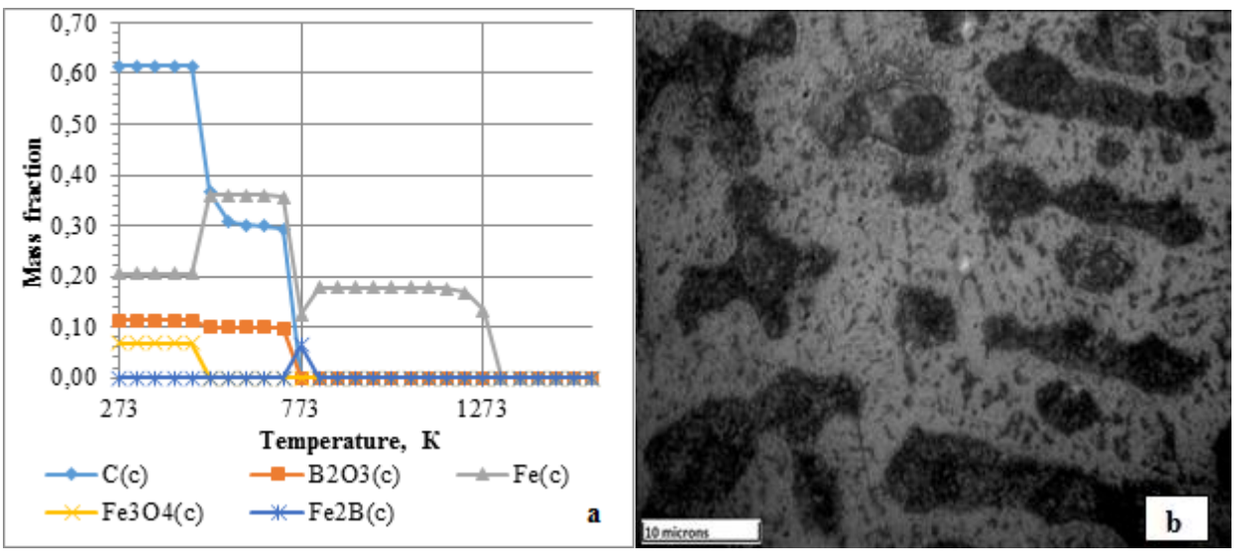

Figure.1. a – the condensed phase composition of Fe2B (4Fe:2B:6C: 3O2) at a pressure of 10^{-3} Pa; b – the structure of the layer.

In the experiment we used steel samples (St 3) in the form of cylinders with a diameter of 15 mm and a height of 7 mm. Reaction daubs of various stoichiometric compositions of Fe2O3: 3B: 3C, Fe2O3: 2B: 3C were deposited on the surface of the sample. Power density of the electron beam W = 5.7×10^2 W/mm^2 (electron beam diameter d = 1 mm), processing time 1-3 min. When the highly concentrated energy flows are exposed on the reaction daub, the SHS process is initiated. As a result of SHS, solid combustion products are formed, in particular, iron borides. After the exposure of the electron beam ends, the crystallization process begins, as a result of which a dendritic-like structure of the layer is formed (Fig. 1, b). The thickness of the layer reaches 250 μm. Investigation of the microhardness of the obtained layers shows that FeB borides have the greatest hardness, their microhardness is 1200-1500 MPa on the average. The microhardness of Fe2B borides is 1100-1300 MPa.

[1] This work was supported by the framework of the project of fundamental research programs of the SB RAS №0336-2016-0005

CALCULATED AND EXPERIMENTAL STUDIES AT CRITICAL FACILITY IN VIEW OF DEVELOPMENT OF A TECHNOLOGY FOR NEUTRON TRANSMUTATION DOPING OF A LARGE SIZE SILICON SPECIMEN IN WWR-K REACTOR

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Neutron transmutation doping of semiconductors, including silicon, is one of the most urgent and perspective radiation technologies. In connection with the rapid development of electronic industry and "green" technologies, the demand for semiconductors increases every year. Doping of semiconductors by neutron transmutation method has several advantages in comparison with industrial methods, therefore this method is more preferable.

Earlier in the Institute of Nuclear Physics, using WWR-K research reactor were carried out studies to develop the technology of neutron-transmutation doping of silicon ingots with a diameter of 6 inches and a height of 280 mm. As a result, were obtained silicon ingots with an uneven electrical resistivity at the level of ~6%.

In 2017, together with the Chiyoda Technol Corporation, (Japan) were started the activities to develop a technology of neutron-transmutation doping of large sized silicon with a diameter of more than 12 inches and a height of up to 500 mm for the WWR-K reactor. The main goal is to achieve an irregularity electrical resistivity in terms of the volume of the silicon ingot at the level of 3-4 %.

At the first stage, were performed the works on the formation of the thermal neutron field in irradiation device. The object of investigation is a silicon ingot with a diameter of 6 inches and a height of 500 mm. For reduction of the height irregularity are used neutron absorbers arranged along the height of the ingot. For reduction of the radial irregularity, the ingot is rotated about the central axis. The development of irradiation device with a neutron absorber and the corresponding neutron measurements were performed at the critical facility. The silicon ingot is modeled by a block of pure aluminium. Calculated studies are carried out using a three-dimensional software program MCU-REA that implements the Monte Carlo method.

At the second stage it is planned to perform the works on the WWR-K reactor to test the developed technology with the further irradiation of a silicon ingot.

In the present paper there are the results of investigations performed on the critical facility.

MODIFICATION OF STAINLESS STEEL BY LOW-ENERGY FOCUSED NITROGEN ION BEAM[1]

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The results of experiments on the modification of stainless steel 12Kh18N10T (SUS321 analog) by a beam of nitrogen ions extracted from the gas plasma of a PINK-P plasma source [1] based on a non-self-sustained arc discharge with a thermionic cathode are presented. The extraction and focusing of the ion beam was carried out across a grid electrode of a given curvature [2]. A ballistic-focused beam of nitrogen ions is formed by applying a pulsed negative electric bias to the grid electrode and specimen, which are under the same potential.

All experiments were carried out on a specially created laboratory facility whose simplified scheme is shown in Fig. 1.

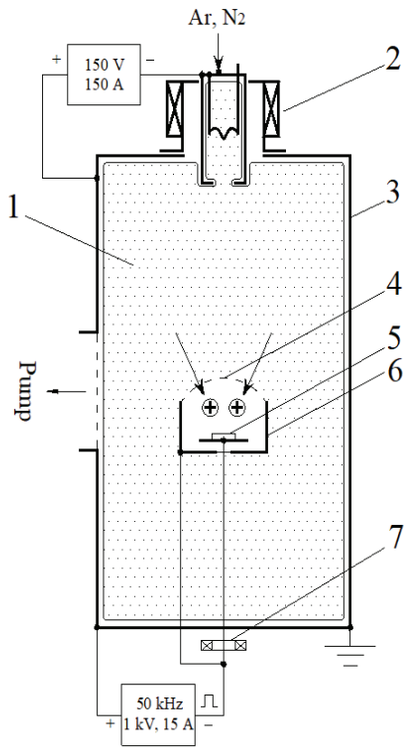

Fig. 1. The simplified scheme of the experiments: *1*-plasma, *2*-PINK-P plasma source, *3*-vacuum chamber, *4*-grid electrode, *5*-specimen, *6*- ion beam forming system, *7*-Rogowski coil.

Experiments have shown that after treatment a layer with increased hardness is formed on the surface of the stainless steel. Measurements of the microhardness on the cross section of specimen also revealed a layer with a gradiently varying hardness. Besides, the results of tribological and x-ray diffraction measurements of specimen treated in this system for 1 hour are presented.

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[1] The work was supported by the Russian Science Foundation (project No. 17-19-01169)

AISI 5140 STEEL NITRIDING IN A PLASMA OF A NON-SELF-SUSTAINING ARC DISCHARGE WITH A THERMIONIC CATHODE UNDER THE PULSE ACTION OF IONS[1]

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The results of experiments of AISI 5140 steel nitriding in a plasma of a non-self-sustained arc discharge with a thermionic cathode [1] in a pure nitrogen atmosphere with pulsed (50 kHz) low-energy (up to 1 kV) ionic action are described. The main task of the experiments was to investigate of the effect of ion energy (pulse amplitude) and ion current density on the thickness of the nitride layer formed on the surface and the diffusion nitrogen saturation layer over the depth of specimens. A set of experiments where the specimen temperature and the process time remained unchanged was performed to determine the effect of ion energy and the ion current density on the efficiency of nitriding,

All experiments were carried out on an "COMPLEX" automated vacuum system [2]. Simplified scheme of experiments is shown in Fig. 1.

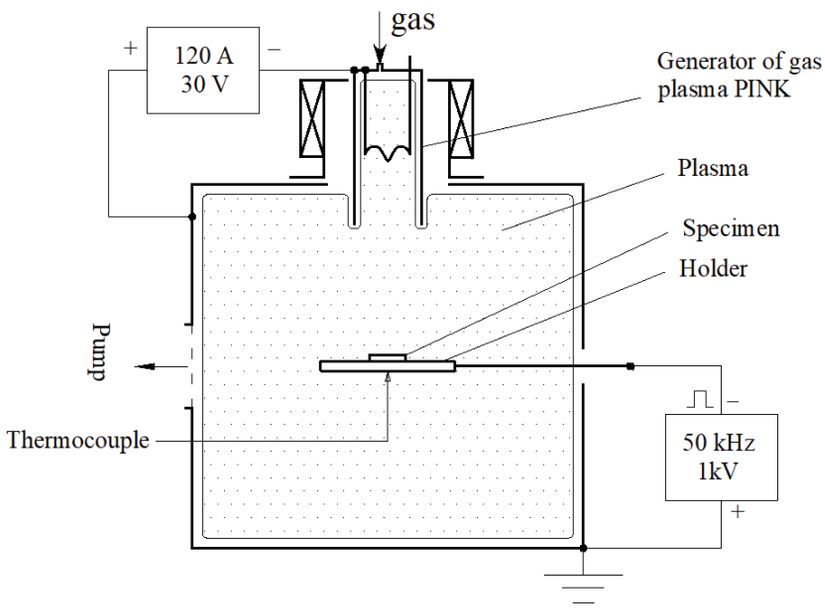

Fig. 1. The simplified scheme of the experiments.

It is shown that the microhardness of this steel after nitriding at a temperature of 500° C can increase almost 3 times (from 3 GPa to 8 GPa) with the formation of a modified layer with a depth of up to 150 microns per hour. It is shown that the parameters of the pulsed ionic effect affect the shape of the microhardness distribution profile on the depth and the efficiency of ion etching.

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[1] The work was supported by the Russian Science Foundation (project No. 14-29-00091)

MODIFICATION OF VANADIUM BORIDES THE SURFACE OF ALLOY TOOL STEEL T31507 POWERFUL ELECTRON BEAMS IN A VACUUM

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Properties of steel T31507 cause features of application of this material. Tool alloy steel T31507 is used to create critical parts, for example, the manufacture of cutting and measuring tools, for which the warping of the mirror during hardening is unacceptable, as well as for cylindrical, disk and modular cutters, as well as for cold-heading dies and tooling punches.

The report considers the features of surface hardening of T31507 steel under the influence of high-power electron beams due to hardening and formation of layers based on vanadium borides (VB_2, V_3B_4, VB).

First, thermodynamically researched (program complex TERRA (TERRA and TRIANGLE interfaces)) the interaction of vanadium oxide with carbon and boron under equilibrium conditions at a pressure in the range from 10^{-2} to 10^{-4} Pa. The process of self-propagating high-temperature synthesis (SHS) of vanadium borides was simulated under conditions of adiabatic expansion.

Further, it has been simulated the interaction of the reaction mixture V_2O_3:B:C with surface of carbon steel T31507 for the formation of a composite coating to a depth of 5-150 microns.

The calculations showed that the boride VB_2 with the use of a stoichiometric mixture of V_2O_3-B-C can not be obtained due to the formation of iron borides Fe_2B, FeB (the interaction with the metal base) and borides of alloying elements (CrB_2, WB, MnB_2). The introduction of excess amounts of boron and carbon allowed to choose the optimal compositions for the production of composite layers with a maximum yield of borides (Fig.1).

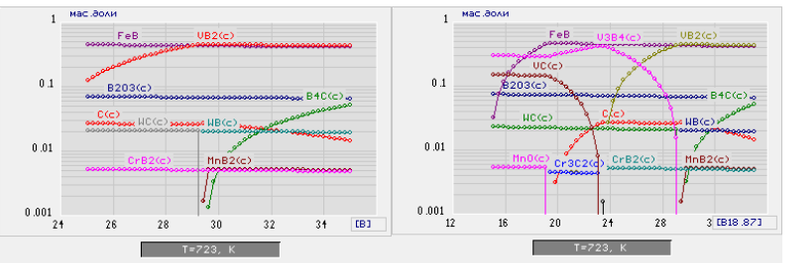

Fig. 1. An exit of the condensed phases at synthesis of VB_2 boride on T31507 steel ($P=10^{-3}$ Pas) in a composite thickness: a - 50 microns; b - 100 microns

Synthesis of vanadium borides was carried out on the surface of T31507 die steel. The samples were prepared by applying a coating on the pre-prepared surface of the steel. The composition of the coating was 1:1 by volume mixture of oxide V_2O_3, boron amorphous and carbon, as well as organic binder – a solution of 1:10 BF-6 glue in acetone. Electron heating by a continuous beam was carried out for 1-3 minutes at a specific power $W= 5{,}7\times10^2$ W/mm^2 (electron beam diameter d=1 mm). The residual pressure in the vacuum chamber did not exceed $2x10^{-3}$ Pa.

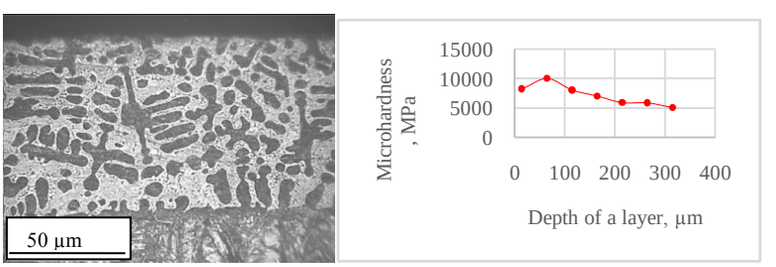

Fig. 2. Structure and a microhardness of a layer on the basis of VB_2 on T31507 steel (the continuous electron beam)

When measuring the microhardness of vanadium boride layers in steps of 30-50 microns, an uneven distribution of its thickness was found (Fig. 2). However, in all the samples studied there was a natural distribution of microhardness depending on the thickness of the layer. Some very rare inclusions have HV≈15,000 MPa and are located in the near-surface zones of the layer. Layers are characterized by the most complex disordered structure. The increase in the microhardness of the substrate to HV≈5000 MPa is explained by the fact that it has undergone hardening as a result of exposure to an electron beam.

ION-BEAM CHEMICAL-THERMAL TREATMENT OF ALUMINUM [1]

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The experimental results on the chemical-thermal treatment with nitrogen ions of aluminum alloy A7 are presented. The treatment of test specimens by an ion beam produsedby a source based on a non-self-sustaining glow discharge with a hollow cathode was performed. Specimens were placed in a plasma generated by a source with a combined thermionic and hollow cathode and simultaneously treated by an accelerated ions and gas neutrals (fig. 1). Negative bias to the specimens was not applied. This allowed to treat of dielectric surfaces without the formation of cathode spots on them. The charge on the surface was compensated by electrons from the initial gas plasma of PINK plasmagenerator. The temperature of the specimens was maintained independently of the ion beam energy and current by means of an active thermal screen. The screen temperature was set by a negative electrical bias and PINK discharge current. The possibility of forming a nitride film on the aluminum surface by this method has been shown.

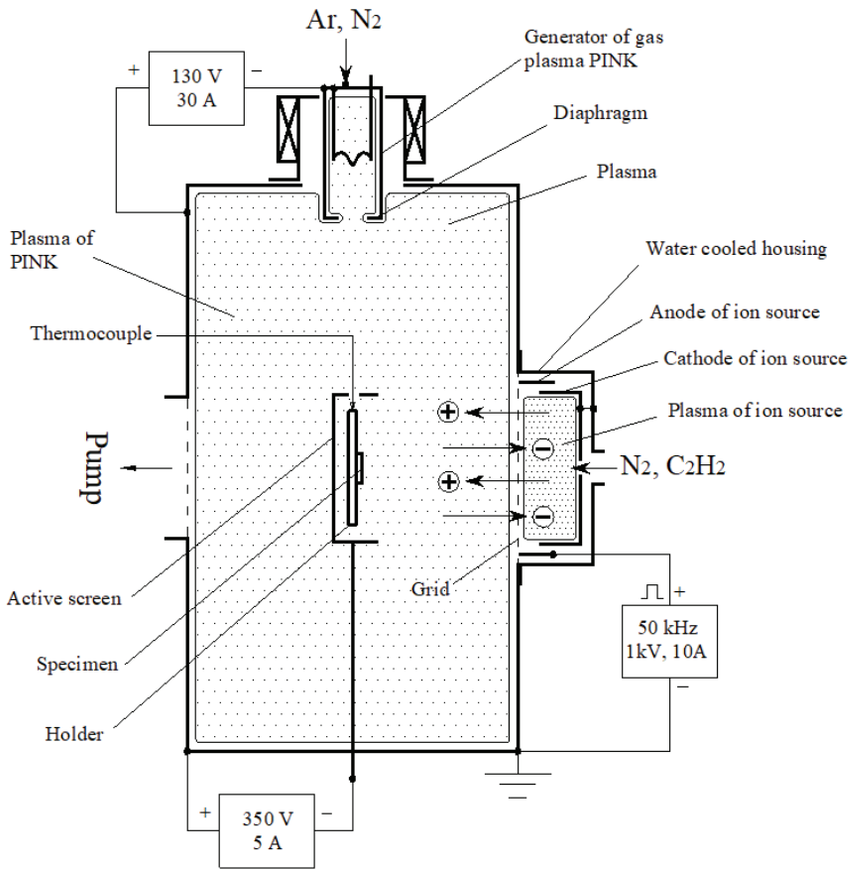

Fig. 1. Experemental sheme

[1] The work was supported by the Russian Science Foundation (project No. 14-29-00091)

NANO-SCRATCH TEST OF NANOSTUCTURED SURFACE LAYER TI-6AL-4V ALLOY [1]

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Titanium alloy Ti-6Al-4V is widely used in numerous and various fields of application, such as aircraft and rocket construction, shipbuilding, chemical industry, etc. [1,2]. However, use of the titanium alloy in friction units is constrained by its low wear resistance [3]. The formation of nanostructured states in the titanium alloy is one of the most promising ways to increase its wear resistance. The mechanisms of deformation of Ti-6Al-4V samples subjected to ultrasonic impact and electron-beam treatments under scratch test are studied in this paper. The scratch test helps reveal the effect of the structure, phase and element composition of a thin nanostructured layer on the regularity of elastic scratch restoration when normal or tangential loads are applied.

TEM studies showed that the titanium alloy in the initial state consisted of equiaxed grains of α-phase with an average size of 2 μm and β-phase grains up to 500 nm in size, located along the boundaries or in triple junctions of the α-phase grains. Electron-beam treatment of the titanium alloy Ti-6Al-4V as a result of rapid heating, melting, and crystallization in a thin melted layer leads to the formation of martensite plates of the α′-phase with a size of 80 nm; within these plates, an α′′-martensite with transverse dimensions of 5 nm is observed. The heat affected zone is characterized by the presence of large (5-10 μm) grains of the primary α-phase also having lamellar morphology and β-phase grains with an average size of ~1 μm. Ultrasonic impact treatment of the titanium alloy leads to the formation of a fragmented band structure with α-phase fragments of 50-100 nm in size in the surface layers. As the distance from the surface of the sample increases, the dimensions of the fragments increase and, at a depth of 75-80 μm from the surface, the original structure of the titanium alloy is observed.

Analysis of the microhardness distribution along the lateral face of samples subjected to ultrasonic impact and electron-beam treatments showed that hardness of the surface layer increased in both cases, reaching 6 GPa. However, according to the nanoindentation data, softening of a thin surface layer with a thickness of up to 1 μm is observed in the samples subjected to electron-beam treatment. As a result, its hardness is comparable to the as-received one.

The scratch test showed that the formation of the α′′-martensite phase in a soft surface layer under the electron-beam treatment led to a significant restoration of the scratch. Thus, at a maximum load of 300 mN, the total penetration depth of the indenter is 900 nm, but after the load is removed, the scratch is partially restored and the scratch depth is reduced to 400 nm. In this case, the bottom of the scratch assumes a convex shape, i.e. positive curvature. In the samples subjected to ultrasonic impact treatment, where a nanocrystalline structure of the α-phase was also formed, but the α′′-martensite phase was not observed, the restoration of the scratch is much smaller.

Mechanisms that explain the role of structural phase transformations during plastic material displacement and the pile-up formation at the edges of a scratch in the process of scratch testing of the titanium alloy samples with nanostructured surface layers are proposed.

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[1] This work was supported by the Russian Science Foundation (grant No. 17-79-10362)

WEAR OF CARBIDE INSERTS WITH SURFACE ELECTRON BEAM ALLOYING WHEN MILLING NICKEL ALLOY[1]

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For experiments milling inserts with uncoated fine-grained alloy H13A produced by Sandvik (N15, S20, K25) were used. For milling operations, 345R-1305E-KL inserts without coating (cutting group K) were used. Modified inserts in the course of experiments were compared with a 345R13T5E-ML insert (cutting group M) having a proprietary wear resistant coating.

Some inserts were subjected to alloying of the near-surface layer before application of a wear-resistant coating. The treatment was performed using a RITM-SP station. Depositing of a thin layer of carbide-forming chemicals (in this particular case targets of $Nb_{70}Hf_{22}Ti_8$ alloy) on the surface of the tool before its treatment with electron beam makes it possible to achieve a modified structure on the cutting surfaces owing to a heat-generating reaction between the metal of the film and the carbon contained in the hard alloy. The external layer is enriched with γ-WC and (NbHf)C high-melting carbide phases. The maximum attainable thicknesses to obtain the modified structure are 4 μm.

Finally it was coated at π80+ installations. TiAlN-ML (proprietary name) multilayer coating is made up of alternating nitride phase layers with varying Al and Ti content, and it is grown on an adhesive TiN layer.

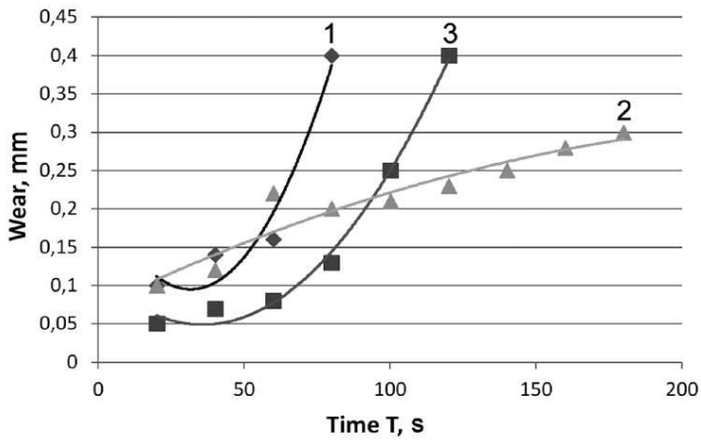

Fig. 1. Rear surface wear of indexable H13A inserts treated differently during face milling of the $NiCr_{20}TiAl$ alloy.
1 – TiAlN-ML coated insert, 2 – TiAlN-ML coated and surface micro alloyed insert, 3 - (AlTi)N coated Sandvik Coromant insert.
v = 95 m/min, t = 0.2 mm, s = 0.1 mm/tooth

The effect of the electron-beam surface alloying on the operation of replaceable inserts was studied during face milling of a forged blank made of $NiCr_{20}TiAl$ alloy having hardness of 330 HB. Milling was performed using a vertical milling console machine VM 127M, under a symmetrical scheme.

By comparing lines 1 and 3 in Fig. 1, the effectiveness of operation of surface antifriction layer in Sandvik insert's coating can be expressly determined. This insert worked almost twice longer than the insert which has a similar coating (TiAlN-ML) and was characterized by considerable buildup formation of the machined material on the areas adjacent to the cutting edge. At the catastrophic wear area, where the effect of antifriction layer is canceled out, abrasive wear of the tool prevails, and the curves behave almost equally.

A somewhat different phenomenon can be observed when milling with a tool subjected to combined surface treatment (line 2). Due to alloying of the near-surface layer underlying the coating, the insert resists abrasive wear much better. At the cutting regimes specified above, its resistance is two times higher than of a Sandvik insert and three times as high as a TiAlN-ML coated insert, despite the fact that an alloyed insert loses at the initial stage of wear. It should be noted that this is considerable advantage of tools subjected only to surface alloying which occurs at high cutting speeds when loads on base material substantially increase.

[1] We gratefully acknowledge the financial support of this work by the Ministry of Education and Science of the Russian Federation, in the framework of the state task in the field of scientific activity of MSTU "STANKIN" (No 11.1817.2017/4.6).

SURFACE PROPERTY MODIFICATION OF BIODEGRADABLE POLYMER AND COMPOSITES BY LOW-TEMPERATURE ATMOSPHERIC PLASMA TREATMENT[1]

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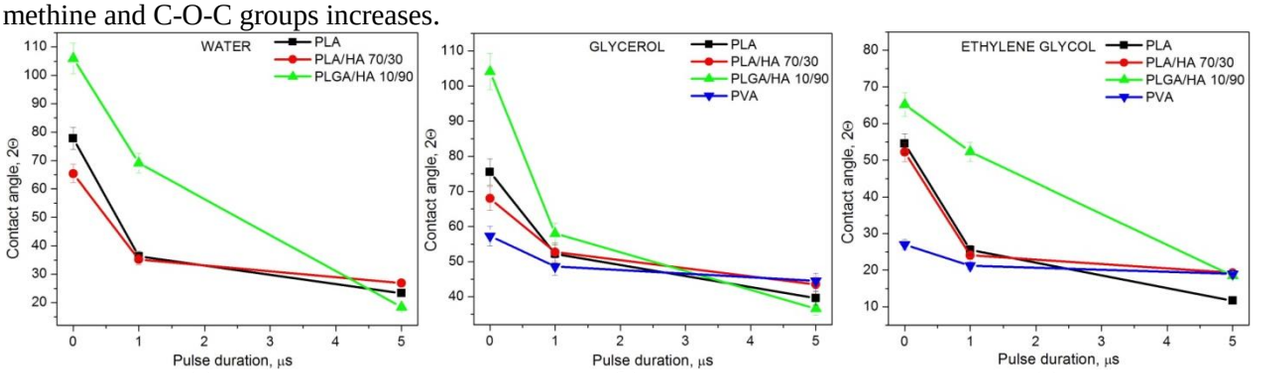
The effect of argon flow low-temperature atmospheric plasma treatment on surface physicochemical properties of biodegradable and biocompatible polymers (polylactic acid – PLA and polyvinyl alcohol – PVA) and composites based on polylactic acid and co-poly(lactic-glycolic acid) with hydroxuapatite (PLA/HA 70/30, PLGA/HA 10/90) was investigated. The plasma treatment conditions were following: the amplitude of discharge voltage – 300 V; the amplitude of discharge current – 40 mA; pulse duration – 1 and 5 μs; exposure time – 3 min; frequency – 100 kHz; the electron temperature – 0.3 eV; the plasma concentration – 5×10^{11} cm^{-3}. Influence of gas-discharge atmospheric plasma on polymer materials is accompanied by their surface property alteration as wettability, microhardness and surface resistivity which caused by destruction and new molecular bond formation, surface microrelief modification – smoothing or cratering [1]. X-ray diffraction analysis reveals that phase composition of the plasma-treated materials remains identical to the initial state; new peaks and diffraction line displacement are not occurred, however the intensity increasing and the peak narrowing of the treated samples indicate that the degree of crystallinity enhances. Infrared spectroscopy reveals that after plasma treatment of PVA, lines in the 1710 cm^{-1} region occur, distinctive for carbonyl (-C=O) stretching vibrations and related to surface oxidation [2]. This may be due both to local heating by plasma irradiation and to the presence of excess electrons from polymer chain scission. For other materials new line is not emerged; IR-spectra of the composites contain lines of both polylactic/glycolic acid and hydroxuapatite. In the IR-spectra of plasma treated PLA, the number of methyl, methine and C-O-C groups increases.



(a) (b) (c)

Fig. 1. The contact angle polylactic acid, composites PLA/HA 70/30, PLGA/HA 10/90 and polyvinyl alcohol depending on the irradiation conditions when wetted with: a) water b) glycerol c) ethylene glycol

Wettability of the materials after plasma treatment is significantly improved, as evidenced by a decrease in the contact angle when wetted with water, glycerol and ethylene glycol (Fig. 1), and also accompanied by an increase in free surface energy. Surface energy modification can significantly affect the bioavailability and surface cell absorption. Implants may have greater or lesser wettability, ability to adsorb cells that participate in electrochemical processes, and bioresorption characteristics. Microhardness of the plasma-treated samples is reduced. Surface resistivity of PL, PLA/HA and PVA does not change significantly, but the electrical conductivity of PLGA/HA increases by 3 orders of magnitude. Thus, it is shown that argon flow low-temperature atmospheric plasma treatment is an effective technique for biocompatible polymer and composite surface physicochemical and mechanical property modification.

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[1] Work supported by the Tomsk State University competitiveness improvement program (project # 8.2.06.2017).

EFFECT OF THERMAL TREATMENT ON THE STRUCTURE, PHASE COMPOSITION AND PROPERTIES OF STEELS SURFACE LAYER MODIFIED BY COMPRESSION PLASMA FLOWS

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Mixing of a "coating/substrate" system by ion, electron, plasma and laser beams allows alloying the substrate material with the coating elements. This process is of special interest in producing nonequilibrium, immiscible and metastable compounds. The use of such a technique for materials treatment leads to formation of surface layers with improved properties. At the same time metastable structure of the modified layer may be not effective for materials working at the high temperature. Thus investigation of the effect of thermal treatment on the structure, phase composition and properties of steels surface layers modified by compression plasma flows was the main aim of this work.

Different types of steels (plain carbon steel, instrumental steel and high-speed steel) were the research objects. Before compression plasma flows (CPF) treatment metal coating was deposited on the steel surface using the vacuum arc vapor deposition technique. CPF were obtained in nitrogen atmosphere using a gas-discharge magneto-plasma compressor of compact geometry. Structure, element and phase composition of the surface layer were characterized by the X-ray diffraction analysis, scanning electron microscopy and energy-dispersive X-ray microanalysis. Vickers microhardness and tribological tests were carried out. Annealing in air in the temperature range of 200-600^{o}C and period of 1-9 hours was carried out to investigate modified layers structure and properties stability.

The findings showed that CPF treatment of the "coating/steel" system samples led to melting of the coating and substrate surface layer, liquid phase mixing by convection flows initiated by hydrodynamic Kelvin-Helmholtz instability and subsequent crystallization under conditions of high-speed cooling. CPF treatment of high-speed steel resulted in carbides dissolution and formation of the oversaturated solid solutions on the basis of α- and γ-Fe, thus leading to softening of the surface layer. In spite of surface properties worsening such treatment can be used as high temperature quenching, which provides the formation of a homogeneous structure. Subsequent annealing allows recovering the structure and obtaining more homogeneous distribution of hardening carbides than carbide distribution formed by traditional thermal treatment, thus preventing microchipping cutting tools made from high-speed steels. Annealing in air of the plasma treated sample of AISI T1 steel for 9 hours at the temperature of 600^{o}C resulted 1.3 times microhardness increase (up to 12 GPa) compared to the initial steel sample

Alloying of instrumental steel U9 with Cr, Zr and Si allowed improving it resistance to oxidation. The findings showed that the phase composition and structure of the layer alloyed with Zr and Si were stable up to the temperature of 400 ^{o}C. Formation of iron oxides on the alloyed surface was found at 600^{o}C in contrast to initial steel sample where iron oxides started to form at 400^{o}C. At the same time annealing of alloyed sample at the temperature of 600^{o}C led to the internal oxidation accompanied with formation of iron oxide scale at the surface and oxygen atoms penetration at a whole depth of the alloyed layer. Similar effect was found in the surface layer of U9 steel alloyed with Cr atoms. Weight measurements showed that alloying with 13% of Cr improved steel oxidation resistance in spite of internal oxidation.

The mechanisms and the reasons of the observed effects are discussed.

INVESTIGATION OF THE PLASMA-CHEMICAL SYNTHESIS OF FULLERENES AND MODIFICATION OF BUILDING MATERIALS BY FULLERENES

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The influence of fullerene synthesis parameters on the yield and content of fullerenes in carbon soot was investigated. The synthesis of the initial fullerene mixture was carried out in a plasma chemical reactor.[1] In this reactor, fullerene synthesis is performed at atmospheric pressure. The arc discharge is in a closed sealed volume filled with helium. The content of fullerenes in the carbon soot changes with increasing helium pressure in the chamber.

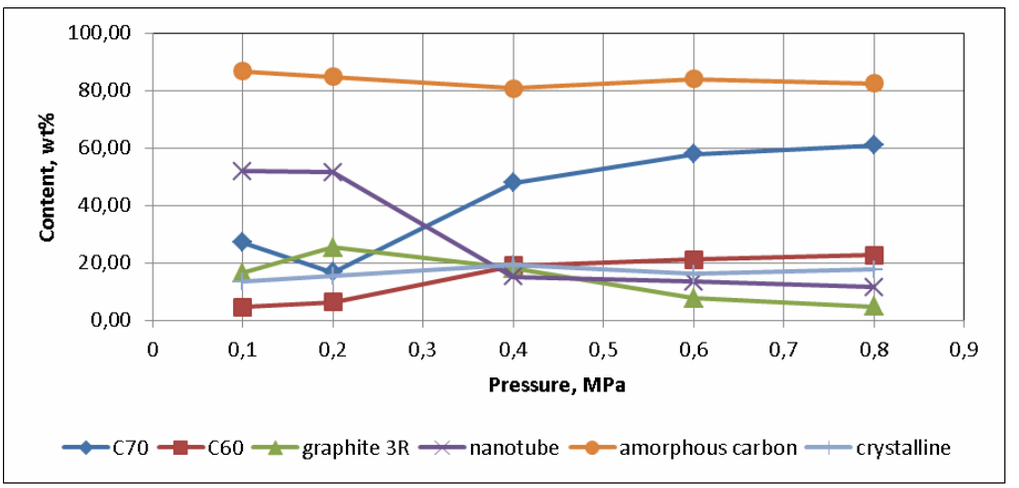

Fig. 1. The dependence of the yield of fullerenes from the gas pressure

Carbon soot containing fullerenes was used as the carbon nanomodifier (CNM) to improve the properties of construction materials. The phase composition, structure, and physical and mechanical properties of building materials change when using carbon nanomodifier. Carbon nanomodifier changes the properties of mixing water, creating around their directionally oriented particles of hydrated shell, which lead to the change of rheological characteristics of cement paste. Furthermore, carbon nanomodifier particles are the centers of crystallization of cement hydration products, which accelerates the processes of hydration and hardening of cement, especially in the initial period of hardening. Porosity of a cement stone is reduced when using carbon nanomodifier, which leads to high strength performance cement. The kinetics of phase hydration in the process of cement stone formation was investigated by thermodynamic modeling and x-ray phase analysis. Complex physico-chemical analysis of hydrate compositions with additives was carried out. CNM has a structural effect on the cement system with the formation of more calcium hydrosilicates, the synthesis of which allows accelerating the processes of hydration and hardening of cement binders. The introduction of carbon nanomodifier in the amount of 0.01% by weight of cement leads to an increase in strength by 10%, and in the amount of 0.001% - by 35%.[2]

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STRUCTURE AND PROPERTIES OF HIGH-CHROMIUM STEEL TREATEDED BY LOW-ENERGY ELECTRON BEAM WITH PULSE DURATION OF (50-450) MICROSECONDS[1]

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The purpose of the present work is the analysis of structure, mechanical and tribological properties of the high-chromium 420 steel (USA analogue; 20Cr13 – Russian analogue) irradiated by an intensive low-energy pulsed electron beam in the wide range of pulse duration. The radiation of steel is carried out by electron beam with pulse duration of (50-200) μs on SOLO installation [1]; and with pulse duration of 450 μs on COMPLEX installation [2]. In the both cases the electron beam energy density and pulse number were 40 J/cm^2 and 3, respectively. The research of phase and elemental composition, state of a defective substructure were carried out by methods of the scanning and transmission electron microscopy; the phase structure and state of a crystal lattice were studied by methods of X-ray phase analysis; the mechanical properties of the irradiated surface were characterized by microhardness; tribological properties were characterized by wear resistance.

It is shown that electron beam treatment of 420 steel in the mode of melting of surface layer by an electron beam with any pulse duration is followed by dissolution of particles of an initial carbide phase with $M_{23}C_6$ ($(Cr, Fe)_{23}C_6$) composition, by saturation of a crystal lattice of surface layer with chrome atoms, by formation of dendritic crystallization cells of submicron sizes (Fig. 1, a), by allocation of nanodimensional particles of chrome carbide (Cr_3C_2) and intermetallic compound (FeCr), by formation of martensitic structure (Fig. 1, b). In total it has allowed to increase the hardness and wear resistance of surface layer of 420 steel. It is revealed that the microhardness of the irradiated steel increases by 1,6 times compared with that of an initial state, and it isn't dependent on pulse duration. The wear resistance of steel essentially depends on electron beam pulse duration and its value at 450 μs exceeds wear resistance of initial steel by many times.

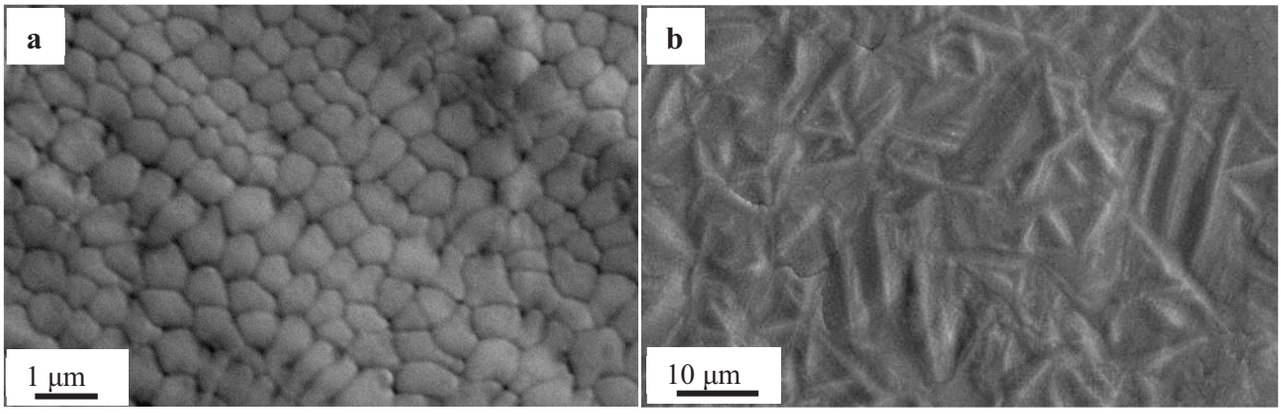

Fig. 1. Surface structure of 420 steel specimen irradiated with an intensive pulse electron beam with pulse duration of 200 μs and electron beam energy density of: a – 40 J/cm^2, b – 20 J/cm^2. Scanning electron microscopy.

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[1] This work was supported by RFBR (project № 16-43-700659)

STRUCTURE AND OPTICAL PROPERTIES OF SELENIUM INMPALNTED SILICON PROCESSED WITH SHORT-TIME ANNEALINGS[1] *F*

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Silicon is widely used for fabricating optoelectronic devices, but silicon's indirect 1.12 eV bandgap limits its applications in the infrared (IR) field. Selenium supersaturated silicon is a promising candidate for IR photodetectors and efficient solar cells because of its sub band gap absorption [1,2].

In this work silicon was hyperdoped with selenium by ion implantation (E = 125 keV, D = $1x10^{16}$ cm^{-2}) with subsequent pulsed laser melting (PLM). Pulsed laser melting was performed by ruby laser (690 nm, 70 ns FWHM) in conditions described in [3]. Pulse energy density W was set at 1.5, 2 and 2.5 J/cm2. For each W two specimens was irradiated by one and three laser shots respectively. Rutherford backscattering with channeling (RBS/C) was performed to obtain impurity depth distribution (Fig. 1) along with silicon crystallinity profiles. Photoluminescence spectra, Raman scattering spectra, SEM, TEM and optical microscopy images of near-surface region were obtained.

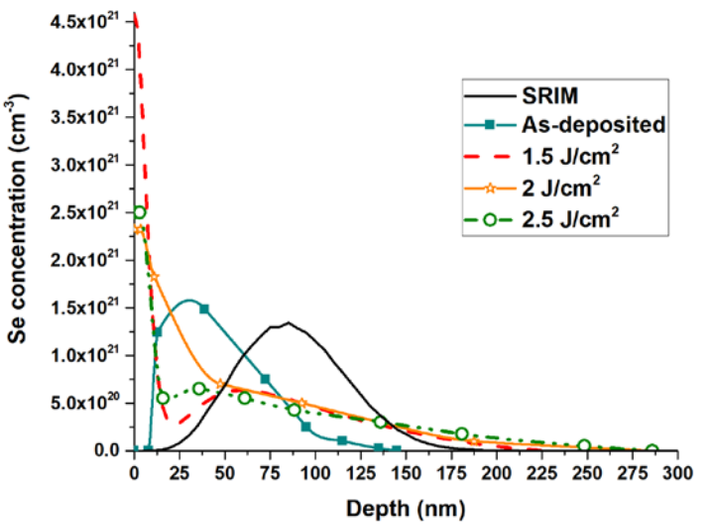

Fig. 1. Simulated (SRIM-2013) and calculated from RMS spectra depth profiles of impurity concentration in Si after $^{80}Se^{+}$ implantation (125 keV, $1x10^{16}$ cm^{-2}) and subsequent pulsed laser melting (690 nm, 70 ns FWHM) with energy density varied from 1.5 to 2.5 J/cm^2 with 1 pulse. PLM

leads to a significant impurity redistribution forming a uniform plateau with a concentration of Se around ($3x10^{19}$ – $1x10^{20}$) cm^{-3} up to depth of 300–400 nm with an accumulation of impurity in a near-surface oxide layer 5–10 nm. PL spectra shows that the PLM of the implanted layer at the smallest W = 1.5 J/cm^2 leads to the formation of vacancy and interstitial clusters. As the energy in the pulse increases, these types of defects in the PL spectra do not appear. An increase in the number of pulses leads to an increase in the intensity of the bands of dislocation luminescence. SEM and optical microscope images show that PLM with W higher than 2 j/cm^2 lead to insignificant irregularities on the surface.

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[1] This work was supported by the Belarusian Republican Foundation for Fundamental Research, grant no. F17RM-079.

INFLUENCE OF MICROWAVE RADIATION ON CHEMICAL REACTIVITY OF ALUMINUM POWDER[1]

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Experimental results on the effect of microwave radiation on the chemical activity of aluminum powder ASD-6M when heated in air and combustion in the mixture composition based on ammonium perchlorate and butadiene rubber are presented.

To activate micron aluminum powder by microwave radiation, an experimental stand is used, which includes a microwave source based on a magnetron generator of 3 cm. The aluminum powder was activated in a flux of microwave radiation with a density of 80 W/cm 2 for 20 seconds. Dispersity of the initial and activated aluminum powder was determined on the «Mastersizer 2000» instrument, using the laser diffraction method. The efficiency of the microwave process for the activation of ASD-6M powder was determined using the differential thermal analysis method on the «STD Q600» instrument to change the exothermal effect of aluminum oxidation when a sample weighing 5 mg was heated in air at a rate of 10 °C/min. The error in determining the values of the thermal effects is ± 1.8%. The efficiency of the effect of microwave activation of aluminum powder on the process of its combustion in the mixture composition was determined from the linear burning rate in the air medium at atmospheric pressure, and also by the calorific value of the combustion in calorimetric bomb. To this end, aluminum powders were mixing into the mixture of ammonium perchlorate and butadiene rubber, followed by hardening of the initial mass and the production of model propellant samples.

The results of the investigation of the dispersion of aluminum powder, the temperature of the onset of oxidation and the thermal effects of aluminum oxidation under non-isothermal conditions, as well as the burning rate of the mixture compositions and the heat of combustion in a calorimetric bomb are given in Table 1.

Table 1. Results of investigation of aluminum powder and compositions based on.

Parameters of aluminum powder			Parameters of the mixture composition		
Powder type	Dispersion, D_{43}, μm	Oxidation onset temperature, $T_{н.о.}$, °C	Heat of oxidation, ΔH, J/g	Burning rate, mm/s	Heat of combustion, MJ/kg
ASD-6M	4.74	~ 540	7270	1.57 ± 0.04	5.6 ± 0.1
ASD-6M (microwave radiation)	3.71	~ 435	8076	1.52 ± 0.03	6.2 ± 0.1

It is seen that the activation of microwave radiation increases the dispersion of aluminum powder ASD-6M, the average volume diameter of the particles is reduced by 22%. The oxidation temperature of the activated powder decreases by more than 100 °C, and the total thermal effect of the oxidation reaction increases by 11%. Substitution the batch of industrial aluminum powder with the activated microwave radiation practically did not affect the value of the linear burning rate of the mixture composition.

Thus, the obtained results indicate that activation of aluminum powder ASD-6M with the help of microwave radiation increases heat release during oxidation of aluminum under non-isothermal conditions and during combustion of propellant samples.

[1] This work was supported by the Ministry of Education and Science of the Russian Federation in terms of state order, project code 9.9063.2017/8.9

SIMULATION OF IRRADIATION EFFECTS WITH IONS ON THE RFQ LINAC HIPR[1]

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Radiation damage is the limiting factor in the choice of structural materials for advanced fission and fusion reactors. Neutron irradiation of materials is a slow and cost intensive process often extending over several years. Heavy ion irradiation is the only viable means to conduct accelerated testing to assess irradiated microstructures at high dpa levels. In the NRC "Kurchatov Institute" – ITEP the samples of perspective steels and alloys are irradiated at the RFQ Heavy Ion Prototype (HIPr) by a beam of accelerated heavy ions (Fe, Ti, V, …) up to dose 10^{17} ions/cm^2. During irradiation the investigated samples are heated up 500 °C.

We have now studied the heavy ion distribution on the target. The results of beam profiles measurements for different irradiation regimes are presented. To improve the beam control a device was developed and produced. This device is based on the Arduino platform and software for analysing the data received in the streaming mode. The device allows to control all BPM signals together and takes account of these signals for the dose calculation.

[1] This work was supported by the Russian Science Foundation (14-13-01380).

PHYSICAL AND CHEMICAL ASPECTS OF ION ETCHING PROCESS FOR CUTTING TOOLS REFURBISHING

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Presently, various kinds of hard film coatings are used in combination with cemented carbide and high speed steel cutting tools for increased longevity [1]. For further improvement of the coated tools' life-span, their regrinding and recoating are commonly implemented. Successful recoating requires stripping off of a previous coating in order to provide sufficient adhesion. Wet electro-chemical etching process is commonly applied for the stripping purpose. Recently, dry ion-plasma etching (IPE) and ion beam etching (IBE) based approaches were introduced, offering substantial advantages over the conventional methods [2,3]. The novel approaches utilize inert and reactive gases to remove composite and diamond based thin films respectively. In addition to the attractive technical features, the IBE and IPE stripping methods have a strong sensitivity to the tool geometry, due to the ion shadowing and ion focusing effects, which must be thoroughly studied in order to verify the applicability of the methods to the wide range of the existent cutting tools.

Both IBE and IPE stripping methods discussed in this work are based on the same combination of physical sputtering and chemical erosion of the thin films, caused by bombardment with energetic ions and radical fluxes. However, ion transport modes are rather different in the two cases, which significantly influence the erosion rate distribution over a non-trivial geometry workpieces.

In the present work composite PVD films (TiAlN, TiN) and CVD-diamond films deposited on the cemented carbide (WC-Co) and highs speed steel (HSS) drill bits were IPE and IBE treated, and spatial erosion rate distributions were studied in the context of the target tool geometry, ion kinetic energy and concentration of reactive species. Moreover a simplified 2D mathematical model for the IBE and IPE stripping processes was suggested in order to support the experimental findings.

In the IBE setup the coated workpieces were exposed to multiple high current (~100 mA) large aperture (10 cm) ion sources, generating directional flows of ions with average kinetic energy in the range of 1 keV.

The IPE process was carried out in a DC discharge volume type Ar/O_2 plasmas of ~10^{10} cm^{-3} density. Plasma extraction of the ions and acceleration was achieved via pulsating negative bias in the range of 1kV. In this case, the ions were moving in generally non-uniform electrostatic field formed between plasma boundary and workpiece surface.

The erosion rate distribution was evaluated from SEM images of the workpieces' cross-sections, acquired before and after the etching.

Computer simulation of the ion fluxes and erosion rate distribution was carried out through combination of FEA of the electrostatic field and semi-analytical solution of the ion motion equations.

Experimentally, it was shown that IPE and IBE result in rather uniform erosion of the cylindrical land portions of the drill bits, while the concave flute portions were eroded at significantly different rates. The IBE was shown to be preferable, providing better overall homogeneity of stripping, while IPE have demonstrated incomplete stripping in certain cases (e.g. see figure 1).

Suggested simulation model have shown good agreement with the experimental results and have supplemented the discussion on the erosion rate distribution variance.

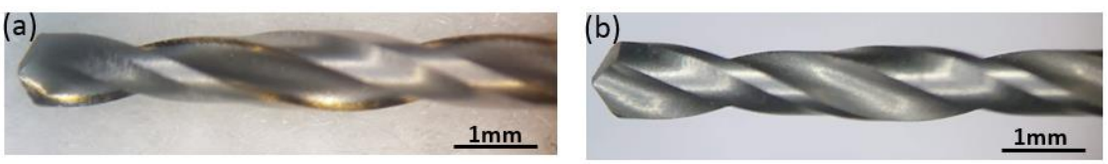

Fig. 1. Optical microscopy of TiN coated HSS drills, showing incomplete stripping in the case of IPE – (a) and complete stripping in the case of IBE – (b).

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NANOSCALE DYNAMIC EFFECTS AND LONG-RANGE EFFECTS IN CONDENSED MATTER UNDER CASCADE-FORMING IRRADIATION[1]

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Classical radiation physics describes well a number of known phenomena observed under irradiation of metals and alloys (radiation embrittlement, radiative swelling, radiation creep), based on relatively slow processes of thermo- and radiation-enhanced diffusion. Mechanisms based on the description of the defect migration processes can not, however, explain the "low-dose effect" under neutron irradiation and the low-dose "long-range effect" under irradiation with accelerated ions $E \sim (10^4 - k \cdot 10^5)$ keV ($1 \leq k \lesssim 3$).

The report is devoted to a brief review of the model that takes into account the nanoscale dynamic effects during cascade-forming irradiation. We are talking about explosive energy release in the regions of dense cascades of atomic displacements (thermal spikes) emitting powerful post-cascaded solitary waves, which can initiate structural-phase transformations in metastable media, theoretically, at unlimited distances. The distances at which the effect of accelerated $(10^4 - k \cdot 10^5)$ keV ion beams is observed (in the continuous irradiation mode) are sometimes more than a few tens/hundreds of micrometers (at projected ion ranges of less than 1 μm) and, as recent studies have shown, can reach 1–10 millimeters. These effects are considered on the basis of experimental research data of more than ten different systems. The foundations of the theory of undamped propagation of plane and spherical waves in metastable media are presented. It is noted that the most probable energy of recoil atoms generated by reactor neutrons and fission fragments also belong to the above energy range, which indicates the need to take into account the nanoscale dynamic effects, regardless of the type of the cascade-forming irradiation.

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EFFECT OF ION IRRADIATION OF ZIRCONIUM ALLOY E110 AND ITS LASER WELDS ON CORROSION RESISTANCE AND HIGH-TEMPERATURE OXIDATION

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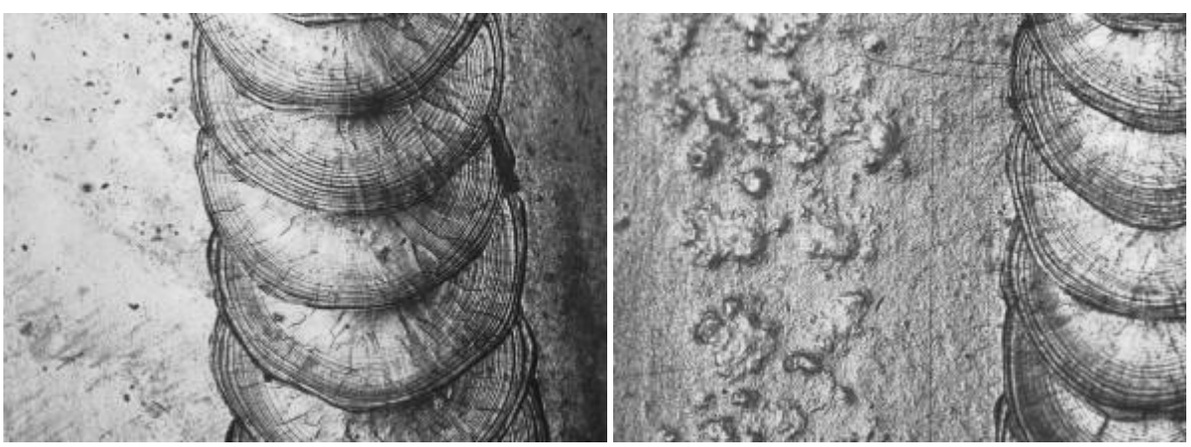
Zirconium alloy E110 is one of the main structural materials of fuel assemblies for nuclear reactors. Requirements for reactor safety and their economic efficiency are constantly increasing because of attempts to increase burn-up and reduce the cost of production of fuel cells. One of the ways to solve these problems is to replace widely used electron beam welding with a laser, which does not require vacuum chambers. In addition, the fact that an aggressive environment (radiation, corrosion, high temperature, etc.) acts through the surface of the fuel rods, makes it relevant the application of coatings or surface modification by concentrated energy flows.

In this paper, we investigated the effect of modification of the surface layer of the E110 zirconium alloy sheets as well as its butt welds (made by laser pulsed welding using different modes) by a high-power pulsed ion beam. The beam was composed of ions of carbon (70%) and protons (30%) with the energy of particles ~ 250 keV, the current density of a pulse was 80 A/cm^2, the number of pulses – 5. The irradiation was carried out in a vacuum chamber at room temperature and pressure of 10^{-4} Torr.

Figure 1. Samples after corrosion test (top – irradiated; bottom – without irradiation)



a) b)

Figure 2. Top views of the samples: a) after irradiation; b) after irradiation and high temperature oxidation

The results of microstructural analysis of surface layers of the base metal and welds after irradiation as well as corrosion autoclave test (figure 1) and high-temperature oxidation at a temperature of 1200 °C (figure 2) which simulate loss-of-coolant-accident are presented.

ULTRA-HIGH FLUENCE LOW ION ENERGY IMPLANTATION OF Al INTO Ti

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The work describes the first experimental results of ultra-high fluence ion implantation of aluminum into titanium. A DC vacuum arc was used to produce aluminum plasma flow. A repetitively pulsed macroparticle-free high intensity aluminum ion beam was formed using a plasma immersion ion extraction combined with ion beam focusing. A very high current ion beam with the current up to 1 A and the corresponding ion current density up to hundreds of mA/cm^2 was obtained. Titanium substrates were irradiated by low-energy aluminum ions (mean ion energy of units of keV) at the fluences reaching $10^{21}ion/cm^2$. The integral substrate temperature varied from 300° C up to 900° C. The experimental investigations showed the deep penetration of the implanted dopant as well as the formation of Ti-Al intermetallic phases. After the irradiation at elevated temperatures, the surface morphology demonstrate the formation of melted surface layer, which indicates the existence of significant temperature gradient. The simulation results of the influence of high intensity ion beam implantation on the temperature distribution, ion sputtering and dopant penetration at elevated ion current densities are also presented.

EFFECT OF PLASMA ION-IMMERSION TREATMENT ON THE STRUCTURED AND PHASE STATE OF THE TINI ALLOY FOR MEDICAL IMPLANTS

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TiNi-based alloys with shape memory and superelastic effects are the most preferred materials for medical implants [1]. Modification of alloy surface enhances the functional properties of TiNi alloys (biocompatibility, corrosion resistance, etc.) and limits the release of toxic Ni into the recipient's organism [2]. The method of plasma-immersion ion implantation with deposition allows modifying the surface of medical products of complex geometric shape. This treatment includes a controlled heating of the samples, facilitating their surface alloying due to thermally activated diffusion [3]. However, for aging TiNi alloys it is necessary to take into account that stimulation of diffusion processes creates conditions for the decomposition of the solid solution and can lead to a change in the temperature range of the martensitic transformations [1].

In this paper, we present the results of studying the structure and phase composition of surface and near-surface layers of samples widely used in medicine for Ti-50.8 at. % Ni. The samples were in a different structural phase state: (i) coarse-grained (CG) TiNi (average grain size D = 15 μm) and (ii) recrystallized ultrafine-grained (UFG) TiNi (D = 170 nm) containing particles Ti_3Ni_4. Ion-plasma treatment with Si ions was carried out on a "SPRUT" unit (NI TSU, Tomsk) with a mode: pulsed negative voltage bias Us = 1000 V, f = 30 kHz, power of magnetrons P = 0.8 kW, t = 60 min. The microstructure was examined by the transmission electron microscopy using the JEM 2100 (JEOL) microscope ("Nanotech" Center, ISPMS SB RAS). Thin foils in the transverse and longitudinal sections were prepared by ionic thinning at the EM 09100IS (JEOL). The investigation of the elemental composition of the surface was performed by the EDS and RFS methods using "Nanoscan 50" ("Nanostructures" Center, Novosibirsk).

We have shown that after the ion-plasma treatment, independently of the initial structure of TiNi samples, a separate amorphous layer 5 ÷ 10 nm thick is formed on their surface containing up to 7 at.% Si for the alloy CG and up to 14 at.% Si for the UFG alloys. The concentration of Si atoms decreased with the increasing distance inward from the surface of the sample to the values at the detection limit of the spectrometer at a depth of 80 nm for the alloy CG and up to 1.5 μm for the UFG TiNi alloy. It was found that in the process of plasma ion-immersion treatment of TiNi alloys in the near-surface layers, diffusion and martensitic transformations occurs, the characteristics of which were determined by the state of the initial material. For CG and UFG alloys, a significant increase in the temperature range of the martensitic transformations was observed, which was associated with the aging of B2 TiNi solid solution. At the same time, aging occurred in the CG alloy, with the excess precipitates of Ti_3Ni_4 predominantly distributed in a homogeneous decay mechanism and the B2 + R martensitic structure was formed. For UFG TiNi alloys containing in the initial state of Ti_3Ni_4 precipitates, recrystallization processes were characteristic, accompanied by coagulation and particle growth along the grain boundaries. The alloy was in the B2 + R state, the diffusion disintegration inside the UFG grains was not detected.

Mechanisms of structural and phase transformations, as well as an increase in the temperature range of martensitic transformations of TiNi alloys after plasma-immersion ion implantation of materials, are discussed. The obtained data on thermally activated processes in TiNi during plasma ion-immersion implantation of a surface must be taken into account when selecting ion-plasma treatment regimes.

The work was carried out within the framework of the Program of Fundamental Scientific Research of the State Academies of Sciences for 2013-2020, III 23.2.2.

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EFFECT OF PULSED ELECTRON-BEAM TREATMENT AND SUBSEQUENT ADDITIVE THIN-FILM SYNTHESIS OF SURFACE TI-TA-NI ALLOYS ON NANOCOMPOSITE STRUCTURE, RESIDUAL STRESS, AND SHAPE MEMORY CHARACTERISTICS OF TINI SUBSTRATES[1]

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TiNi shape memory alloys (SMAs) have found many advanced applications in medical and engineering devices due to combination of superelastisity and high corrosion resistance. Two of the critical issues limiting the application of TiNi SMAs are a danger of toxic Ni release into the adjacent tissues, as well as insufficient level of X-ray visibility. These limitations can be overcome by fabrication of a Ti-Ta based surface alloy on the TiNi substrate, because of Ti-Ta alloys being high-temperature SMAs are attractive biomaterials with potentially good mechanical compatibility with TiNi substrate. In this work, this approach is realized through the multiple (20 cycles) alternation of magnetron co-deposition of $Ti_{70}Ta_{30}$ (at.%) thin (50 nm) films and their liquid-phase mixing with the TiNi substrate by microsecond low-energy, high current electron beam (≤ 15 keV, ~2 J/cm^2). Surface SEM/EDS, AES, XRD/GIXRD and cross-sectional HRTEM/EDS/SAED techniques were used for microstructural characterization of the studied surface alloy. It was found, that ~1 μm in thick surface alloy of near $Ti_{55}Ta_{25}Ni_{10}O_5$ (at. %) composition has been fabricated on TiNi substrate, and the total thickness of Ta alloyed surface layer is ~2.5 μm. The surface alloy possesses higher X-Ray visibility compared to TiNi. It has multilayer structure associated with additive fabrication processes and consist of the subsurface amorphous sublayer and several (≥5) underlying nanocrystalline sublayers with fine (10-15 nm) and "coarse" (80-100 nm) alternating with each other. Nanocrystalline sublayers has predominantly α″-martensite structure based on Ti-Ta solid solution. It was revealed by nanoindentation possesses a predicted mechanical compatibility to TiNi substrate.

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PULSED ELECTRON BEAM SCANNING TREATMENT OF TITANIUM ALLOYS

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Among the variety of methods of the surface treatment of materials based on the use of high-concentration energy sources, a special place belongs to electron beam treatment. The simultaneous radiation, thermal, and impact mechanical action on the surface of a metallic sample, which is accompanied by ultra-high speeds of heating (to temperatures that substantially exceed the melting point) and cooling, leads to form in it an amorphous, nanocrystalline, or submicrocrystalline structure. This modification of the microstructure and phase state of the surface of structural materials makes it possible to substantially increase their wear resistance, corrosion resistance, and dynamic strength, to decrease the friction coefficient of articles, etc.

One of the most promising direction for the electron-beam treatment is post processing of the metal parts produced by additive manufacturing. The treatment results to smooth surface of 3D-printed products thereby increasing its corrosion resistance and fatigue life as well as to inhibit dislocation nucleation in their surface layers under the following mechanical loading that provides considerable increasing their strength. In addition, electron beams irradiation allows simultaneous thermal annealing of the specimen bulk, and, accordingly, to reduce the degree of disequilibrium of the 3D-printed specimens, and, consequently, to increase their impact toughness.

In the present paper the effect of pulsed electron beam scanning treatment on the microstructure and mechanical properties of titanium parts manufactured by electron beam melting and titanium cast alloy was investigated. As the materials for the studies, the commercially pure titanium and titanium Ti-6Al-4V alloy were chosen. Pulsed electron beam scanning treatment was conducted on system 6E400 equipped with a manipulator moving in three mutually-perpendicular planes. It allows processing parts of different shapes and sizes. The total depth of the melted surface layer and the heat affected zone was 200 μm.

The effect of pulsed electron beam scanning treatment on the surface morphology of 3D printed titanium parts and titanium cast alloy subjected to was studied using atomic force microscope and optical profilometer. The regularities in the formation of the microstructure and phase and element composition of the materials under study were investigated using X-ray diffraction, transmission electron microscope and electron backscatter diffraction. The influence of the internal microstructure of modified surface layer on their microhardness and mechanical properties under uniaxial static tension was demonstrated. The possibility of efficient control of the mechanical properties of 3D-printed titanium specimens was shown due to the melting of their surface layer and simultaneous heating of their volume in the process of pulsed electron beam scanning treatment.

INFLUENCE OF COMBINED ION-PLASMA TREATMENT ON WEAR-RESISTANCE OF DIE STEEL CR6VW [1]

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A number of works [1, 2] have shown that coating deposition and ion-plasma nitriding of the surface of steels allows a considerable, to several times, increase the tools service life. In the literature mainly studies on nitriding in an anomalous glow discharge plasma are presented, in which the pressure of the working medium is 2-3 orders of magnitude higher than in the plasma of low-pressure discharges (≈ 1 Pa), and nitriding rate 2-3 times below. In addition, there are practically no papers devoted to the study of the structure and properties of die-cast steels after modification by a combined method, in which an extended nitrided layer is created on the surface and a hard coating on top of it.

The purpose of the study is to identify the regularities of evolution of the structure, phase composition and physical and mechanical properties of the near-surface layers of hardened steel Cr6VW modified or created under the influence of ion currents and plasma, and the development on their basis of effective regimes of ion-plasma treatment of die-cast steels to improve wear resistance in complex operating conditions.

In the work, the formation of modified surface layers was carried out in several ways. Three groups of samples from hardened steel Cr6VW were processed. The first group was subjected to nitriding in a nitrogen plasma generated by a non-self-sustained low-pressure glow discharge with a hollow cathode. On the surface of the second group of samples produced the deposition of CrN coating up to several micrometers thick by the arc plasma-assisted deposition method. The third group of samples was processed by a combination of these methods and included preliminary nitriding and the deposition of a wear-resistant CrN coating.

The effect of combined ion-plasma treatment on the structure and phase composition of the near-surface layers of the steel Cr6VW was studied by optical, scanning electron microscopy, X-ray phase analysis. Mechanical (microhardness) and tribological (wear resistance, coefficient of friction) characteristics of modified layers are determined.

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STRUCTURE AND PROPERTIES OF TITANIUM AFTER NITRIDING IN A PLASMA OF PULSED HOLLOW CATHODE GLOW DISCHARGE [1]

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Titanium alloys, which have a unique combination of mechanical, physical and technological properties, such as high specific strength, low density, high corrosion resistance and heat resistance, are widely used in aviation and space technology, medicine, etc. [1]. The shortcomings of titanium alloys, especially unalloyed ones, are low hardness as well as poor tribotechnical characteristics [2]. One of the methods for improving the tribotechnical and mechanical properties of titanium alloys is ion-plasma nitriding. Nitriding process in the plasma of low-pressure discharges takes 2-3 times less time than in an anomalous glow discharge plasma. Intensification of the nitriding process in the plasma of low-pressure discharges at the same temperature of the details is achieved by using the pulsed mode of low-pressure discharge which allows increase the saturating capacity of the plasma medium. The principle is that plasma is generated during discharge pulse with increased density in comparison with average plasma density in the steady-state combustion regime. As a result during the pulse the number of active states of the nitrogen-containing plasma is much larger than in the steady-state plasma. The duration of the pause between the pulses is chosen such that the plasma density does not decrease significantly. Provided that during the pause between the pulses the nitriding process continues, the total amount of nitrogen atoms diffusing deep into the surface, and hence the hardness of the treated layer will be larger after processing in the plasma of a pulsed glow discharge.

The processes of nitriding of titanium VT1-0 in the plasma of a non-self-sustained glow discharge and the study of the mechanical characteristics, structure and phase composition of the modified surface layer were carried out. The pulsed combustion mode of a glow discharge makes it possible to obtain a larger content of nitrogen and a Ti_2N nitride phase in the nitrided layer of titanium VT1-0 than in the modified layer after nitriding in the stationary plasma of such discharge at the same nitriding temperatures, average ion current density to the surface and ion energy. As a result, the hardness of the nitride layer of the sample formed in a pulsed mode is one-quarter greater than for a sample treated in a stationary mode of a glow discharge. In addition, the wear resistance of titanium after nitriding in a pulsed glow discharge plasma is approximately 5 times greater than of titanium in the initial state and is 60 % higher than after nitriding in a stationary glow discharge plasma.

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RESIDUAL STRESSES FORMATION IN THE SURFACE LAYERS OF TARGETS FROM REFRACTORY TITANIUM ALLOYS DURING THEIR IRRADIATION WITH INTENSE PULSED ELECTRON BEAMS

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The present paper reviews the experimental results dedicated by the effect of the irradiation with intense pulsed electron beams under the melting regime on residual stresses creation taking place on the surface of titanium alloy targets (VT6, VT8,VT9, VT18U). Investigations of physical and chemical state in the surface layers before and after irradiation were made with the transmission electron microscopy, optical metallography, X-rays analysis and microhardness measurements. It was showed that irradiation with intense pulsed electron beams under the melting regime of VT6 and VT8 alloys leads to formation the stress texture into the surface layer with thickness up to 20 μm. Also fine dispersional lamilar-globular structure with orientation of α- α'- α'' lamilars are parallel to the surface ofthese alloys. It can lead to increase of fatigue strength during tests.

APLICATION OF INTENSE PULSED ELECTRON BEAMS FOR REPAIR AND PROPERTY RECOVERY OF GhS32 NICKEL ALLOY TURBINE BLADES WITH NiCrAlY+NiAl COATING AND PERFORATE HOLES

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The present paper reviews the experimental results dedicated to the effect of irradiating conditions with intense pulsed electron beams on ablation kinetics of the surface layer of gas turbine engine blades from GhS32 with NiCrAlY+NiAl resistant coatings. Application of intense pulsed electron beam allows one to ablate per a pulse the surface layers fractured during operation with thickness of 5-10 μm, if the energy density is equal to 50-55 J/cm^2.

It is shown that intense pulsed electron beam of microsecond duration is a high effective instrument for modification and repair of turbine blades with perforate holes and resistant NiCrAlY+NiAl coatings without the decrease of fatigue strength.

THE EFFECT OF IRRADIATION WITH INTENSE PULSED ELECTRON BEAMS HEAT RESISTANCE OF GAS TURBINE ENGINE COMPRESSOR BLADES FROM EP866-Sh REFRACTORY STEEL

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The objective of the present research is the discussion of test results, dedicated to the effect of intense pulsed electron beam irradiation regimes upon the oxidation resistance of refractory EP866sh steel. The electron beam treatment was realized by means of the GESA-MMP accelerator under the following conditions: electron energy – E=115-120 keV; pulse duration - τ=30-40 μs; and the energy density in a pulse (w) as well as the number of pulses (n) were varied from w=20-22 J/cm^2, n=1 up to w=36-38 J/cm^2, n=4. Some targets after irradiation were subjected to vacuum annealing for 2 hours at their service temperatures. Corrosion tests of initial and irradiated blades were performed under the operating temperatures. The target surface state prior to and after tests was studied by electron Auger spectroscopy, scanning electron microscopy, transmission electron microscopy and X-ray structural analysis. The test results showed that the corrosion resistance of samples, subjected to electron beam irradiation with the post-process vacuum annealing at the operating conditions, could be enhanced in 2 times.

CELL ADHESION AND GROWTH ON MODIFIED SURFACES[1]

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Surface modifications have been widely used for cell growth for various applications. In this work, we present three different techniques for surface modification: plasma treatment, ion implantation and microfabrication of microstructures. The cell growth analysis on the modified surfaces was performed using living CHO (Chinese Hamster Ovary) cells and Human Dental Stem Cell (hDSC).

Diamond, DLC (hydrogen free diamond-like carbon) and SU-8(epoxi photoresist) surfaces were modified and evaluated concerning to the interaction of living cells.

Plasma treatment was proceeded on diamond and DLC for surface modification. The DLC and diamond samples were treated with oxygen plasma and/or with sulfur hexafluoride (SF_6) plasma, using a small hollow-cathode plasma gun, generating surfaces with oxygen terminations and fluorine terminations, respectively.

In diamond surfaces, we formed adjacent regions with different terminations. Initially diamond films were deposited by plasma assisted chemical vapor deposition, generating surface with hydrogen terminations. Following, the surface was masked using lithographed electron-resist (PMMA) and an oxygen plasma treatment was performed. Then,hDSC were cultured on that diamond surfaces. After the cells reached confluence (28 days) the diamond surface was washed with distilled water, removing all the cells, and the substrate analyzed by SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy). The EDS analysis detected much higher calcium, oxygen and phosphorus concentration for the oxygen terminations regions, suggesting that the extra cellular matrix (ECM) was considerably more developed in the oxygen terminations regions, as compared to the hydrogen terminations regions.

DLC surfaces were treated by oxygen plasma (DLC-O) and sulfur hexafluoride plasma (DLC-F), also using the small hollow-cathode plasma gun. After 24 hours of CHOcell culture, the number of cells on DLC-O was higher than on DLC-F surface.

Silver ion implantation was performed on SU-8 generating silver nanoparticles self-assembled12 nm below the surface. During the implantation, the excess of metal atom concentration above the solubility limit leads to nucleation and growth of metal nanoparticles. CHO cell growth was proceeded on SU-8 with and without Ag implantation. After 24 hours of cell culture, the number of cells on SU-8 /Ag surface was higher than on original SU-8.

SU-8 was lithographed with periodic 3D hexagonal microcavity array morphology with different sizes.The range of microcavity size (inscribed circle diameter) was from 12 μm to 560 μm. Then CHO cell cultures were grown on the surfaces. CHO cells were grown also on flat SU-8 surfaces. The highest cell growth density was obtained for microcavity radius was 80 μm,being even greater than the growth density on a flat substrate (SU-8).

[1] Work supported by FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo) and CNPq (ConselhoNacional de DesenvolvimentoCientífico e Tecnológico).

LOW-ENERGY PLASMA-IMMERSION IMPLANTATION OF NITROGEN IONS IN TITANIUM BY A BEAM WITH BALLISTIC FOCUSING [1]

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The results of experiments on low-energy implantation of nitrogen ions in a titanium alloy VT-1.0 are presented. The treatment by a pulsed beam of nitrogen ions obtained by means of a ballistic ion focusing system was performed. The ion focusing system consisted of a box-shaped case with a rectangular bottom. One side of the box by a metal grid in the form of a half-cylinder was covered. The specimens were located on the collector of the system, which was placed inside the box in the focal plane of the grid. Pulse negative bias on the box and collector was applied. The ions were extracted from the nitrogen plasma source of the arc discharge with a thermionic cathode of extended configuration. It was shown that the treatment of specimens of a titanium alloy BT-1.0 in such a system leads to their saturation with nitrogen. However, intense ion etching occurs on the surface of specimens. Thus, not only the phase composition of the near-surface layers of the samples changes, but also their surface profile.

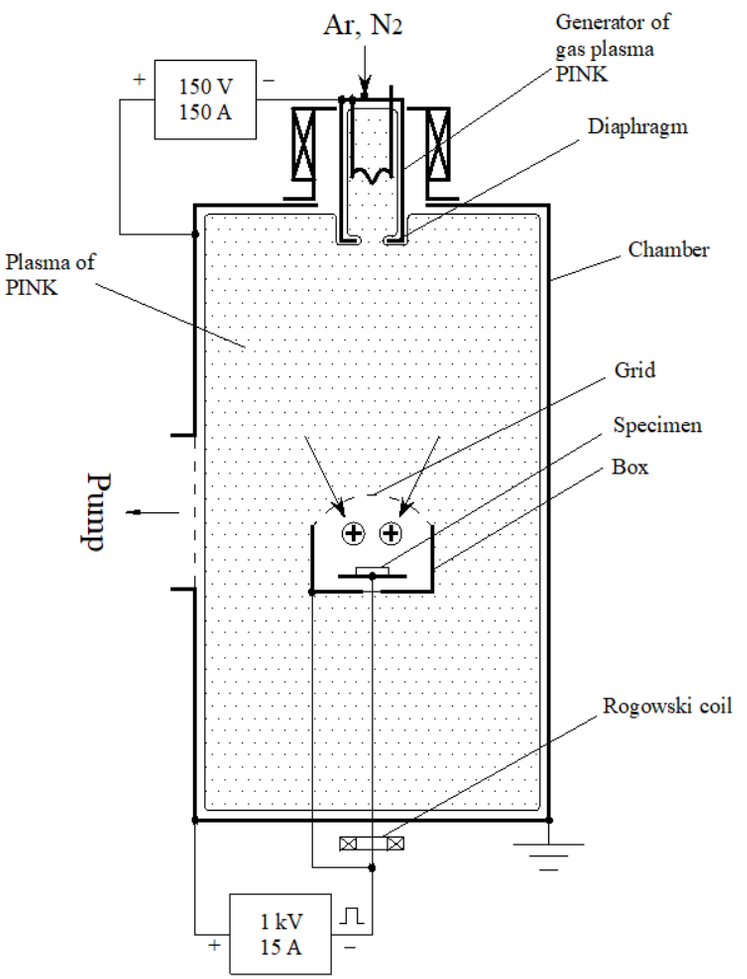

Fig. 1. Experemental sheme

[1] The work was supported by the Russian Science Foundation (project No. 17-19-01169)

EVOLUTION OF THE STRUCTURE AND PROPERTIES OF THE SURFACE LAYER OF HIGH-CHROMIUM STEEL IRRADIATED WITH A PULSED ELECTRON BEAM OF SUBMILLISECOND DURATION[1]

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The purpose of the present work is the analysis of structure, mechanical and tribological properties of the high-chromium 420 steel (USA analogue; 20Cr13 – Russian analogue) irradiated by an intensive low-energy pulsed electron beam submillisecond duration. The radiation of steel is carried out by electron beam with pulse duration of 450 μs on COMPLEX installation [1]. The electron beam energy density and pulse number were (23-43) J/cm^2 and 3, respectively. The research of phase and elemental composition, state of a defective substructure were carried out by methods of the scanning and transmission electron microscopy; the phase structure and state of a crystal lattice were studied by methods of X-ray phase analysis; the mechanical properties of the irradiated surface were characterized by microhardness; tribological properties were characterized by wear resistance.

It has been shown that the 420 steel electron-beam treatment in the surface layer melting mode accompanied by dissolution of the particles of the initial carbide phase of the $M_{23}C_6$ ((Cr, Fe,)$_{23}C_6$) composition, the saturation of the crystal lattice of the surface layer by chromium and carbon atoms, the formation of dendritic crystallization cells (Fig. 1, a), the release of nanoscale chromium carbide particles, and the formation of a martensitic structure (Fig. 1, b), regardless of the energy density of the electron beam (23-43 J/cm^2). In total it allowed to increase the hardness and wear resistance of the surface layer of steel. It has been found that the microhardness and wear resistance of irradiated steel reaches its maximum values at an electron beam energy density of 36 J/cm^2. The microhardness of irradiated steel exceeds the microhardness of steel in the initial state by 1.6 times; wear resistance - more than 50 times.

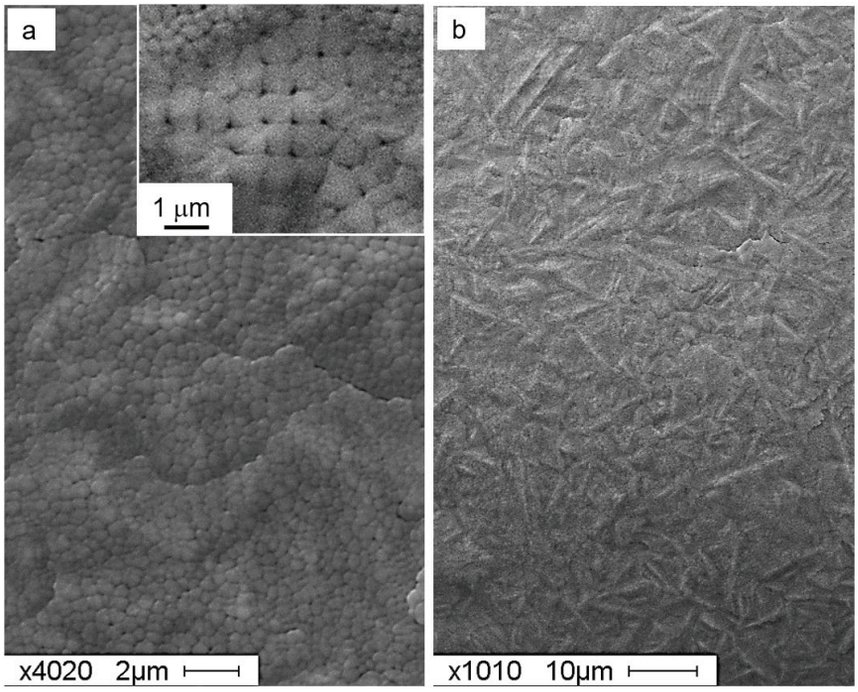

Fig. 1. Surface structure of 420 steel specimen irradiated with an intensive pulse electron beam with pulse duration of 450 μs and electron beam energy density of 36 J/cm^2. Scanning electron microscopy.

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TRIBOMECHANICAL PROPERTIES OF THE SURFACE OF INSTRUMENTAL STEEL AFTER LASER MODIFICATION

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Reducing wear of machine parts and metalworking tools is one of the main problems of machine building. Numerous studies are devoted to the search for effective ways to solve it. Laser treatment in air can be considered as one of the ways which is able to provide a reduction in wear rate of the contacting surfaces. The presence of a hardened layer in the contact area of the details reduces the adhesive bonds of the contacting surfaces, preventing them from grasping, and also reduces wear, and increases the durability of machine parts.

As a rule, tribomechanical properties of the hardened surface of the material are considered at pressure in the contact zone not exceeding 10 MPa. A higher initial contact pressure of 300 MPa [1] was achieved by loading according to the scheme "plane – roller." However, due to the chosen loading scheme, the contact pressure did not exceed 50 MPa at the end of the test, which is not commensurate, for example, with stresses while cutting metals.

In connection with the foregoing, the purpose of this work was the experimental evaluation of the adhesion component of the friction coefficient in contact with tool and structural steels, after laser treatment of the contact surface, at high values of normal contact stresses.

The study of adhesion characteristics of the contact surface was carried out on the diagnostic complex described in [2], at the basis of which the physical model is based on the method by I.V. Kragelsky [3]. According to this model, a sample-indenter in the form of a truncated cone (made of tool steel) is inserted into the cylindrical sample, after which a normal load (N) is applied to the cone, and the cylinder is rotated about its own axis, while the cone-shaped sample remains stationary. The force consumed by the rotation of the cylindrical sample (F) is mainly related to the shearing strength of the adhesive bonds, since the deformation component of the tangential forces is negligible. The adhesive component of the coefficient of friction is defined as the ratio of the average tangential shearing stress (τ_n) to the value of the normal contact stresses (p_n) from the experimentally measured values of the forces N and F.

In experiments, the contact interaction between the cylindrical sample made of structural carbon steel 20 (USA, ASTM analog – steel 1020) and the cone made of alloy tool steel 9XC (Germany DIN standard – 150Cr14), blanking steel X12M (USA analog – D2, Germany DIN standard – X165CrMoV12) and carbon tool steel У7 (USA analog – W1-7, Germany DIN standard – C70W2) was simulated. Laser treatment was carried out in the air on the forming surface of the cone with an energy density of 2,2-2,4 J/mm^2. In this case, the depth of zone of laser influence of about 65-70 microns was provided, and microhardness was increased up to 9 GPa, compared to 6.5-7.0 GPa at the base material. The calculated value of normal stresses at the contact was in the range of 120-135 MPa.

The results of the experiments showed that laser treatment of the contact surface leads to decreasing the friction coefficient at the contact "tool and work materials" by 45-50% at normal contact stresses up to 135 MPa. These results are in good agreement with the data of [2], where the tribomechanical characteristics of the contact pairs steel 20-P6M5 (USA analog – M2), steel 20-P18 (USA analog – T1) under the analogous loading conditions were analyzed. The established reduction in the friction coefficient is an essential factor ensuring high performance characteristics of hardened parts of machines and metalworking tools, working in conditions of high pressure contact.

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INFLUENCE OF HYDROGEN CONTENT IN WORKING GAS ON GROWTH KINETICS OF HARDENED LAYER AT ION NITRIDING OF STEELS

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At present, increasing the wear resistance of machine parts is an important task. Nitriding in a glow discharge is an effective method of surface hardening [1]. However, ion nitriding is a long-time process (25-30 h). It is known [2-4] that when nitriding in a glow discharge, the composition of the gas atmosphere has a significant effect on the structure, properties, and growth rate of the strengthened layer. Thus, the regulation of working gas composition makes it possible to increase the growth kinetics of a strengthened layer and this is an actual problem.

The aim of this work is to study the effect of hydrogen content in working gas on the growth kinetics of a hardened steel layer during nitriding in a glow discharge.

The samples of austenitic, martensitic and perlitic steels were subjected to ion nitriding. The treatment was carried out in a mixture of nitrogen, argon and hydrogen gases at 550° C for 6 hours. The hydrogen content in the working gas was varied from 10 to 30%. Measurements of microhardness profiles were carried out. It is established that increasing of hydrogen content in gas mixture makes it possible to significantly increase the microhardness and kinetics of growth of a nitrided layer on high-chromium steels (Fig. 1).

The microstructure of the samples after nitriding in different compositions of the working gas is represented by a clearly revealed diffusion zone and a core microstructure. When processing high-chromium steels, from 10 to 20% of hydrogen content in working gas does not cause hydrogen embrittlement. When nitriding in atmosphere with hydrogen content from 25 to 30%, microcracks and chips are observed in near-surface areas.

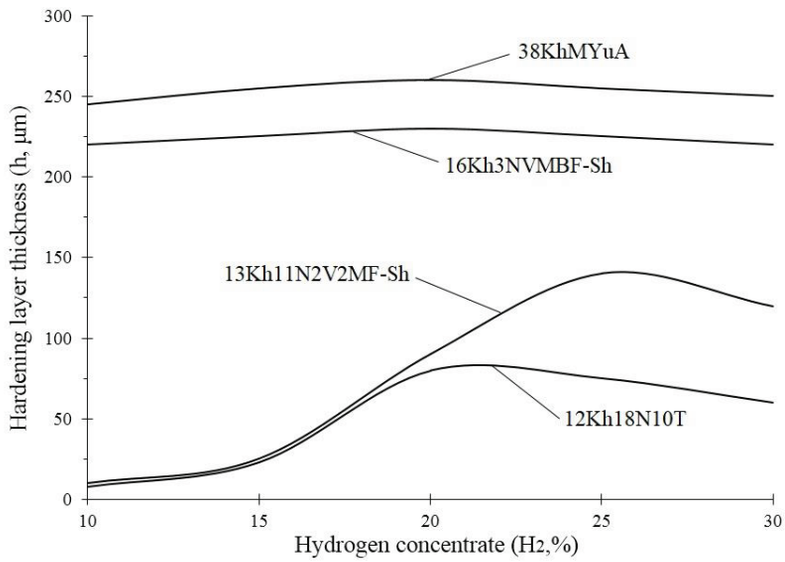

Fig. 1. Dependence of the hardened layer thickness on the hydrogen content in a working gas for ion nitriding.

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LOW TEMPERATURE ION NITRIDING TITANIUM ALLOY Ti-6Al-4V IN THE COARSE GRAINED AND ULTRAFINE-GRAINED STATES[1]

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Titanium alloys have wide application among structural materials in the aviation and aerospace industries due to their high specific strength and corrosion resistance. Nonetheless, the low antifriction characteristics and low hardness limit wide applications of titanium alloys. One of the promising directions for increasing strength of structural materials is severe plastic deformation (SPD). However, ion nitriding is usually performed at a very high temperature 800-950° C [1, 2]. With such thermal effects, processing of titanium alloys with an ultrafine-grained (UFG) structure is not possible because the UFG structure is thermally unstable and grain growth occurs due to the recrystallization process. It leads to decreasing of mechanical characteristics of processing material. Therefore, it is necessary to carry out ion nitriding at relatively low temperature.

The aim of this work is to study the effect of low-temperature ion nitriding on the mechanical and operating properties of Ti-6Al-4V titanium alloy.

A low-temperature ion nitriding of a two-phase coarse-grained Ti-6Al-4V titanium alloy (thermal treatment: annealing at a temperature of 750° C) and UFG states was carried out. To determine the effect of the process temperature on the kinetics of modified layer growth, nitriding was carried out in a temperature range 450-600° C in an atmosphere of 15% N 2 and 85% Ar at a pressure of 150 Pa for various processing time. UFG structure was obtained by equal-channel angular pressing (ECAP) under different processing parameters. In the first mode, samples were subjected to two pressing cycles at a temperature of 700° C, in the second mode to five cycles at 600° C.

To determine the effect of the nitriding temperature on the mechanical properties, as well as to determine the depth of the nitrided layer, microhardness measurements of modified layer were made. The microstructure was studied using optical and scanning electron microscopy. Also, the grain size was measured before and after nitriding by scanning electron microscopy. To determine changes in the tribotechnical properties of the surface, the wear resistance tests were carried out.

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EFFECT OF PULSED ELECTRON BEAM EXPOSURE ON STRUCTURE AND DIFFUSIVE PROPERTIES OF THE ULTRAFINE-GRAINED MOLYBDENUM[1]

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In the process of electron beam exposure without melting the rate of radiation-induced defect formation in metallic materials (as a rule, at temperatures below $0.4T_{melt}$) is higher than the rate of their annealing [1]. Under these conditions, the accumulation of radiation-induced defects in material occurs. In metallic polycrystals, the sinks of various types of defects, including radiation-induced ones, are the grain boundaries [2]. Accumulation of defects in grain boundaries can lead to the energy fluctuating and misorientation of grain boundaries and, as a consequence, change in their kinetic characteristics (migratory mobility, diffusion permeability, et al.) [3].

In this paper, we experimentally studied the effect of pulsed electron beam irradiation in the regime of melting absence on the structural changes and diffusion development in the ultrafine-grained molybdenum obtained with the use of severe plastic deformation.

Surface irradiation by pulsed electron beams without melting is found to activate the grain boundary migration and reduce the temperature of grain growth beginning in the ultrafine-grained molybdenum by 150-200 K as compared to vacuum annealing. Grain growth under the electron beam exposure leads to decrease in the fraction of grain boundaries with misorientations $\theta < 4°$ and increase in fraction of grain boundaries with misorientation angles 45-55° in the grain boundary ensemble of the ultrafine-grained molybdenum.

Pulsed electron beam irradiation at temperature below the temperature of the grain growth beginning results in a rise in dislocation density in the surface layer of the ultrafine-grained molybdenum by almost an order of magnitude from $5 \cdot 10^{13}$ m^{-2} to $2.5 \cdot 10^{14}$ m^{-2}, and to an increase in the microdistortions of crystal lattice from $1.2 \cdot 10^{-3}$ to $5.8 \cdot 10^{-3}$. This indicates the appearance of additional internal stresses. At the same time, fraction of low angle boundaries with misorientations $\theta < 4°$ increases in the grain boundary ensemble of the surface layer. In the main bulk of irradiated molybdenum dislocation density and value of the crystal lattice microdistortion decrease to $\sim 10^{13}$ m^{-2} and $\sim 8 \cdot 10^{-4}$, respectively, as compared to initial state. This is evidence of tempering of deformed structure of main material bulk under the simultaneous temperature and irradiation exposure.

The width of the heat penetration of the ultrafine-grained molybdenum was estimated on the basis of a mathematical model of the material electron-beam treatment. Effect of the pulsed electron beam treatment parameters on the depth and character of the diffusion and stress fields was demonstrated.

Profiles of the nickel concentration distribution in the ultrafine-grained molybdenum were experimentally determined after isothermal diffusion annealing and surface irradiation by pulsed electron beam at a temperature of 923 K. From the data obtained it follows that as a result of electron beam irradiation the mutual penetration of the atoms of the diffusing element and the substrate becomes more active in the surface layer and the diffusion regimes in the grain boundaries are changed in comparison with regimes observed under the isothermal diffusion annealing. These changes are shown to be associated with an increase in dislocation density in the surface layer and appearance of additional internal stresses caused by pulsed electron beam exposure.

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FORMATION FEATURES OF COMPOSITE COATINGS BASED ON TITANIUM NITRIDE BY METHOD OF VACUUM-ARC EVAPORATION AND MAGNETRON SPUTTERING[1]

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The paper studies the formation conditions, structure and properties of composite layers based on TiN which obtained by combining different growth processes in the vacuum chamber. The composite layers deposition was carried out in a modernized installation [1] equipped a vacuum-arc evaporator and a planar magnetron (Fig. 1). Some results on the composite coatings TiN-Cu synthesis have been received. The presence of a separation diaphragm inside the vacuum chamber is a distinctive feature of the vacuum installation. In the first place, the mutual influence of different forms of discharges (vacuum-arc and magnetron) on their steady-state combustion does not allow the diaphragm, and secondly, the diaphragm will prevent the ingress of titanium vapors on the copper cathode of the magnetron. The synthesis of TiN-Cu composite coatings on the substrate takes place with their accompanying modification by means of bombardment of the plasma-forming gas (nitrogen) and titanium low-energy ions.

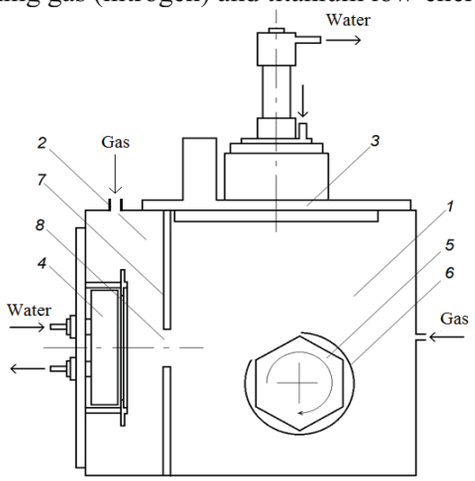

Fig. 1. The schematic diagram of the plasma-chemical reactor: 1-compartment of chemical reaction between Ti and N, 2-compartment of Cu vaporization, 3-arc evaporator of Ti, 4-planar magnetron with the copper cathode, 5-substrate holder, 6-shield, 7-diaphragm, and 8-metering orifice

The composition of the reaction gas (mixture of nitrogen and argon or nitrogen), the pressure in the chamber and the time of deposition varied during the experiments on deposition of the composite TiN-Cu layers. The optimal values of arc current (I_A=80 A) and current of the magnetron discharge (I_m=0.9 A) were determined in the course of earlier studies and their values did not change. The optimal distances of the evaporator cathode – substrate (230 mm) and magnetron cathode – substrate (190mm.) [2] have been determined proceeding on the design features. This allow to achieve deposited layers distribution uniformity along the substrate. The layers thickness of composite TiN-Cu was 5-7 μm.

According to X-ray analysis, in the composite layer there are reflexes of TiN and the reflexes of copper with the intensity of about 1-2%. The surface of the composite has a rather homogeneous structure with minimal inclusions of the drop phase.

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ELECTRON-BEAM SYNTHESIS OF GRADED METAL-CERAMIC MATERIALS IN THE FOREVACUUM PRESSURE RANGE[1]

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Ceramic functional graded materials are used for making cutting tools in instrument manufacture, and they are also in demand in other industries. Additive printing technologies, such as the LOM method, selective laser sintering (SLS) or melting (SLM), are increasingly being used to create gradient materials [1], in addition to traditional heat treatment technologies. An alternative method of creating functionally graded materials is layer-by-layer electron-beam sintering. The electron beam, in terms of the specific power, the efficiency of energy transfer, as well as the possibility of independent regulation and control of its parameters, is much superior to other sources of high-energy impact. Using electron sources functioning in the forevacuum range of pressures makes it possible to solve the problem of electron beam processing dielectrics [2].

The previously obtained results, such as electron-beam sintering of ceramics, deposition of dielectric coatings by evaporation of ceramics, layer-by-layer synthesis of ceramic products, indicate the successful application of this method for the production of functionally graded metal-ceramic materials. The first experiments on the sintering of a non-pressed layer of a metal-ceramic powder, consisting of a different ratio of alumina and titanium, were conduct according to the scheme shown in Fig. 1.

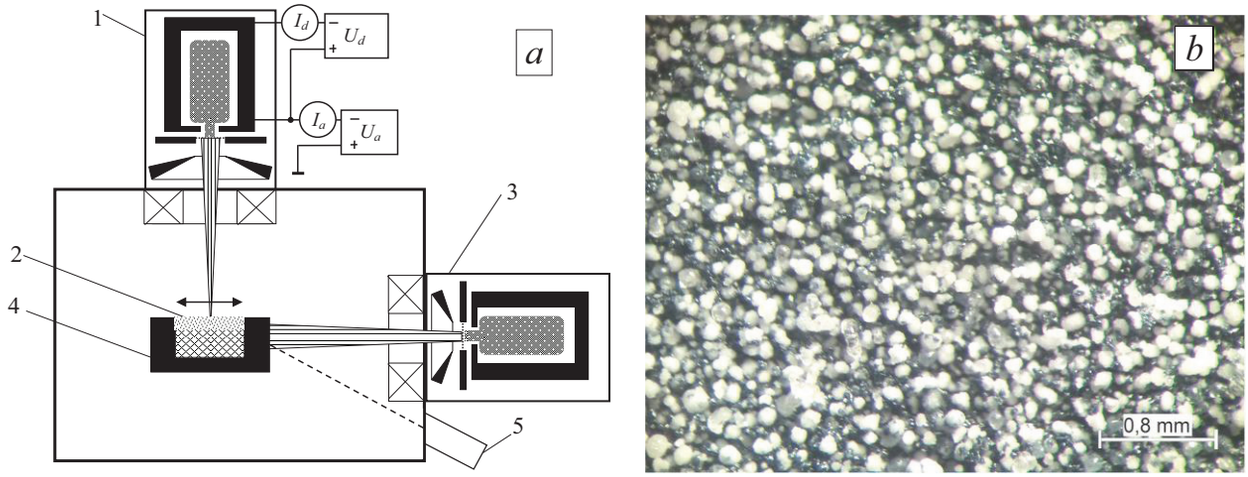

Fig. 1. The scheme of the experiment *a* and the result of sintering *b*. 1 – plasma electron source-1; 2 – metal-ceramic powder; 3 – plasma electron source -2, 4 – graphite crucible; 5 – pyrometer.

The electron source 1 was used for local exposure to powder, and the electron source -2 was used to prevent the powder particles from spreading due to gas desorption from its surface. As a result of half-hour irradiation, it was possible to obtain the sample with the thickness of 2 mm and the diameter of 10 mm, which is not destroyed during manual manipulations. Further research is planned to be directed to the search for electron-beam exposure modes contributing to obtaining a more dense structure of samples.

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OBTAINING AL / SIC COMPOSITE FROM THE PRODUCT OF SI-C SYSTEM DC ARC DISCHARGE SYNTHESIS[1]

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One of the most important problems of high-power and medium-power power electronics is the removal of thermal energy. For this purpose, various materials with relatively high thermal conductivity and a low coefficient of thermal linear expansion are used. One such material is a composite consisting of an aluminum matrix Al infilled with SiC carbide particles. Excellent thermophysical properties [1] of this material allow it to be used for the manufacture of a number of power electronic devices [2], as well as brake pads for ultra-high speed vehicles [3]. A known method of obtaining such materials is the spark plasma sintering of SPS [4].

In the present work, a series of experiments was conducted to obtain an Al / SiC composite material. As raw materials, commercial Al powder and synthesized by the authors in a DC arc discharge plasma SiC were used. In the experiments, the mass fraction of SiC was varied. The specimens were sintered on spark plasma sintering Advanced Technologies SPS-10-4 system at temperature of 560 °C, at pressure of 60 MPa for 10 min. After sintering, the surface of the samples was cleaned for conducting analysis. A typical X-ray diffraction pattern of a sintered specimen is shown in Fig. 1. Two crystalline cubic phases of Al and SiC are identified.

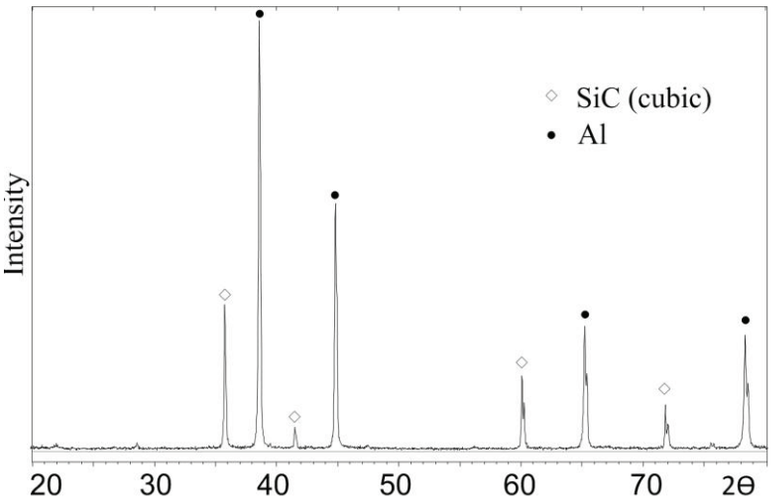

Fig. 1. A typical XRD pattern of a sintered sample

The analysis of the thermal conductivity of the obtained specimens on the DLA-1200 TA thermal diffusivity analyzer was carried out. At this stage of the study it was possible to obtain the highest value of the thermal conductivity about 130 W / (m · K) with a mass content of microcrystalline SiC of 25%. The volume fraction of SiC according to XRD was 21%. At this SiC fraction content, information on a higher level of thermal conductivity of about 160 W / (m · K) is presented in the current literature. The reason of the lower thermal conductivity of the obtained samples probably can be pores. The relative density of the resulting Al / SiC material is ~96% (relative to the calculated single crystal). Further research will be directed to a systematic study of the issue of increasing the thermal conductivity of the samples.

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INTRODUCING OF HYDROGEN INTO TITANIUM BY PLASMA METHODS

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Modification of metals by the introduction of hydrogen is important in materials science in terms of studying the mechanisms of hydrogen embrittlement of materials and coatings. In the present work, hydrogen was introduced from 2 types of plasma: 1) hydrogen plasma of a high-frequency discharge, while the saturated sample was "suspended" (not grounded and not energized) in the plasma generator; 2) gas-discharge plasma based on the source PINK [1] at the facility [2], while the hydrogen ions were extracted from the plasma and accelerated. The ion beam focused in the equipotential drift space of the plasma-immersion system located at a pulsed periodic bias with an amplitude of –1.5 kV, for multiple increase of the current density.

The aim of the work was to study the mechanisms of hydrogen storage in titanium during irradiating by low-energy ions with high-intensity pulsed-periodic beams and, for comparison, from a hydrogen plasma of high-frequency discharge (HFD). For the study, samples of titanium VT1-0 with sizes 20×20×1 mm were prepared. The surface of titanium samples prior to irradiation with hydrogen was mechanically ground and polished to remove near-surface oxide films.

Fig.1 obtained by the thermally stimulated desorption (TSD) method. The figure shows the temperature spectra of H_2 hydrogen yield (TSHY) from the samples saturated with two methods: irradiation of high-intensity pulsed-periodic beams of low-energy ions at different interstitial parameters (1-Ti_1, 2-Ti_2). Ti_1: current density – 0.11 A/cm^2, dose –7.4·10^{20} ion/cm^2, sample temperature – 360 ° C and Ti_2: current density – 0.17 A/cm^2, dose – 1.1·10^{21} ion/cm^2, sample temperature – 390 ° C). From the HFD plasma (curve 3): the pressure in the plasma reactor ~10^{-1} mm Hg, the sample temperature – 400 ° C, the saturation time – 95 min.

In the TSHY from the Ti_1 sample (curve 1) two peaks are observed, one low intense, corresponding to

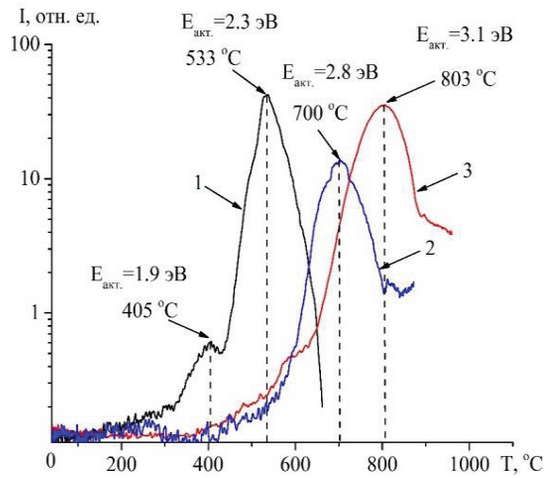

Рис 1. TSHY from the samples of the titanium alloy VT1-0 saturated with different methods: 1, 2 - samples after irradiation with hydrogen ions from PINK plasma; 3 - from HFD plasma

a temperature of 405 °C and one highly intense at a peak temperature of 533 °C. That corresponds to the desorption activation energies of 1.9 eV and 2.3 eV. In the TSHY from Ti_2 sample, a peak observed at 700 °C, with an activation energy of 2.8 eV. The difference between the behavior of curve 3 from 1 and 2 is explain by the mechanism of hydrogen penetration, which consists in the following. When irradiated with low-energy hydrogen ions, most of the ions are reflected from the surface and do not pass into the volume. The HDR plasma "surrounds" the sample surface at thermal energies, and hydrogen captured by surface defects with subsequent diffusion to volume defects. Significant differences in the TSHY explained by the capture of hydrogen by different types of traps. It can be assumed, that the temperature of 405-533 °C corresponds to the capture of hydrogen by surface defects, 700 °C - volume vacancies, 803 °C - interstices and vacancy clusters.

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MECHANISMS FOR THE STRENGTHENING OF SILUMINS[1]

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The development of a new class of wear-resistant nanocomposite materials based on silumin eutectic composition was the purpose of this work. Alloys have been formed by irradiating of the silumin surface with high-intensity electron beam of micro- and submillisecond exposure times in surface layer melting mode. A submicro-nanocrystalline multiphase structure has been formed in a surface layer of thickness up to 500 μm (Fig. 1) as a result of high-speed crystallization. The irradiation has been carried out in argon at a gas pressure of 0.02 Pa. The industrial antifriction eutectic aluminum-based alloy (silumin) of the following composition (in at.%) have been used as the study material: 12.49% Si, 2.36% Mg, 0.6% Cu, 0.35% Ni, 0.3% Fe, Al.

To determine the possibility of obtaining in a particular alloy a given level of properties and methods for improving them, it is necessary to understand the mechanisms of hardening, the knowledge of the factors that control these mechanisms, and their effect on strength and plasticity.

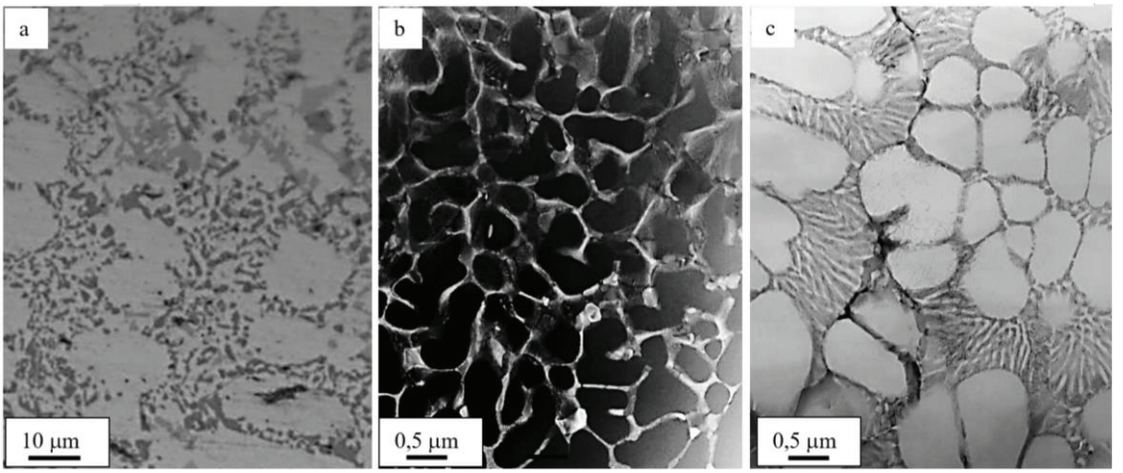

Fig. 1. Structure of the silumin of the eutectic composition in the cast state (a) and after irradiation with an intense pulsed electron beam (b, c).

A qualitative and quantitative analysis of the hardening mechanisms based on the results of quantitative estimates of the silumin structure parameters treated by a pulsed electron beam under different exposure regimes have been carried out.

It was found that a model based on the application of the rule of a mixture, the constituent elements of which are structural components: aluminum based grains of a solid solution and Al-Si eutectic is the most suitable for describing hardening of eutectic alloys[1-3].

In this case, the equation describing the dependence of the yield stress on the contributions of structural characteristics is as follows:

$$\sigma_{0,2} = (1 - f_{eut})(\sigma_{ss} + \sigma_\rho + \sigma_{gb}) + f_{eut}(\sigma_{ss} + \sigma_p + \sigma_\rho),$$

where f_{eut} — is the volume fraction of the eutectic (in the investigated case, f_{eut} = 89%); σ is the hardening value, MPa (σ_{ss} is solid-solution, σ_ρ-dislocation, σ_{gb} is grain-boundary, σp from the second-phase particles).

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Absorption of Light by YAG:Nd Nanopowder-Based Laser Ceramics Irradiated by Pulsed Electronic Beam

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In this work we study optical absorption of YAG:Nd single-crystal and YAG:Nd-based optically transparent ceramic samples. The samples of YAG:Nd ceramics were made in IEP UD RAS by the method of solid-state reaction sintering of nanopowders with an additional annealing of oxide mixture before compaction. As a material for ceramics fabrication, we used nanoscale particles of $Nd_{0.02}Y_{1.98}O_3$ и Al_2O_3 produced by the method of laser ablation. The samples of single-crystals were Czochralski-grown. Spectra of optical absorption of the samples were measured in the range of 200-1100 nm using spectrophotometer. Lattice defects in YAG:Nd are F and F^+-centers which absorb radiation at ~162 and ~182 nm, respectively, and interstitial ions Al^{3+} located near cation and anion vacancies, which absorb at ~260 nm. We observe fivefold increase of absorption at ~260 nm in ceramics in comparison with single-crystal sample. This fact shows that there is a large amount of vacancy defects in subsurface layer of nanoparticles. Induced absorption in YAG:Nd samples was studied using an electron accelerator, a source of light and an optical registration system. We obtain a structureless spectrum of induced absorption after irradiation of the samples by 25-ns high-current pulsed electron beam with an average electron energy of ~0.25 MeV. Optical density slightly increase from ~360 nm to ~730 nm with a sharp decline at ~1200 nm. Probably, the crystal was doped by the ions of Cr, Fe, Ce from the materials of the equipment during the growing process. Radiation-induced color centers formation at electron beam irradiation is described by the model which takes $Me^{3+} \rightarrow Me^{4+}$ transition consideration. When $Me^{4+}:[O^{2-}]_6$ cluster captures an electron, it forms a system which is similar to localized exciton. The curve of relaxation of induced absorption signal for ceramic sample demonstrates significantly faster decrease than that for crystal. Luminescence spectrum of the samples in the range of ~230–900 nm consists of narrow lines which correspond to intracenter electron transitions in Nd ion. We observe a faster decrease of optical signal in the ceramic sample in comparison with single-crystal sample. We suppose that a large amount of lattice defects in ceramics sample leads to multiple capture of the charge carriers which prevents their transfer within nanoscale YAG:Nd crystals.

INFLUENCE OF HIGH-INTENSE PULSED ION IRRADIATION ON OPTICAL PROPERTIES OF AL–SI–N NANOCOMPOSITE COATINGS[1]

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Increase of radiation resistance (RR) of materials, used for creation of electronic devices, which keep operational parameters under radiation for a long time, stimulates the investigations of changes of its electronic properties after irradiation and the optimization of technologies of its production. Nanocomposite coatings Al–Si–N have not only high RR but are optically transparent [1] and they represent a new class of coatings for protection of the glass and the dielectric materials, used in devices under the radiation. These coatings consist of two or more separated phases with nanocrystalline and amorphous structure [1]. The growth defects (GDs) have a dominant influence on properties of Al–Si–N. The aims of this work were to study the optical properties of Al–Si–N coatings, deposited by reactive magnetron sputtering, and to determine the energetic characteristics of GDs and radiation defects (RDs), induced by high-intense pulsed ion beam (HIPIB) irradiation (pulse duration – 90 ns, accelerating voltage – 200-230 kV, beam composition: carbon ions – 85%, protons – 15%) [2], and to identify the nature of GDs and RDs and to assess the degree of their influence on RR of coatings.

The property changes of Al–Si–N after HIPIB were caused by the accumulation of RDs whose energetic levels were continuously distributed in band gap in interval 1.3–3.6 eV. RR of Al–Si–N coatings was increased with the increasing of silicon content in them. The changes in parameters of band structure of Al–Si–N and in energetic characteristics of the defects levels after HIPIB were negligible. The local levels of absorption and luminescence were identified with points RDs, located in the crystallites of aluminum nitride and in the intercrystallite interlayers consisting from amorphous silicon nitride. RDs had the concentration $(1–20)\cdot10^{18}$ cm^{-3} and they revealed the different kinetics of its accumulation and annealing. Radiation and thermal annealing, which was peculiar to HIPIB, stimulated the annihilation of unstable RDs with energetic levels at 2.0–3.0 eV. The general patterns of the defect formation in Al–Si–N and in alternative dielectrics with different properties and structure were established. The RR of coatings Al–Si–N to HIPIB had exceeded its value for the boron carbide films and the plates of quartz and calcium sodium silicate glass. Besides, the RR of coatings Al–Si–N was comparable with resistance of aluminum nitride and silicon nitride films and plates of pyrolytic boron nitride and ceramics silicon carbide. The main reasons of high RR of Al–Si–N were the high concentration of GDs at $(0.1–2)\cdot10^{18}$ cm^{-3} and the strong interaction between the defect levels due to electronic exchange and the big band gap value at 5.0 eV and the very broad absorption edge, formed by the localized defects states at 1.5–3.4 eV. Nanoscale structural component gave also the certain contribution into RR of coatings due to the spatial localization and recombination of charge carriers.

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COMPOSITION OF CATALYTIC LAYERS PREPARED BY ION BEAM ASSISTED DEPOSITION OF PLATINUM AND YTTERBIUM ON CARBON FIBER PAPER CATALYST CARRIERS [1]

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Surface layers were prepared by ion beam assisted deposition (IBAD) of catalytic metal – platinum and ytterbium as activating additive on the carbon based Toray Carbon Fiber Paper TGP-H-060 T (TorayCFP) and AVCarb® Carbon Fiber Paper P50 (AVCarbCFP) catalysts carriers. The basis of the carriers AVCarb® Carbon Fiber Paper P50 and Toray Carbon Fiber Paper TGP-H-060T are fibers of polyacrylonitrile, which undergoes thermooxidative stabilization and subsequent carbonization. They have an irregular porous structure. The carrier of Toray Carbon Fiber Paper TGP-H-060T is hydrophobized with polytetrafluoroethylene, the carrier of AVCarb® Carbon Fiber Paper P50 is not hydrophobized.

The deposition method is characterized by the use of deposited-metal ions as assisting ions. Deposition of metal and mixing of the deposited layer with the substrate surface by accelerated (U = 5 kV) ions of the same metal have been carried in the experimental unit, respectively, from neutral fraction of metal vapor and ionized plasma of vacuum pulsed electric arc.

Investigation of the composition and morphology of prepared layers was carried out by RBS, SEM, EDX and XRF methods. According to EDX, XRF and RBS (Fig. 1) results of investigations atoms of deposited metals, carbon substrates, and oxygen as impurity enter into the composition of layers. At the same time, the surfaces contain deposited metals (Pt, Yb) inclusions with sizes of several micrometers (Fig. 2), which arise from the precipitation of metal droplets from the arc discharge of the ion source.

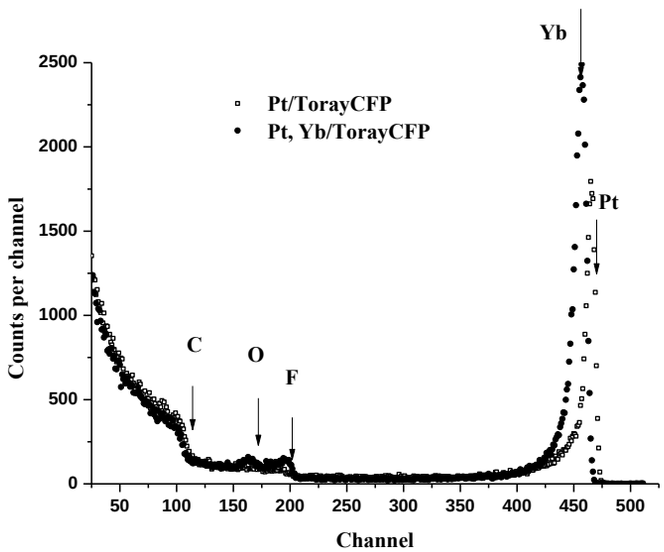

Fig. 1. Rutherford backscattering spectra of ^{4}He ions from surfaces of investigated samples. E_0 = 1,5 МэВ

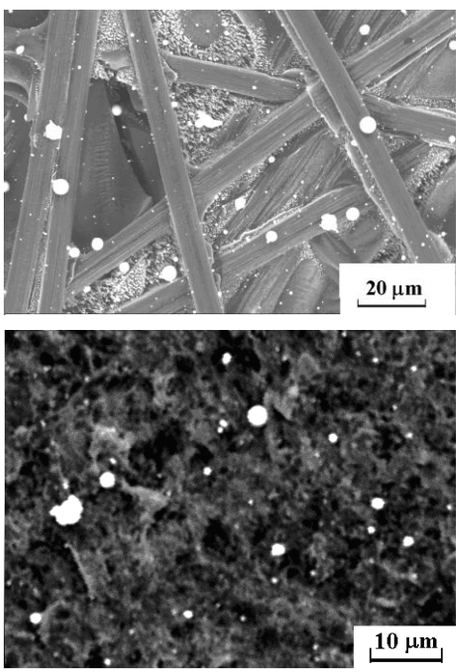

Fig. 2. SEM images of the layer formed by the IBAD of Pt and Yb on TorayCFP and AVCarbCFP catalysts carriers

The thickness of the prepared layers is ~30 nm; content of each of deposited metal atoms in the layers – ~2×10^{16} cm^{-2}. In the distribution maximum located at near the surface the concentration of each from deposited metals is several atomic percent. In the process of ion-assisted deposition of metals in the proposed mode, ionic mixing of all components of the layer being formed takes place.

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THE EFFECTS OF ELECTRON RADIATION ON POLYMERIC MATERIALS IN SPACE[1]

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One of the main problems associated with the implementation of long-term manned space flights is the protection of astronauts from space radiation. Some experts believe that metal screens will help protect the crew of the spacecraft from streams of charged particles. However, other scientists are of the opinion that it is much more efficient to use polymers for these purposes. The fact that the metal contributes to the appearance of secondary radiation caused by the impact on the body of the spacecraft cosmic radiation.

The available literature information on the behavior of polymer materials in flight on space station "Mir" were obtained under conditions of simultaneous exposure to multiple factors radiation exposure, which is almost impossible to fully simulate in conducting the accelerated laboratory tests. Therefore, to determine the radiation resistance of polymers we used an electron beam. The pulsed high-current electron accelerator was used as a source of pulsed electron beam for processing polymers with the characteristics: accelerating voltage 90 kV, the number of pulses 4 per second, the processing time 15 minutes. As objects of research for polymers containing heteroatom: polyimide, polyetherimide, polysulfone, polyethersulfone, polyetheretherketone, polyvinylcarbazole and its modifications.

IR spectroscopy methods found that changes in the surface properties of polymers due to the flow of oxidative processes, the destruction of macromolecules, as well as the decay of image cycles and aromatic rings of polyimide. The least resistant to the influence of electron irradiation were polymers containing in their molecule oxygen.

[1] The work is performed in the framework of the State task for HCEI SB RAS (project #9.5.2).

THE RELAXATION OF ELECTROPHYSICAL PROPERTIES OF MCT EPITAXIAL FILMS AFTER INFLUENCE OF A HIGH FREQUENCY NANOSECOND VOLUME DISCHARGE IN ATMOSPHERIC PRESSURE AIR [1]

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In modern times, infrared photoreceivers and devices based on them are widely used. The progress achieved in the study of the infrared range has led to the creation of a variety of medical, military, scientific and industrial techniques. A wide range of semiconductor materials exist to create photonic IR receivers, but the most promising is the triple bond of mercury cadmium telluride ($Hg_{1-x}Cd_xTe$), due to its large spectral range and low carrier concentration at operating temperatures. Much attention is paid to the development of multielement photodetective matrices based on MCT films, grown by molecular beam epitaxy. The main problem is the production of films of large area and uniformity. The task of purposefully changing the parameters of the material remains relevant. One of the most common methods of changing parameters is radiation methods. Recently for modification of near-surface layers of various materials discharges of various types and electron beams began to be used. The first experiments on the effect of low-frequency discharge [1, 2] showed the possibility of using this method in relation for MCT. The effect exerted on epitaxial films under the action of a volume discharge is complex, but while each of the effects is individually well studied, the complex effect in the literature is little studied, and is of interest in further research.

To achieve this goal, studies of prepared epitaxial films of p-type conductivity ($p = 7 \div 8\times10^{16}$ cm^{-3}, μ_p = 400 $cm^2V^{-1}s^{-1}$) were carried out, grown by molecular beam epitaxy in the Institute of Semiconductor Physics Siberian Branch of the Russian Academy of Sciences (ISP SB RAS) in Novosibirsk. The composition of the working layer of epitaxial films was x = 0.22. Samples were subjected to a high-frequency volumetric nanosecond discharge in air at atmospheric pressure. Prepared MCT samples were irradiated with a volume nanosecond discharge in a pulse-periodic mode with a repetition rate of 1200 Hz. The duration of exposure was 30 seconds, 1, 2, 5, 10 and 20 minutes. The electrophysical parameters of the MCT samples before and after the discharge action were determined from measurements of the Hall effect by the Van der Pauw method. The measurements were carried out at a constant current flowing through the sample (I = 1 μA) for two directions of the current and two directions of the constant magnetic field.

From the experimental data we made conclusion that with an increase in the irradiation time t significant changes are observed both in the value of the Hall coefficient R_{hall} and in the behavior of the field dependence. At t = 30 and 60 seconds, there is a decrease in R_{hall}, at t = 120 and 300 seconds there is an alternating dependence of R_{hall}. For samples 5 and 6 irradiated 600 and 1200 seconds, respectively, a complete inversion of the sign of R_{hall} is observed in comparison with the initial values.

According to the results of the studies carried out, it can be concluded that with an increase in the discharge time, a significant change is observed, both in the value of the Hall coefficient, and in the nature of the dependence on the magnetic field. The observed changes in the field dependence of irradiated samples can be explained by the formation of a high-conductivity layer of the n-type conductivity in the near-surface region of the material, and with increasing exposure time the integral conductivity increases. It should also be noted that, over time, there is a relaxation of the Hall coefficient to the initial values.

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RED SHIFT OF ABSORPTION SPECTRA OF METAL-DOPED TiO_2 COATINGS

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The effect of metal elements doping (Cu, Fe, Ni, V, Cr) on the shift of the absorption spectra of the titanium dioxide composite coatings was studied experimentally. It is shown that the most significant effect on the red shift of the absorption spectra (up to 470 nm) is observed for Fe-TiO_2 coatings with Fe doping level up to 20 at. %. Fe-TiO_2 and Ni-TiO_2 coatings with doping levels of 15 at. % also have an extended absorption in the visible region (410÷420 nm).

Keywords: plasma, gaseous discharge, ion sputtering, titanium dioxide, composite, absorption spectra.

RADIATION RESISTANCE OF MULTILAYERED THIN FILMS UNDER HIGH-INTENSE SHORT-PULSED ION BEAM IMPACT[1]

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A major of latest works demonstrates enhanced radiation stability of nanostrucutred materials under ion (or electron) irradiation. It occurs due to increased amount of interfaces and grain boundaries which serves as effective sinks for primary radiation defects. Formation of multilayered films with altered phases undissolved in each other is a principle novel approach for nanostrucutred radiation resistant materials production. In the present work it was proposed to synthesis of the multilayered thin films with altered amorphous SiN_x phase and nanocrystalline AlN phase. In this case the whole amorphous SiN_x layer can be considered as a sink which has a defects absorption power sufficiently higher than monoatomic interface. The main purpose of the work was the investigation of the structure and phase state changes in the multilayered films after high-intense short-pulsed ion beam impact.

The multilayered AlN/SiN_x films were synthesized by a magnetron sputtering. The thickness of the AlN layers was varied in the range from 10 to 50 nm, while the thickness of the amorphous SiN_x phase was a constant (5 nm) in each film. The total thickness of the films was 30 nm. The formed multilayered films were subjected to high-intense short-pulsed ion beam impact in the TEMP-4M accelerator with the following parameters: ion energy is 30 keV, pulse duration is 80 ns, absorbed energy density is 2 J/cm^2. The treatment was made with 5, 50 and 500 pulses.

The volume fraction of the crystalline AlN phase in the multilayered films was found to decrease with pulse number rising from 5 to 500. This effect can be connected to surface ablation under the ion beam impact, the single-layered AlN films degrading higher. In the multilayered AlN/SiN_x films the ablation process occurs only in the separate layers. Nevertheless, in the AlN/SiN_x multilayered system with a AlN thickness of 10 nm the volume fraction of the crystalline AlN phase increases with pulse number rising. It is a result of competitive process of the ablation in separate layers and defect amount decreasing. Indeed, the primary point defect arose in the films after ion beams impact are absorbed in the amorphous SiN_x layer when it thickness is comparable to the thickness of the AlN layer.

The obtained results showed the increase of radiation resistance of multilayered AlN/SiN_x films with a separate layers thickness in nanometer scale.

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CRACKS FORMATION IN TUNGSTEN AFTER COMBINED COMPRESSION PLASMA FLOWS AND HIGH-INTENSE ION BEAM INFLUENCE

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Having the highest melting point, tungsten is a main candidate to the first wall materials in thermonuclear reactors. So, a lot of strong demands are required for such materials, one of the main of them is a surface erosion resistance. The evaporation, ablation, cracks and craters formation of the surface should be excluded or minimized. The intensity of the mentioned processes depends on the thermophysical properties of the surface, its elastic and plastic parameters, and etc. In this regards, the previous modification of the tungsten surface can effectively influence on its physical properties and decrease the surface eruptions.

In the present work the cracks formation on the tungsten surface after the high-intense short-pulsed ion beams impact is discussed. The previous treatment of the samples by the compression plasma flows was made to modify of the surface.

The tungsten samples were plates with 10×10 mm and thickness of 2 mm. On the first step the samples were treated by compression plasma flows in magnetoplasma compressor of compact geometry. The plasma flows were formed in the residual nitrogen atmosphere. The treatment was made by three pulses with pulse duration of 100 μs. The absorbed energy density equaled to 25-30 J/cm^2. The treated samples were subjected to high-intense short-pulsed ions beams irradiation with pulse duration about 100 ns. The pulse number was up to 80. The absorbed energy density was varied in the range of 1 – 2 J/cm^2.

The surface erosion process after the combined treatment was investigated with weight lost estimation. The influence of the high-intense short-pulsed ion beams without previous compression plasma flows treatment revealed the decrease in the samples weight in 100-200 μg after the first 10 pulses. This change in mass can be contributed to the surface cleaning and different impurities deleting. After all other pulses the weight did not change. It indicates the absence of sufficient surface erosion after the impact. The investigation of the surface morphology with optical microscope revealed the signs of melting after 40 pulses. The melting process is started in the surface roughness.

The previous compression plasma flows impact allows to melt a deep layer with a thickness of several μm. The surface is characterized by big roughness arisen after moving of the melt during plasma influence. At the end of plasma pulse a solidification process with a high cooling rate takes place. It results in a lot of cracks formation due to mechanical stress distribution. The boundaries of the cracks are the places of melting process realization after high-intense short-pulsed ion beams impact. It was revealed disappear of small cracks that is a result of their melting and smoothing of the surface. The analysis of the samples weight showed the change in weight after the first 10 pulses in 100-200 μg that also can be contributed to cleaning process. But in contrast to the ion beams impact without previous treatment, the samples treated by compression plasma flows lost about 200 μg every 20 pulses of ion beams. It indicates the melting process (on the cracks boundaries) and evaporation from the melt state.

Therefore, the provided experiments showed the role of cracks formed by compression plasma flows impact on the erosion process of the surface after high-intense short-pulsed ion beam influence. The latter can be effectively used for decrease in the small cracks on the surface due to their re-melting.

Another approach was realized by alloying of the surface layers of tungsten with other metals. For this purpose a thin coating (depth of 1 – 2 μm) of Nb, Zr or Ti was deposited before the compression plasma lows impact. After the plasma influence the surface layer including the whole coating and a part of the tungsten substrate were melted and mixed. So, after the solidification the modified layer was an alloy with a composition determining by the plasma flows regimes. In this case the thermophysical parameters of the surface are changed and the process of cracks development is decreased. In the work the mechanism of cracks formation and propagation in the alloyed tungsten are discussed.

INVESTIGATION OF THE EFFECT OF SOFT X-RAY RADIATION ON THE ELECTROPHYSICAL CHARACTERISTICS OF EPITAXIAL LAYERS *n*-$Hg_{1-x}Cd_xTe$ [1]

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Earlier it was shown that soft X-ray radiation (SXR) of laser plasma leads to a modification to the modification of surface morphology [1] of single crystals and epitaxial layers of $Hg_{1-x}Cd_xTe$ solid solutions. When studying the capacitance-voltage (C-V) characteristics of MIS structures created on the surface of irradiated epitaxial layers *p*-$Hg_{0.77}Cd_{0.23}Te$ [2] and *n*-$Hg_{0.81}Cd_{0.19}Te$ [3], changes in the carrier concentration and the density of the built-in charge were observed, which can be associated with the generation of radiation defects in the near-surface region during irradiation. The nature of these defects for the studied material has been little investigated. In this work, the influence of SXR on the electrophysical properties of MIS structures based on *n*-$Hg_{0.76}Cd_{0.24}Te$ epitaxial layers is studied.

The investigated heteroepitaxial structures based on *n*-$Hg_{0.76}Cd_{0.24}Te$ were grown by the method of molecular beam epitaxy (MBE) on GaAs (013) substrates in the ISP SB RAS. The working layer 15 μm thick was surrounded on both sides by the graded-gap layers with a thickness of about 0.3 μm with a CdTe content on the surface equal to 0.45. The plasma of a laser-induced vacuum spark containing quanta with energy in the range 0.5÷10 keV was used as a source of SXR. The spectrum of the generated SXR consisted of plasma bremsstrahlung and recombination characteristic radiation of excited ions of cathode material and extended to 10 keV. To cut off visible radiation and fluxes of corpuscular particles, a filter (aluminized mylar with a thickness of 3 μm) was transparent in the region above 0.75 keV. The calculated irradiation doses of the test samples were up to 1.5 J/cm^{-2}. The duration of the X-ray pulse did not exceed 200 ns.

MIS structures were created after irradiation by plasma-enhanced atomic layer deposition of Al_2O_3 insulator onto the epitaxial layers. In wide ranges of temperatures (9-77 K) and frequencies (1-2000 kHz), the admittance of MIS structures was investigated by the methods described in [4]. The measurements were carried out on an automated setup of admittance spectroscopy based on the Janis cryostat and the Agilent E4980A immittance meter.

Determined from capacitive measurements electron concentration at 77 K non-monotonically increases with the irradiation dose. In the absence of SXR irradiation, the electron concentration was 2.6×10^{15} cm^{-3}, and at the maximum dose (1.5 J/cm^{-2}) reached 3.9×10^{15} cm^{-3}. Investigations of the C-V curves showed that the SXR leads to a change in the spectra of the fast surface states of MIS structures. These changes are non-monotonic with increasing doses. The reason for the described effects is the alteration of the impurity-defective system of the insulator-semiconductor interface and the near-surface layer of the semiconductor, the presence of a dose-dependent dependence of the effects indicates their radiative character.

One of the most probable mechanisms for their occurrence is defect formation in the decay of electronic excitations. Probably, the primary effect of SXR interaction with a solid solution of HgCdTe is the excitation of internal electron shells of Hg ions, which is accompanied by an external photoelectric effect. As a result, a triply charged Hg^{3+} ion is formed, whose lifetime is limited by the Auger effect and is estimated at 10^{-13} s. As the model of defect formation assumes in the decay of electronic excitations during this time, as a result of the Coulomb interaction, an ion with an additional charge can move to the interstitial space forming a vacancy or complex (vacancy and atom in the interstitial space).

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DISTRIBUTION PROFILES OF RADIATION DONOR DEFECTS IN ARSENIC-IMPLANTED HgCdTe FILMS

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Currently, the most promising design of *p–n* junctions used in photodiodes based on HgCdTe (MCT), the basic material for infrared photo–electronics, relies on fabrication of local *p*–type regions in an *n*–type base with the use of ion implantation of arsenic [1]. Such p^+–*n* junctions demonstrate smaller dark currents than their n^+–*p* counterparts, and this allows for increasing the operating temperature of photodiodes or for extending their photosensitivity cut-off wavelength. Ion implantation, however, leads to the formation of various types of radiation donor defects, so to form a required *p*–type region one needs to anneal the defects and to activate the introduced arsenic atoms electrically. To perform an effective annealing, it is necessary to know the exact nature of the donor defects and their location in the implanted material. For arsenic implantation in MCT such knowledge is not yet available, so the task of this work was to investigate distribution profiles of radiation donor defects in arsenic-implanted MCT.

The studies were performed on films grown with molecular-beam epitaxy on Si substrates with ZnTe/CdTe buffer layers. The films were doped with indium during the growth and initially had *n*–type conductivity with electron concentration at the temperature T=77 K, n=3.9·10^{15} cm^{-3}. They were brought to *p*–type with hole concentration p=5.1·10^{15} cm^{-3} via annealing at low mercury pressure (such annealing generates mercury vacancies, acceptors in MCT). After the annealing, the films were implanted with arsenic ions with 190 keV energy and 10^{15} cm^{-2} fluence. Electrical parameters of the films after the growth and annealing, as well as the distribution of electrically active radiation donor defects after the implantation were determined by studying magnetic field B dependencies of the Hall coefficient $R_H(B)$ and conductivity $\sigma(B)$ at T=77 K in B=0.01–1.5 T range with step-by-step chemical etching of the material. The obtained $R_H(B)$ and $\sigma(B)$ dependencies were analyzed with the use of discrete mobility spectrum analysis (DMSA) [2], which allowed for determining the types of carriers and their parameters (mobility, average concentration n_{av} and average partial conductivity σ_{av}, reduced to the whole thickness of the etched layer) after every etching step.

It was established that after ion implantation an n^+–*n–p* structure was formed, at its conductivity was contributed by three types of electrons with different mobilities. In particular, the n^+–layer was formed by extended and quasi-point donor defects (represented by electrons with low and intermediate mobility), while a thin (~ 1 μm-thick) *n*–layer (electrons with high mobility) was formed as a result of diffusion of interstitial mercury atoms and their annihilation with mercury vacancies. On the basis of the values of n_{av} and σ_{av} we calculated layered concentration N_s and layered partial conductivity Σ_s for each type of electrons at each etching step. The calculated values of N_s and Σ_s were used to plot the volume electron concentration and partial conductivity for each type of carriers against the thickness of the etched material.

The analysis of the obtained dependencies allowed for determining the distribution of radiation donor defect in arsenic-implanted HgCdTe films grown with MBE. In particular, low-mobility electrons (~5000 cm^2/(V·s)), which gave dominating contribution to the conductivity, appeared to be located in a layer with 400 nm depth; this layer coincides with the area of localization of extended structural defects and implanted arsenic ion profile [3]. These electrons are likely related to donor defects formed when interstitial mercury is captured by dislocation loops. Intermediate-mobility electrons (~20000 cm^2/(V·s)) are located down to the depth of 700 nm. They are related to defect complexes formed by interstitial mercury with various point defects. High-mobility electrons (~90000 cm^2/(V·s)) are located in *n*–layer extending beyond the depth of 700 nm. The obtained results clarify the details of defect structure of arsenic-implanted MCT and can be useful for the developers of MCT-based photo–electronic devices.

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COMBINED ELECTRON-ION-PLASMA TREATMENT OF HIGH-CHROMIUM STEEL SURFACE [1]

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The purpose of the present work is the analysis of structure, mechanical and tribological properties of the high-chromium 420 steel (USA analogue; 20Cr13 – Russian analogue) after the treatment combining deposition of a metal film with Zr-Ti-Cu composition (film thickness of 0.5 μm), irradiation of a film/substrate system by an intensive pulsed electron. Combined treatment was carried out in a single vacuum cycle on the «COMPLEX» installation [1]. The research of phase and element composition, a state of a defective substructure were carried out by methods of the scanning and transmission electron microscopy; the phase structure and a state of a crystal lattice were studied by methods of the X-ray phase analysis; mechanical properties of the irradiated surface were characterized by microhardness; tribological properties were characterized by wear resistance.

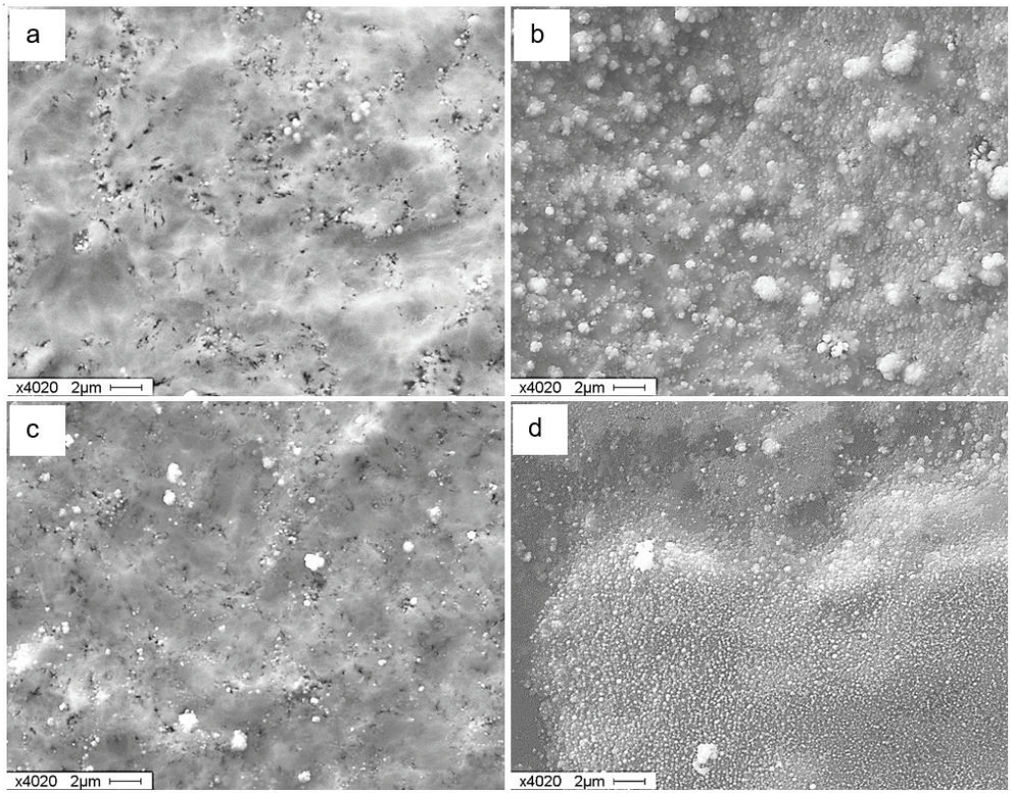

Fig. 1. Structure of 420 steel surface after nitriding in an initial state (a); after irradiation by an intensive pulsed electron beam (40 J/cm^2, 200 μs, 3 pulses) (b); after deposition of a Zr-Ti-Cu film (c); after deposition of Zr-Ti-Cu film and irradiation by an intensive pulsed electron beam (30 J/cm^2, 200 μs, 3 pulses) (d). Scanning electron microscopy.

By the methods of the x-ray microanalysis it is shown that irradiation of steel by an electron beam leads to increase of nitrogen concentration in the modified layer by 1.4 times. It is established that the maximum values of microhardness and wear resistance of steel are reached after the treatment combining irradiation of the film/substrate system by an intensive pulsed electron beam and the subsequent nitriding. These increase by 2.4 times and by many times, respectively, compared with the characteristics in an initial state.

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SURFACE ALLOYING OF HIGH-CHROMIUM STEEL: STRUCTURE AND PROPERTIES[1]

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The method of non-equilibrium surface alloys formation has been realized for the first time in the eighties last century at the use of nanosecond laser [1] and nanosecond (≈50 ns) low-energy high-current electron [2] beams. In the work [3] at surface alloying of 420 steel (USA analogue; 20Cr13 – Russian analogue) melting of the "Zr-Ti-Cu film / 420 steel substrate" system by submillisecond intensive pulsed electron beam the formation of multiphase submicro- and nanosized structure of cellular crystallization (Fig. 1, a, b) has been revealed. The increased concentration of zirconium in alloy [3] hasn't allowed to create hardening structure and, therefore, to achieve the high strength and tribological properties. In the present work which is continuation of [3], the created surface alloy was treated by nitriding (550 ºC, 4 hours) in plasma of the arc low-pressure discharge. The combined treatment was carried out in a single vacuum cycle on the COMPLEX installation [4]. The research of phase and elemental composition, a state of a defective substructure were carried out by methods of X-ray phase analysis, scanning and transmission electron microscopies; mechanical properties and tribological properties of the modified steel were characterized by microhardness and wear resistance, correspondingly.

It is established that nitriding of the surface alloy created at irradiation of the film/substrate system by an intensive pulsed electron beam leads to formation of multiphase structure (Fig. 1, c-e), to increase of microhardness and wear resistance of the surface alloyed layer by 2,1 times and by many times, respectively.

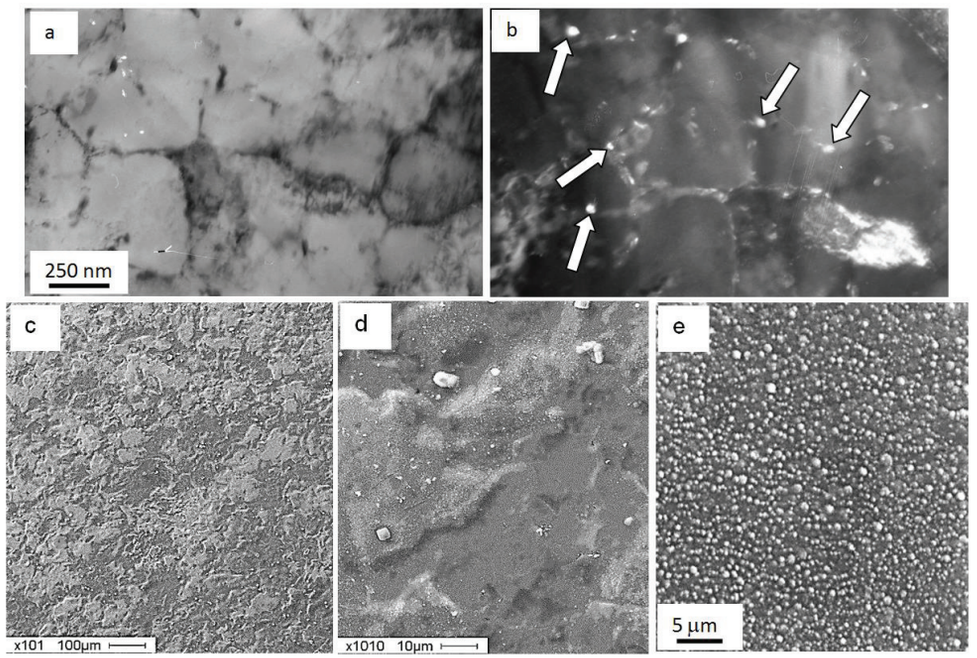

Fig. 1. The structure of the "Zr-Ti-Cu film / 420 steel substrate" system irradiated by an intensive pulsed electron beam (30 J/cm^2, 200 μs, 3 pulses) (a, b) and treated by the subsequent nitriding (c-e); a, b – transmission and c-e – scanning electron microscopy; the arrows indicate ZrC carbide particles in (b).

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THE INITIAL STAGE OF PARTICLE BEAM ACTION ON THE TAGET SURFACE[1]

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The requirements to surface properties of metals are higher every year. Vacuum ion-plasma methods are widely used for enhancing material properties and also for modifying the surface layer composition [1,2]. But some processes arising during processing cannot be research experimentally. That is why theoretical investigation have significant role for their detailed understanding.

The particles interaction with metal surface is accompanied by a variety of physical and chemical processes. First of all is the impact of particles on the surface leads to the appearance of mechanical stress. The experiment did not allow register these processes on the first moment. There are many works where the stress or strain propagation are investigated [3,4]. Often authors used the thermoelastic model for description of surface treatment process [5]. But the processes of impurity introduce and stress redistributions are interdependent in these papers. That is why the mathematical model must account this fact. In [6], the interaction of impurity diffusion and deformation was studied. However, the mathematical model in this work does not take into account the change of the substrate temperature during processing.

The paper is aimed at investigating the interaction of impurity diffusion and mechanical stress wave propagation under the action of a single pulse under non-isothermal conditions.

The mathematical model assumes that the occurring stresses are elastic; the velocities, accelerations and deformations are small. Then, mass balance equation; the heat equation and the equation of motion are used for description of the process of impurity injection into the metal surface under the action of particle beam. The fluxes of heat and mass taking into account the finiteness of relaxation times, in accordance with the thermodynamics of irreversible processes [7].

The problem is solved numerically with the explicit difference scheme using the sweep method.

The example of coupled problem solution is given in Fig. 1.

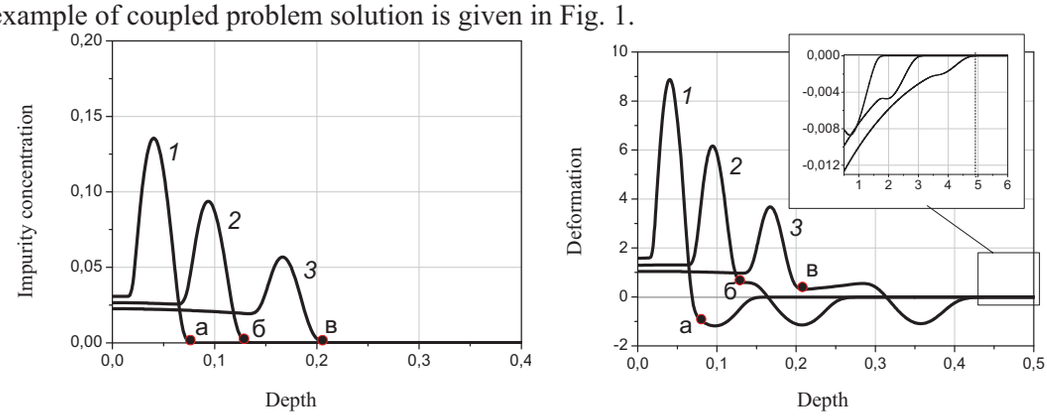

Fig. 1. The example of the coupled problem solutions: (a) the concentration of impurity distribution, (b) the strain distribution. Times moment, τ : (1) 0.014, (2) 0.025, (3) 0.04. Time of impulse action $\tau_{imp} = 0.1$

The interaction of waves leads to the distortion of the deformation (and stress) wave profile. The impurity concentration distribution does not correspond to pure diffusion process.

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DIFFUSION IN THE VOLUME AND ALONG GRAIN BOUNDARIES UNDER ACTION OF AN ELECTRON BEAM[1]

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At present, technologies for surface modification and structuring are progressing actively using high-energy external influences. Changes in the structure and properties of the surface as a result of the interaction of the charged particles fluxes with matter are studied. Of particular interest is the research of mass transfer under such conditions, taking into account the structure of the material. High-energy impact on the substance leads to heating and promotes the acceleration of mass transfer. Diffusion along the grain boundaries occurs faster than in the crystal volume. To describe grain boundary diffusion, the Fisher model and its modifications [1] or the molecular dynamics method are used. These approaches have limitations: the first method can not be applied to materials with a large number of internal surfaces, and the second method requires the presence of significant computing power. For a more detailed modeling of transport processes in systems with surfaces and boundaries, the Stefan approach [2] is used.

In this paper, to describe the mass transfer in the volume and along the boundaries of a material under the action of an electron beam, the following non-isothermal model is proposed, which is a modification of the model [3]. We assume that on the film-coated material acts an electron beam, which is distributed uniformly along the surface. The sample is processed in an inert medium or in a vacuum chamber. The conditions are such that the thermal losses from the lateral sides of the sample can be neglected. The sample size in the cross-section is finite. With pulsed irradiation, a homogeneous heating of the material and diffusion of the impurity from the film to the substrate occur. The substrate material consists of two phases: boundary and volume. Under these conditions, the mathematical formulation of the non-stationary problem includes the heat balance equation, the two-dimensional diffusion equation from the film to the substrate. At the initial time, the temperature of the sample is known, there is no diffusant in the substrate. We assume that the diffusant concentration in the film is constant and far from the diffusion zone, there is no mass outflow. At the internal boundaries between the bulk and boundary phases, the concentrations and their fluxes are equal. The diffusion coefficient in phases depends on the temperature according to the Arrhenius law. The problem is solved numerically using an implicit difference scheme.

Calculations show that with an increase in the diffusion coefficient in the boundary phase with respect to the coefficient in the volume phase over time, diffusion occurs predominantly along the boundaries. With increasing temperature, the ratio of diffusion coefficients in phases increases non-uniformly. If the diffusion coefficients in the volume and boundary phases differ by less than an order of magnitude, a uniform distribution of the concentration in the phases is observed. In the opposite case, with a rise in temperature, an uneven acceleration of diffusion along the boundaries occurs. The results of the calculations are in qualitative agreement with the experimental data.

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[1] This work was supported by Russian Foundation for Basic Research, grant number 16–01–00603

HIGH INTENSITY, LOW ION ENERGY IMPLANTATION OF NITROGEN IN AISI 420 ALLOY STEEL[1]

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This paper presents the results of the formation of deep modified layers in AISI 420 alloy steel using a high-intensity repetitively pulsed nitrogen ion beam with a current density up to 0.25 A/cm^2. An arc generator with a hot cathode provided the DC nitrogen plasma flow. A plasma immersion approach was used for high-frequency, short-pulse very intense nitrogen ion beam formation. A grid hemisphere with radii of 7.5 cm was immersed in the plasma. Negative bias pulses with an amplitude of 1.2 kV, a pulse duration of 4 μs, and a pulse repetition rate of 10^5 pulses per second were applied to the grid. The substrates were implanted at the temperature of 500 ˚C and various processing times ranging from 20 to 120 minutes with 1.2 keV nitrogen ions using a very-high current density up to 0.25 A/cm^2 ion beams. The work explores the surface morphology, elemental composition, and mechanical properties of deep-layer modified AISI 420 alloy steel after low ion energy, very-high-intensity nitrogen ion beam implantation.

[1] This work was supported by the state assignment of the Ministry of Education and Science of the Russian Federation, under grant 3.2415.2017/4.6

EFFECT OF MEGAPLASTIC DEFORMATION AND SUBSEQUENT ION IRRADIATION ON THE AL–CU–MG ALLOY STRUCTURE[1]

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Structural and phase transformations in 1441 alloy based on the Al–Li–Cu–Mg system was studied by transmission electron microscopy after megaplastic deformation (MPD), subsequent low-temperature annealing, and argon ion irradiation.

The samples of the 1441 alloy 2 mm thick were deformed at room temperature and a pressure of 4 GPa in Bridgman anvils to 5 revolutions (angle of the anvil rotation $\varphi = 10\pi$ rad). The thickness of the samples after deformation was 400 μm. Some samples after MPD were annealed at a low temperature of 160°C for 15 h, the rest samples were irradiated in a continuous mode using an ILM-1 ion implanter with a PULSAR-1M [8] source. The implanter emitted a homogeneous beam of accelerated Ar^+ ions ~100 cm^2 in the cross section. We used the following irradiation modes: (1) E = 20 keV, j= 300 μA/cm^2, $F = 1.9\cdot10^{15}$ cm^{-2}, ion beam heating to T_{max} = 160°C; and (2) E = 5 keV, j= 100 μA/cm^2; F= $2.5\cdot10^{16}$ cm^{-2}, ion beam heating to T_{max} = 140°C.

It was shown that MPD of the 1441 alloy caused mainly the formation of a mixed grain structure consisting of submicrocrystals with an average diameter of ~0.3–0.4 μm and nanocrystals with a diameter of less than 100 nm. There were also regions where fragments of deformation bands were observed against a background of the homogeneous submicrocrystalline structure.

It was established that 5- and 20-keV argon ion irradiation of the deformed 1441 alloy resulted in the formation of a completely recrystallized submicrocrystalline structure with equiaxed dislocation-free grains, rectified boundaries, and equilibrium triple junctions. In contrast to low-temperature annealing at T = 160°C for 15 h, no fragments of deformation bands in the alloy after irradiation were observed. The most uniform structure was obtained after argon ion irradiation at E = 5 keV, j = 100 μA/cm^2, $F = 2.5\cdot10^{16}$ cm^{-2} (T_{max} = 140°C): the average diameter of submicrograins was 0.5–0.7 μm. At higher energy of argon ions (20 keV) and the ion current density (300 μA/cm^2), and, respectively, at a higher irradiation-induced heating temperature (160°C) without holding at this temperature (irradiation time of 1 s, fluence $F = 1.9\cdot10^{15}$ cm^{-2}) the microstructure became more nonuniform: there are regions with submicrograins 0.8–1 μm in size along with recrystallized grains with an average diameter of 0.4–0.5 μm.

In addition, it was found that the irradiation of the deformed alloy 1441 with 5- and 20-keV argon ions, in contrast to the annealing, resulted in partial or complete dissolution of S_1 (Al_2LiMg) and T_2 (Al_3CuLi_5) phase particles, which are observed in the severely deformed state. The irradiation also suppressed natural aging with the formation of the metastable phase δ′(Al_3Li).

Thus, we established that powerful argon ion beams can be used for short-term (within a few seconds) radiation annealing to control recrystallization of the 1441 alloy samples ~ 400 μm thick after MPD. The study confirms the radiation-dynamic effect during ion irradiation of metastable media [1], namely, the aluminum–lithium 1441 alloy after MTD, when structural-phase transformations are initiated in the material at a depth of much higher than the projected ranges of the ions, and take place at a higher rate compared with traditional thermal annealing.

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THE EFFECT OF FLUENCE AND ANNEALINGS ON LIGHT-EMITTING PROPERTIES OF SILICON OXIDE FILMS IMPLANTED WITH ZINC AND OXYGEN IONS [1]

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Silicon dioxide with metal Zn inclusions has good prospects for being used in memory devices, lasers, solar cells and field emission display devices [1]. On the other hand, zinc oxide (ZnO) nanoparticles are of specific interest, since ZnO is a direct band material with a band gap of 3.37 eV and high electron-hole energy in the exciton (60 meV) [2]. One of promising method to fabricate Zn-based nanoparticles in SiO_2 matrix is ion beam synthesis.

In this study, thin layers of SiO_2 (600 nm) were implanted at room temperature with Zn^+ (140 keV) and O^+ (50 keV) ions with equal fluences (2.5×10^{16} and 5×10^{16} cm^{-2}). Afterwards, these samples were annealed at 700ºC for 30 min or at 750ºC for 120 min in the air ambient. Photoluminescence (PL) was investigated at low and room temperatures using the He-Cd laser beam at the wavelength λ = 325 nm as the excitation source. The elemental and structural composition of the as-implanted and annealed silicon oxide films were investigated by Rutherford backscattering spectrometry and transmission electron microscopy, respectively. The green and yellow-red bands with maximum at around 480 nm and 650 nm were observed in PL spectra of the as-implanted samples (Fig. 1, curves 1, 2). Besides, the intensity of emission decreases with fluence increasing. Thermal annealing results in the shift of green PL band to blue spectral range and in quenching of yellow-red band. The different effect of annealing on PL intensity is observed for the samples implanted with different fluences. In the case of high fluence (5×10^{16} cm^{-2}) PL intensity decreases while for the lower fluence (2.5×10^{16} cm^{-2}) increases in ~5 times. The origin of PL and effect of annealing treatment are discussed.

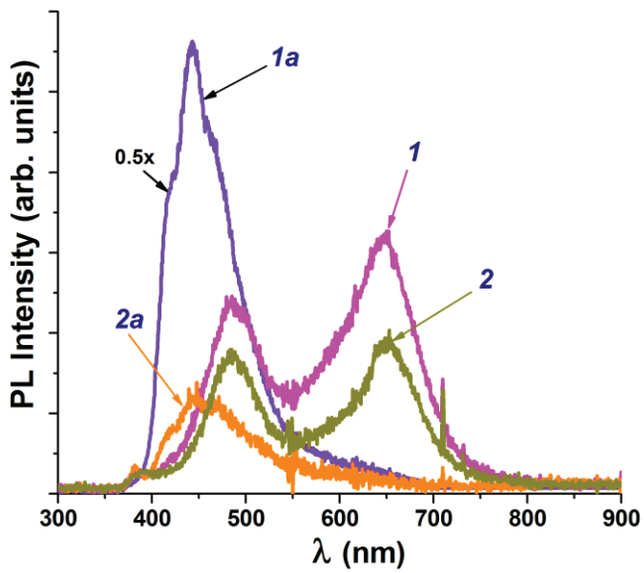

Fig. 1. PL spectra of SiO_2(600 nm) layers co-implanted (curves 1, 2) with Zn^+ (140 keV) and O^+ (50 keV) ions with fluences of 2.5×10^{16} (curves 1, 1a) and 5×10^{16} cm^{-2} (curves 2, 2a) at room temperature and afterwards annealing at 750ºC (curves 1a, 2a) for 120 min in air atmosphere

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THE STRUCTURE AND PROPERTIES OF MODIFIED SURFACE CARBON STEEL BY COMPRESSION PLASMA FLOW

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In connection with the exhaustion of the possibilities of further enhancing the structural material characteristics by traditional methods, such as chemical-thermal treatment, and the high cost of alloying additives, the surface modification methods of the functional material properties become topical.

The surface modification method includes the deposition of films, treatment with high-energy particle flows. The latter direction can be classified according to the sort of particles: ions, electrons, plasma. Accordingly, it is possible to single out methods of high-power ion beams [1], high-current electron beams [2], high-energy plasma flows [3].

In the high-energy plasma flows method, treatment of materials with high-enthalpy compression plasma flows stands out [4]. The advantages of this treatment are high speed and particle concentration (velocity up to $5 \cdot 10^7$ m/s and concentration up to 10^{18} cm^{-3}), and discharge duration of 100-1000 μs.

The report analyzes the effect of the compression plasma flow created by the end type erosive magnetoplasma compressor (MPC) on the structure and properties of a carbon steel sample. The device was operated in two modes: residual gas, vacuum. The effect of axial plasma flow and radial radiation flux was studied. Morphological changes in the sample surface, microhardness were studied. The energy and spectral brightness parameters of the plasma-dynamic discharge were also investigated, and the macrostructure of the plasma flux was fixed using an integrated photograph. The results have been discussing.

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THERMAL STRESSES COMPUTATION UNDER HIGH–CURRENT PULSED RADIATION OF AISI M2 TOOL STEEL

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Abstract. Surface modification of metallic materials and alloys with concentrated energy flows (powerful electron beams and ion plasma flow, laser beams) has been widely used in various fields of manufacturing with the aim of physical and mechanical properties improvement of the critical parts performance fabricated from modified materials. The mentioned energy impacts give rise to radiation, thermal and mechanical effects causing the changes in morphology, microstructure, elemental and phase composition of the surface layers, which in turn may lead to hardness, wear corrosion resistance and red hardness increasing of modified materials. However, as experimental investigations are shown, in some cases irradiation promotes the crystal defects' occurrence and often non-uniform heating leads to the cracks' formation in the surface layers. Thus, selection of incident electrons energy, its current density and pulse duration, taking into account the thermal stress state, is an actual problem of modern radiation technologies.

The paper provides a numerical solution of differential equations in partial derivatives for the temperature fields' computation as well as longitudinal and transverse thermal stresses distribution in R6M5 (AISI M2) high–speed steel exposed to medium–energy (up to 400 keV) high–current (up to 1 kA/cm^2) pulsed (up to 1 μs) electron beam radiation. The obtained results can be useful in the optimal modes selection of tool steels and products surface radiation treatment and evaluation of their service life.

Keywords: radiation processing; electron beam processing; electron energy; beam power; AISI M2 high–speed steel; thermal stresses; temperature distribution.

EXPERIMENTAL AND MODELLING STUDIES OF Sn-Fe LAYERED SYSTEM

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The methods of Mossbauer spectroscopy on ^{57}Fe nuclei were used to study thermally induced phase formation processes in two-layer Sn-Fe systems.

Tin layer was deposited on preliminarily prepared thin Armco iron foils by magnetron sputtering. The thicknesses of the layers of the coating and the substrate were chosen so that the average concentrations of Tin throughout the sample bulk were in the two-phase regions of the phase diagram, which, at given temperatures, consisted of a solution of Tin in α-Fe, and the corresponding intermetallides. The prepared samples were subjected to sequential isochronous annealing in a vacuum furnace in the temperature range 50-700°C. After each annealing at room temperature, Mössbauer measurements were made on ^{57}Fe nuclei in geometry for absorption. The fitting of the experimental Mossbauer spectra was carried out by the methods of model interpretation and reconstruction of the distribution functions of the hyperfine parameters realized in the MSTools software package [1].

As a result of the studies, a sequence of phase transformations is established whose nature is determined by the variation of the local concentration of tin in the sample during the diffusion of the components and corresponds to the features of the phase diagram of the equilibrium states of the binary Fe-Sn system.

With the help of the SPECTR program [1], the spectra of iron nuclei were modeled at various positions of the crystal lattice of phases present on the phase diagram of the Fe-Sn system. Further for the intermetallic phases FeSn and Fe5Sn3, in which the iron atoms occupy several positions, the PHASAN program [1] was applied and the final phase spectra were obtained. Using the state diagram of the binary system Fe-Sn and the "rule of the lever," spectra were reconstructed for Fe-15% at.Sn alloy.

We see a good agreement between the experimental and simulated spectra.

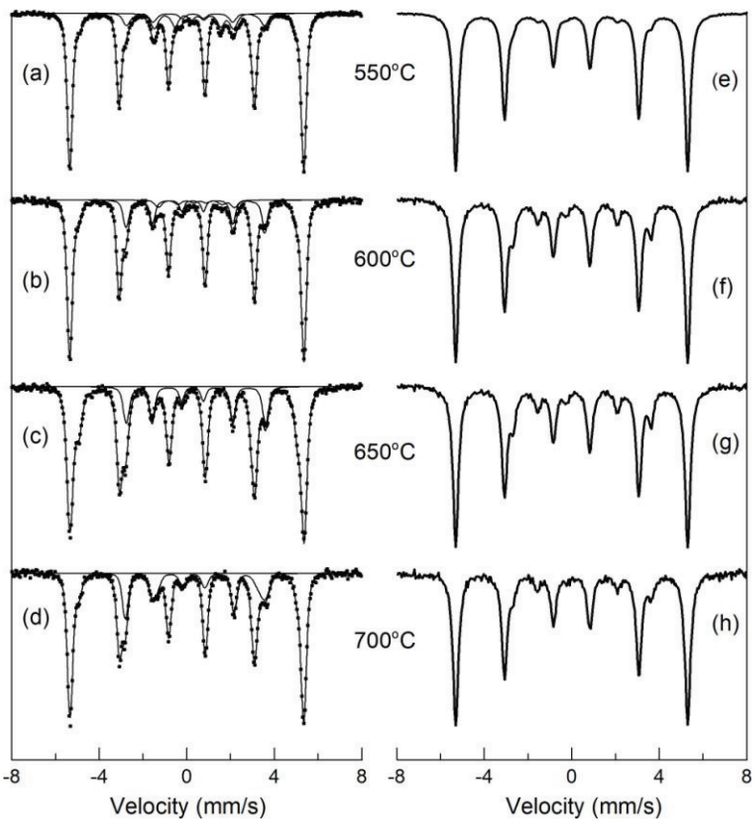

Figure 1. Experimental (a-d) and modeled (e-h) spectra of Sn-Fe layered system at various temperature

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PLASMA DEVICE FOR MATERIAL SURFACE TREATMENT BY HIGH-HEAT PLASMA[1]

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The plasma device at the National Research University "MPEI" (Fig. 1) has been constructed [1] to test materials in frame of the national fusion program developing the thermonuclear reactors (fusion neutron source and DEMO) and the international thermonuclear reactor ITER. The device is a linear plasma trap with a multicusp magnetic field confining a stationary plasma discharge of plasma electron temperature ~ 4-30 eV and plasma density up to 3×10^{18} м$^{-3}$ that provides a powerful plasma-thermal load on the test materials. High-temperature plasma irradiation leads to an inhomogeneous stochastic clustering of the surface with self-similar granularity - fractality on the scale from nanoscale to macroscales [2]. Cauliflower-like and fuzz structure of materials including tungsten and carbon was observed after the treatment under high heat plasma load in fusion devices [3]. The statistical characteristics of hierarchical granularity and scale invariance of structure are estimated to differ qualitatively from the roughness of the ordinary Brownian surface, which is possibly due to the universal mechanisms of stochastic clustering of material surface under the influence of high-temperature plasma [4].

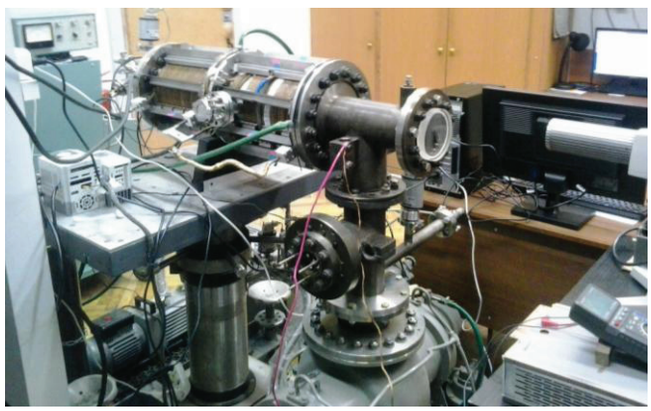

Fig. 1. Plasma device at the National Research University "MPEI"

A nanostructured surface formation on refractory metals (tungsten, molybdenum, titanium) and other materials will be studied. In the experiments, it is planned to develop a new technology for creating a highly corrugated and highly porous surface structure of refractory metals, including the so-called tungsten "fuzz" with pore size and nanofibers of ~ 50 nm, which is of considerable interest for nuclear, energy, biomedical technologies.

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STUDY OF PLASMA INFLUENCE AND STERILIZATION EFFECT ON THE WETTABILITY CHANGING OF POLYLACTIC ACID FILMS

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Polylactic acid is bioactive and biodegradable thermoplastic aliphatic polyester widely used in medicine. There are a lot of medical devices based on polylactic acid such as suture materials, bone plates, abdominal nets, stents, scaffolds for tissue regeneration, and drug delivery systems with controlled degradation [1]. Using the polylactic acid as an implant for ophthalmology particularly for bullous keratopathy treatment [2, 3] is the perspective direction for creating implants of new type. In view of the fact that the virgin surface of polylactic acid has a nonwettable surface we need to treat the material for creating desired properties. All medical implants should be sterilized before operation. The requirements to methods of polymeric material sterilization are very strict due to the need to preserve the shape of the graft and material properties. Sterilization by steam (moist heat) and γ-irradiation are widely spread in the medicine practice. Steam sterilization and effect of γ-irradiation can change physical and chemical properties of materials and can be cause of its destruction.

The aim of this research is the comparative study of plasma influence and sterilization effect on the wettability changing of polylactic acid films.

Materials and methods. The polylactic acid films of 25-30 μm thickness was made by dissolution of polylactic acid in $CHCl_3$. After films preparation the materials surface was treated by low-temperature plasma. The barrier discharge of plasma was carried out using cold plasma source. Low-temperature plasma had 25 kV voltages, 5 kHz frequency, 2 W/cm^2 of the power density. The temperature of treated surface did not exceed 40°C. Plasma treatment time was 30, 60 and 90 seconds. The sterilization was carried out in two ways. The first mode was steam sterilization (temperature was 120°C, pressure was 0.11 MPa). The second one was γ-irradiation of Co^{60} radionuclide with 1 kGy dose.

The surface wettability was studied by contact angle measurement using KRÜSS Easy Drop DSA 20 and deionized water, glycerol and n-hexane. The surface energy was carried out according to Owens-Wendt-Rebel-Kelby method.

The virgin films has 80° contact angle, which characterizes them as hydrophobic films. The full surface energy results of virgin materials showed a low polarity of polymer - 26.0±0.8 mJ/m^2. The plasma treatment contributed an increasing of contact angle by a factor of 1.5, which indicates an increasing of surface hydrophilicity. This effect was explained by the free radicals formation on the polymer's surface after low-temperature plasma treatment. The polarity of materials after plasma treatment was 0.67. Furthermore, the treatment contributed the increasing of surface energy from 26.0±0.8 mJ/m^2 to 44.5 ±1.5 mJ/m^2. The sterilization had not been instrumental in the changing of contact angle and full surface energy of polylactic acid films.

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PRODUCTION OF POROUS HYDROXYAPATITE COATING ON THE TITANIUM SUBSTRATE BY HIGH POWER ION BEAM IRRADIATION

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Porosity is one of the most important microstructural characteristics of natural bone, which significantly affects its properties. Therefore, materials for the replacement of bone defects must be porous and bioresorbable.

In the present work we studied the possibility of obtaining a porous coating of calcium phosphates modified with gelatin and silicate ions synthesized from a model solution of extracellular fluid of SBF type on a titanium substrate made of VT1-0 alloy.

The irradiation was performed on a Temp accelerator by the ion beam (70% C^+ and 30% H^+) with energy $E \approx 200$ keV, duration $\tau = 60$ ns, and a current density range of 20–150 A/cm^2. The residual pressure in the accelerator chamber was $5 \cdot 10^{-3}$ Pa.

As the calcium-phosphate compound, synthetic pure hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ was approved for application to surgical implants with a stoichiometric ratio of calcium and phosphorus of 1.67, which shows a high degree of biocompatibility. Powders of hydroxylapatite (HA) were synthesized in the presence of 3% gelatin (G) and Si-HGA from a solution of SBF with a silicon content of 0.50-5.0 wt. %. All samples synthesized in the extracellular fluid model solution medium are single-phase and represent hydroxylapatite. IR analysis of Si-HA powders showed that the nature of the reagent containing SiO_4^{-4} ions does not affect the structure of hydroxylapatite. The phase composition, the morphology of the obtained powder, and the surface of irradiated alloys were examined by optical, scanning electron, atomic force microscopy, and X-ray phase analysis. The porosity was evaluated using the fluid displacement method.

After irradiation by high power ion beam (HPIB) with a current density of 50 A/cm^2 with two pulses, the crystals of silicon-substituted hydroxylapatite in the presence of a 3% gelatin solution deposited on the titanium substrate are firmly fixed on its surface. Analysis of the morphology of the Si-HGA layer on the surface of irradiated samples showed that for all irradiation regimes used in the study, a coating of HA with a characteristic hexagonal structure of crystals with dimensions of ~ 0.3 μm was formed. Crystallization of HA occurs during the duration of the ion current pulse. When using a titanium implant with a fixed HPIB coating of Si-HGA, such crystals can play the role of nucleus when germinating into natural bone tissues. An investigation of the elemental composition showed that the formation of HA crystals occurs over the entire surface of the sample, but with different intensities, which indicates the heterogeneity of the coating. Irradiation with a high power ion beam with a current density of 50 A/cm^2 by two pulses of silicon-substituted hydroxylapatite in the presence of a 3% gelatin solution on a titanium substrate leads to the formation of a porous structure, the porosity is ~ 60-80%. The pore sizes of the coating vary from 0.17 to 7.24 μm, which practically corresponds to the porosity of the human spongy bone.

FORMATIONS OF WEAR-RESISTANT EXTENDED LAYERS BY COMBINED ELECTRON-ION-PLASMA TREATMENT ON THE SURFACE OF ALUMINUM [1]

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Combined electron-ion-plasma methods became increased interest due to high perspective possibility to change the surface properties of materials and products. In the present work the combined COMPLEX set-up is used for electron-ion-plasma treatment of materials for combination of following methods: nitriding, coating deposition and electron-beam treatment [1]. These methods can be carried out in different sequence and quantity of cycles.

The purposes of the work were obtainment of wear-resistant extended layers by combined electron-ion-plasma treatment on the surface of aluminum and complex investigation of their structure, composition and properties.

At the first stage of investigations the coating of TiCuN system [2] with different thickness in the range of 1-10 μm were deposited by vacuum arc plasma-assisted method on aluminum substrates. At the second stage coating characteristics as residual stress, crystallite size, hardness, Young's modulus, were investigated. At the third stage the coatings with high residual stress were treated by electron beam for these relaxation. At fourth stage the characteristics of treated coating were investigated. At fifth stage optimization of combined treatment (coating deposition and electron beam treatment) was obtained for revelation of treatment mode where coatings possess the best characteristics.

It should be noted that treatment cycle (coating deposition and electron beam treatment) were carried out with different quantity for formation wear-resistant extended layers (~ 10 μm).

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FIELD ION MICROSCOPY OF RADIATION DAMAGES IN MATERIALS AFTER EXPOSURE BY FAST NEUTRONS OR Ar^+ BEAMS

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Investigating interaction mechanisms of accelerated particles with matter and studying the atomic rearrangement and, therefore, formation of crystal lattice defects and changing the phase state of the material are important tasks in radiation physics of solids. Radiation clusters formed under irradiation during the evolution of cascades of atomic displacements are the regions of strong elastic distortions, which affect the motion of dislocations under straining, induce radiation hardening, reduce plasticity, and change the characteristics of elasticity.

Information on the characteristics of radiation clusters such as concentration, size, internal structure, and the number of point defects contained in them is essential for quantitative analysis of the effect of irradiation on the structure and physico-mechanical properties of alloys. This study is devoted to experimental investigation of fundamental physical processes in solids, which are initiating by the interaction of flows of charged gas ions (Ar^+) and neutron beams with the substance.

The main goal was analysis of radiation defects on an atomically pure surface and in the bulk of materials. Materials were induced by neutron bombardment of Pt (99.99) with $E > 0.1$ MeV and ion implantation in Pt and Cu_3Au alloy in the ordered state (E = 30-40 keV) by the methods of field ion microscopy (FIM).

One of the tasks of this work was to establish the adequacy of the effects of different forms of radiation affecting on the same material (Pt) when analyzing radiation damage of the same type. For this purpose, using the methods of FIM, we have studied radiation defects on an atomically clean surface and within a subsurface volume of platinum that are created because of neutron and ion beam bombardment ($E > 0.1$ MeV and E = 30 keV, respectively). FIM allows precise studying to be carried out of the changes in the real crystal lattice structure of metals and alloys occurring as a result of irradiation on the atomic scale. At the same time, this method allows one to analyze the structure in the bulk of the sample by means of consecutive removal of surface atoms by the electric field.

Experimental results on atomic–spatial investigation of radiative defect formation in surface layers of materials, initiated by neutron bombardment (of Pt, E > 0.1 MeV) and ion implantation (in Cu_3Au: E = 40 keV, F = 10^{20} ion/m^2, j = 10^{-3} A/cm^2), are considered. Quantitative estimates obtained for the size, shape, and volume fraction of cascades of atomic displacements formed under various types of irradiation in the surface layers of the materials. It is showing that the average size of radiation clusters after irradiation of platinum to a fast neutron fluence of 6.7×10^{22} m^{-2} (E > 0.1 MeV) is about 3.2 nm. The experimentally established average size of a radiation cluster (disordered zone) in the alloy after ion bombardment is $4 \times 4 \times 1.5$ nm.

STRUCTURAL PHASE TRANSFORMATIONS OF THE SURFACE LAYER OF Ti-SiC SYSTEM UNDER ELECTRON BEAM TREATMENT[1]

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The results of the investigation of evolution structural phase states and microhardness of the surface layer Ti-SiC system are considered (Ti film 0.5 μm thick was deposited on the surface SiC ceramic) under electron beam treatment. Samples of SiC ceramics obtained by SPS-sintering were used. Irradiation with a high-intensity pulsed electron beam of submillisecond duration was carried out at the SOLO device [1, 2] under the condition: energy density of the electron beam of 15 J/cm^2, pulse duration of 200 μs, quantity of pulses – 20 and 30. The XRD was used to analyze the phase composition. The composition of elements and the defective substructure of the surface layer were investigated by scanning electron microscope (SEM). The XRD of the treated Ti-SiC system showed that under the selected irradiation regime the phase composition formed in the surface layer and the volume fraction of the phases depend on the quantity of irradiation pulses. Thus, at 20 pulses, a phase composition with a volume fraction was formed: SiC – 18.5 %, TiC – 36.6 %, Ti_5Si_3 – 44.9 %; at 30 pulses, the following phase relationship: SiC – 81.6 %, TiC – 12.7 %, Si – 0.5 %, C – 5.2 %.

The surface morphology of the Ti-SiC system treated with an intense pulsed electron beam was studied by SEM. It was established that formed the surface layer, irrespective of the quantity of pulses of the electron beam, has a "smoothed" globular structure [3] formed as a result of melting of the titanium film, which contains a droplet fraction (Fig. 1). The particles of the drop fraction have a submicron-nanocrystalline structure (Fig. 1, a) and are enriched of titanium atoms (Fig. 1, b).

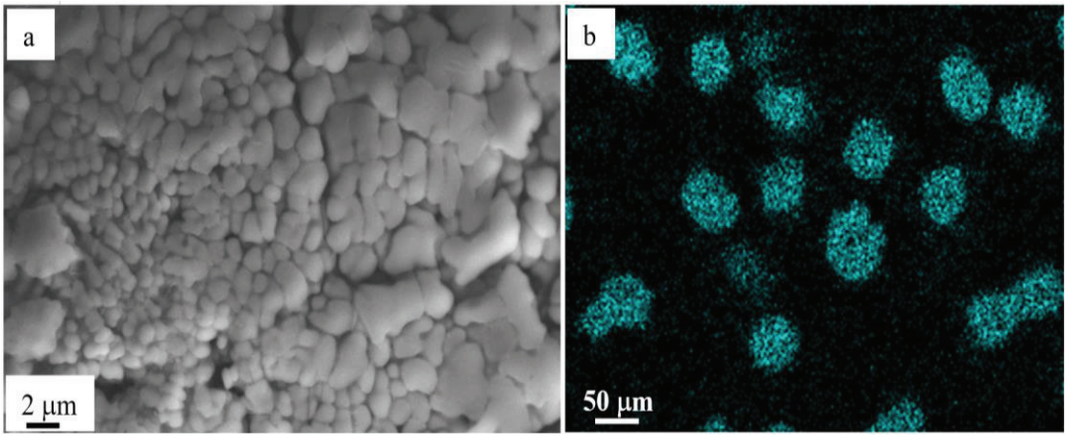

Fig. 1. The structure of the "film (Ti)/(SiC) substrate" system irradiated by an intense pulsed electron beam at 30 pulses, (a); the image of the surface layer of "film (Ti)/(SiC) substrate" system was obtained in X-ray Ti (Kα), (b).

Physico-mechanical studies of the modified layers were carried out by measuring their microhardness on the PMT-3M device. It was established that electron beam treatment with 30 pulses leads to formation of round-shaped regions with maximum microhardness values from 55 GPa to 96 GPa on the surface of irradiation, exceeding the microhardness of the initial SiC ceramic (~ 36 GPa). It was suggested that these regions were formed as a result of electron beam treatment of the surface layer, which contains a drop fraction.

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HYPEREUTECTIC SILUMIN MODIFICATION BY ION-ELECTRON-PLASMA METHOD[1]

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Hypereutectic silumin is the perspective material for industrial application. Silumin is mainly used in the production of plain bearings and pistons. It has relatively (relative to the eutectic silumin) high hardness due to the silicon content (more than 12 wt.%). The hypereutectic silumin in the cast state contains inclusions of primary silicon, whose size reaches 100 microns, the eutectic grains and intermetallics [1]. Into the melt of the hypereutectic silumin modifiers are added to disperse the structure that significantly increase the gas saturation, which is accompanied by the appearance of pores and shells. These disadvantages do not allow the use of hypereutectic silumin in the cast state.

The aim of the work is to reveal the regularities of the evolution of the structural-phase state and the properties of hypereutectic silumin (18-24 wt.% Si) under ion-electron-plasma treatment.

The samples were in the form of a cylinder 5 mm high, 30 mm in diameter. Ion-electron-plasma combined treatment resulted in formation of a thin (0.5 μm) film of Zr-5% Ti-5% Cu composition on the samples surface and subsequent irradiation of the «Ti-Zr-Cu / (Al- (18 -24) wt.% Si) substrate» by an intense pulsed electron beam. This treatment was carried out on the equipment of the ISE SB RAS [2]. The irradiation mode by the electron beam was selected according to the thermal calculations [2, 3].

It is established that the combined treatment is accompanied by the formation of the cellular crystallization structure, dissolution of silicon primary inclusions and intermetallides in the layer up to 50 μm, grain size reduction to 2-4 μm, formation of silicon, zirconium, titanium inclusions and CuZr compound of the submicron-range (Fig. 1). The samples hardness increased in 4.5 times at optimal mode of combined treatment by ion-electron-plasma method, wear resistance increased in 2 times.

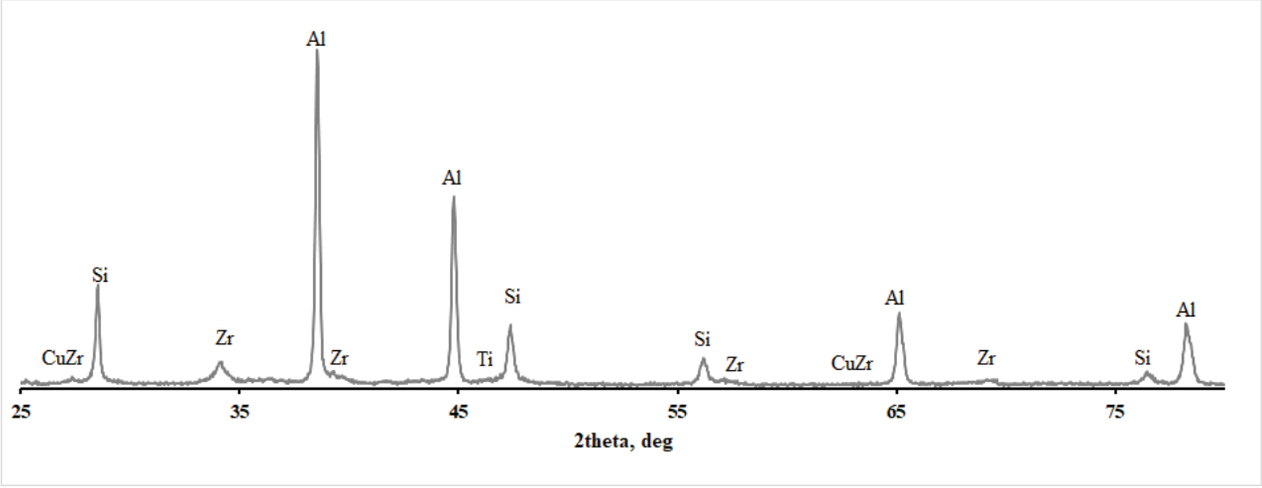

Fig. 1. The X-ray diffraction of the hypereutectic silumin (22-24 wt.% Si) subjected to ion-electron-plasma treatment

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THE FORMATION OF OHMIC CONTACTS OF MOLYBDENUM/SILICON AT ION IMPLANTATION INTO THE INTERFACE REGION

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Ohmic contacts in very larger-scale-integrated circuits and microwave transistors are important because of a more stringent requirement of low resistance and contact reliability due to reduced device sizes. In the study of thermally formed Mo/Si contacts, it has been demonstrated that the presence of the native oxide at the Mo/Siinterface increases the formation temperature and affects reproducibility of the silicide formation [1]. Moreover, the existence of native oxide drastically degrades the quality of metal-to-silicon contact such as ohmic contact or Schottky contact [2].

The usingof refractory metals does not completely solve the problem of reducing the electrical resistivity of contacts and in order to obtain contact resistivity (ρ_c) values of the order$1\cdot10^{-5}$ - $1\cdot10^{-6}$ Ω·cm^2, required to the production of device working in centimetric range and $\rho_c 1\cdot10^{-6}$ - $1\cdot10^{-7}$ Ω·cm^2 for device working in millimetric range, a new approach to the creation of ohmic contacts is required.

One of such new processes intensively studied now is the modification of materials by an ion beam, based on the ability of high-energy ions to mix a metal layer with the surface of a semiconductor as a result of cascades of dynamic displacement caused by the passage of primary ion.

The results of studies the effect of boron, phosphorus ions irradiation, at a dose from $6.25\cdot10^{13}$ to $3.125\cdot10^{15}$ cm^{-2}, argon – $6.25\cdot10^{14}$ cm^{-2} and an annealing in vacuum on the value of contact resistivity of Mo/Si contacts and on the structure of the transition layer are presented. Based on the studies by four-probe method is shown that an annealing at the temperature of 400-500 °C, irradiated by ions of boron of contacts Mo/p^+Si and ions of phosphorus of contacts Mo/n^+Si leads to a decrease in the value of electrical resistance of the contact regions in comparison with non-irradiated in 5-10 times and more than 2 orders of magnitude, respectively, and by the argon ions irradiation leads to irreversible increase in the electrical resistance of the contact regions. Measuring I-V characteristics of the contacts showed ohmic behavior in the irradiated samples, expect for samples with a dose of doping of silicon with boron$6.25\cdot10^{13}$ cm^{-2} (contacts Mo/p^+Si).

Using electron diffraction analysis the phase composition of the transition region Mo/n^+Si contacts, interface, which was subjected to bombardment with ions of phosphorus and an annealing, have been studied. Hexagonal Si_3Mo_5 phase, superstructure Mo_3Si and $MoP_{0.75}$were identified in samples by the phosphorus ions irradiationat a dose $6.25\cdot10^{14}$cm^{-2} and an annealing at 300 °C.

The structureof ion-doped silicon layers was investigated by TEM. It is established, that the nature of structural changes and phase transformations in the transition region of the contacts depends on the surface condition of silicon. Data on the change of resistivity are in a good agreement with the results of electron diffraction studies.

It has been established that in the Mo/n^+Si system a decrease in the ratio of the mean projective path length of phosphorus ions to the Mo film thickness from 1 to 0.7 during implantation leads to a reduction in the number of defects in the near-surface silicon layer under the Mo film, as a result of which there is no abrupt increase in the electrical resistivity and surface resistance silicon. The range of doses of silicon doping by phosphorus ions$6.25\cdot10^{14}$–$4.375\cdot10^{15}$cm^{-2}is established, in which this effect is observed, which is explained by the change in the concentration of active impurity and the absence of a disordered layer in silicon under the Mo film.

In this work, a method for changing the silicon bipolar microwave power transistor's characteristics in a direct and deliberate manner by modifying the chemical composition at the Mo/Si boundary, the electrophysical properties of Mo/Si contacts, and the electrophysical characteristics of transistor structure areas by the phosphorus ions irradiation of generated ohmic Mo/Si contacts to the transistor emitters is proposed for the first time. The possibilities of this method are investigated and confirmed experimentally. At the same time, both the power and frequency characteristics of the transistor are improved, the radiation resistance increases.

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STRUCTURING OF STEEL SURFACE BY POWERFULL PLASMA PINCH

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The Method of treatment based on ultrafast plasma pinch energy impact in the surface layers of the material. The same effects of pulsed plasma influence on the material surface are: heating to a high temperature, melting and fast cooling, and hardening of subsurface layer [1-2]. The samples of industrial steels AISI 201 and 321 were treated by pinch flow with fluxes 10-50 J/cm^2 once and several (10-20-30) times. The velocity of flow about 4-9 cm/μs, and duration of each pulse 12-14 μs. We used an air plasma flow in all experiments usually. On initial surface of steel samples under a magnification x 1000 (SEM), grain pattern etched with a pronounced relief is clearly visible. The grains have different size of 10 to 40 μm.

Using atomic force microscopy (AFM) can consider in detail the surface topography of two types of image – plane and 3D. Thus, three-dimensional images of different areas were obtained. Figures 1 presents the AFM image of the surface of exposed to a twofold and tenfold effects of plasma flow. As shown by the AFM analysis of the initial surface, etched grain structure is clearly seen, obtained by electrolytic etching on sample preparation step. Block structure becomes more pronounced after exposure to plasma, which confirms the possibility of plasma etching at the nanoscale for the given parameters of processing even without etching the original surface. At the same time, in some cases, plasma etching is accompanied by a «delamination» of surface structure and clearly enhances the outline of the track lines. Moreover, the two-time surface melting treatment results in a local formation of blisters and there are areas with columnar crystallites formed in the direction perpendicular to the surface. Ten –time treatment leads to enhancement of the effect of double processing, and columnar crystallites are moved mainly to the grain boundaries (blocks) as shown in Figure 1.

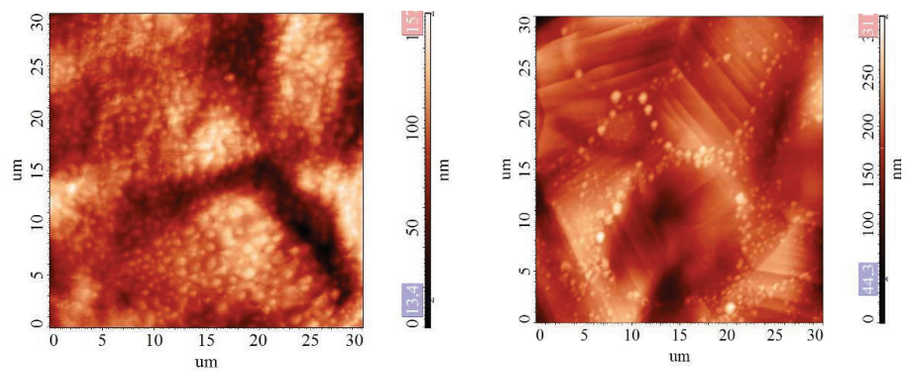

Fig. 1. AFM images of the of 2-time (left) and 10 –time (right) treated AISI 201 steel sample surface

AFM analysis of the results of experiments on processing steel 321 showed that, unlike steel 201, it has the height of the columnar crystals in the double treatment much more than typical for steel 201, but columnar crystallites are mainly located along the grain boundaries as in the case of 201 steel.

We can conclude that treatment with pulsed plasma flows leads to structural and phase changes and defects in subsurface region of the material. The result of SEM analysis revealed that the surface melts after plasma treatment. As a result of rapid cooling after plasma irradiation, nanocrystalline blocks are formed on subsurface area, oriented vertically to the surface flat. This study revealed that these blocks are arranged at the crystal's grain boundaries, and their size depends on the number of treatments. AFM analysis revealed that the surface of the material in the double treatment shows traces of blistering, the presence of the layered structure and tracks of the formation of columnar structures, which may be due to the planar and linear defects. When the same processing is tenfold, more ordered structure, columnar blocks are arranged relatively uniformly over the surface and the tracks are located mainly at grain boundaries.

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FIRST PRINCIPLES INVESTIGATION ON CATALYTIC PROPERTIES OF N-DOPED Co_3O_4 BULK[1]

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Today, we observe worldwide significant progress in the development of the transition to "green" energy. Such development in the countries is connected not only with the strongest negative impact on the environment from emissions, but also with the advanced strategic development of the economies of these countries in this area. Possession of such technologies makes the country's economy more attractive to investments in the energy sector.

Response of decomposition of water takes place with energy absorption as a result of which the free energy of Gibbs increases by 237 kJ of $mole^{-1}$. This additional energy necessary for photocatalytic and photoelectrochemical decomposition of water is provided by means of energy of sunlight. For this purpose in photoelectrochemical cells as the cathode noble metals on which there is a restoration of the hydrogen formed in the course of water expansion, and as the anode on which there is a decomposition of water under the influence of sunlight are used, semiconductors in which under the influence of sunlight are excited an electron - hole couples are used. These excited charge carriers in the course of a relaxation are transferred to surface-active parts where participate in water expansion response.

In the first part we plan to find the most energetically favourable positions of N atoms in the host material. Several calculations with different concentration of the dopant have to be carried out in order to check stability of doped material. Lattice constant expected to change with the concentration. It is important for the further surface and defect calculations to have a perfectly optimized bulk structure. For the concentration, with the most stable energetics, we create a surface. Surface model needs to be optimized. We have to find the reasonable number of crystallographic planes, and the size of vacuum gap between the terminating planes. Numerous attempts have been made to increase catalytic activity by introducing various impurities. In our recent papers we have considered a detailed theoretical description of mers for fluoride doped with Co_3O_4 [1]. The obtained data were in good agreement with the experimentally obtained results. For this purpose, one of the promising impurities is nitrogen. Xu and others in their experimental studies found good evidence for increasing the catalytic activity of N-doped nano-sheets. The increase in surface area in combination with oxygen vacancies has led to an increase in electrocatalytic activity for RVC. Calculations from the first principles of N-doped Co_3O_4 volume showed an increase in catalytic activity for water molecule splitting.

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PHASE TRANSFORMATIONS IN NANOSTRUCTURED COATINGS BASED ON Zr-Y-O PRODUCED BY A METHOD MAGNETRON SPUTTERING

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The reversible martensitic transformations in metal alloys (the so-called transformational conversion) attract special attention due to their potential practical application in many fields of science, technology, medicine and industry. These alloys belong to the group of the so-called "smart" functional materials, as they allow controlling their behavior [1,2]. These are transformation-hardening materials which are widely used in engineering practice as structural materials. The majority of the above ceramic materials have been developed on the basis of zirconium dioxide that is partially stabilized in the tetragonal phase. The tetragonal phase is capable of a monoclinic martensitic phase transition. The phase transition is accompanied by the shear and volume strain, stress relaxation and closure of surface cracks. The hardening effect can result in producing ceramic materials with the strength properties (fracture toughness and strength) comparable to those of structure materials.

The paper deals with investigation of the change in the grain structure, the grain and interphase boundaries, the structure and phase composition during the heating mode «in-situ» in the microscope column followed by the thermoelastic phase transition in the multi-layer coating based on Si-Al-N / Zr-Y-O in the $Zr_{1-h}Y_xO_2$ layer.

Deposition of the coatings was carried out in the KVANT-03MI unit [6] equipped with a magnetron with a mosaic zirconium-yttrium target. The magnetron was powered from a pulse source at the frequency of 50 kHz. The samples were placed in the chamber on the rotating table. The sample temperature during the deposition was 573K. The temperature was measured using a chromel - alumel thermocouple. Multilayer coating on the basis of the Si-Al-N / Zr-Y-O system with the layers 1000 nm in thickness were produced.

The fine structure of the nanostructured coatings was investigated by transmission electron microscopy (TEM) using the JEM-2100 microscope (Jeol Ltd., Japan, center for collective use of Siberian Federal University) with a built-in high temperature chamber. The investigation temperature was from 400 up to 900^0C. The foils were prepared by the «cross-section» method using the ION SLISER-EM-09100IS installation (Jeol Ltd., Japan). The acceleration voltage was at acceleration voltage of 2 kV, polishing angle was 4.5^o.

By TEM it has been established that coatings on the basis of Zr-Y-O produced by the magnetron sputtering methods have a nanograin column structure where the columns are spread through the entire coating thickness.

There are the tetragonal phase ZrO_2 with a small amount of monoclinic one in the layers on the basis of Zr-Y-O in the initial state.

Heating of the of the layer on the basis of Zr-Y-O in a column of TEM in the “in-situ” mode leads to: 1) turns of grains of the main phase together with change in the angle of disorientation crystallographic planes, 2) martensitic transition of the tetragonal phase to the monoclinic 3) modification of grain boundaries– their total length increases, the form of grains changes, in initial column grains there are cross boundaries, i.e. there is a process of the grain fragmentation.

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IRRADIATION EFFECT ON THE ELECTRICAL PROPERTIES OF $AgGe_{1.6}As_{0.4}(S+CNT)_3$ GLASSY COMPOSITE MATERIAL[1]

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The aim of this work is to study the effect of a charged particles beam (deuterons and protons) on the electrical properties of $AgGe_{1.6}As_{0.4}(S+CNT)_3$ amorphous composite material containing ~7 at.% single-walled carbon nanotubes and having a share of ionic conductivity at least 99% at room temperature [1, 2].

Irradiation of the samples was carried out on a 2 MB van de Graaf accelerator. Samples numbered 0 and 1 were irradiated by deuterons with an energy of the beam of 900 keV during different time intervals, the projective range of deuterons in $AgGe_{1.6}As_{0.4}(S+CNT)_3$ was 8.67 μm. Samples 2 and 3 were irradiated by protons with a beam energy of 762 keV, the projective range of the protons was 7.75 μm. The samples had parallelepipedic form with thickness of 500 μm.

The effect of deuterons irradiation during the 2228 seconds (sample 0) led to significant changes in the surface morphology of sample, as well as to a change in the atomic composition of the material from the irradiated side, not least because of the thermal heating of the material during irradiation. In the case of a deuteron beam with the same energy for 715 seconds (sample 1), as well as when exposed proton beam, the surface morphology practically does not change. Changes in the chemical composition of materials according to EDS data are also not observed.

To study the changes in the electrical properties of materials a direct current (DC) was applied to the irradiated samples in such way that the irradiated side was at a positively charged electrode. For all samples, the increase of electrical resistance with time under DC was observed (Fig. 1.).

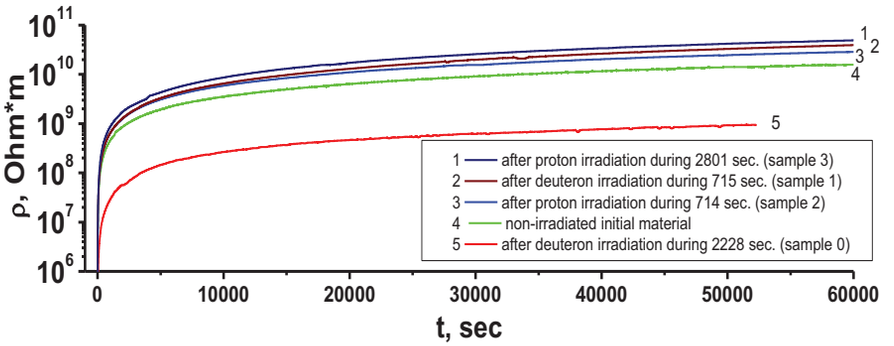

Fig. 1. Electrical resistivity versus time under DC of glassy $AgGe_{1.6}As_{0.4}(S+CNT)_3$ after a different irradiation doses by the deuteron or proton beam.

In the case of sample 0, significant changes in the electrical properties were observed in comparison with the non-irradiated sample. A higher conductivity of the sample is observed during the entire time of DC influence. The area under the resistance curve from the time of exposure to DC is substantially less than that of the non-irradiated sample. That can be the consequence of a strong damage of sample 0 due to thermal heating and changes in its composition and atomic structure. However, the share of ionic conductivity component in the total conductivity remained practically unchanged and consists of 98%. In the case of samples 1; 2; 3, the resistance under DC increases more rapidly comparison with pure material and increase of area under the resistance curve ρ(t) (t - exposure time of DC) is observed. In the case of samples 2 and 3, the area under the ρ(t) curve becomes larger with increase of dose of proton irradiation. This behavior may be due to the contribution of proton conductivity to the ionic conductivity of the material. An increase in the number of positively charged carriers leads to an increase of resistance with higher rate under DC.

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MODIFICATION OF TITANIUM MICROSTRUCTURE UNDER ION IRRADIATION FROM INDUCTIVELY COUPLED PLASMA[1]

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Titanium and its alloys are extensively used in biomedical applications, particularly in orthopedic and dental implantology, due to their beneficial mechanical properties, excellent biocompatibility and corrosion resistance [1]. However, the integration processes of an implant could be facilitated and considerably accelerated provided its surface has uneven porous topology with characteristic feature size of 0.1–10 μm [2–4]. Besides, such engineered surfaces could be utilized as substrates for biofilm growth and protein production.

Among the many options for surface modification of materials, the most effective, flexible and environmentally-friendly are the ion-plasma techniques that enable controlling the surface properties through changing the characteristics of ion fluxes incident at the material. In this contribution, a low-pressure inductively coupled plasma (ICP) source [5] was utilized to roughen the surface of VT1-0 grade titanium under argon ion irradiation.

The correlation between plasma parameters and surface microstructure of VT1-0 grade titanium has been investigated. First, the plasma parameters for relevant treatment regimes have been studied with a double probe. The experimentally measured *I-V* curves have yielded the electron temperature and density values in the ICP discharge at $p_{Ar} = 5\times10^{-3}$ mbar for different radiofrequency power levels (P_{rf} = 800–1600 W) and for different values of external magnetic field. The maximum obtained electron density was $n \sim 10^{12}$ cm^{-3}, and the electron temperature was T_e = 4–7 eV depending on P_{rf}.

The VT1-0 samples for surface engineering experiments were rectangle strips 30 mm × 70 mm × 0.4 mm thick. Each of samples was polished, ultrasonically pre-cleaned in alcohol, and then irradiated in argon ICP plasma for 30 min under pulsed bias voltage of –900 V. The surfaces of samples before and after the processing were examined in a scanning electron microscope (SEM) Tescan Vega 3 (Fig. 1).

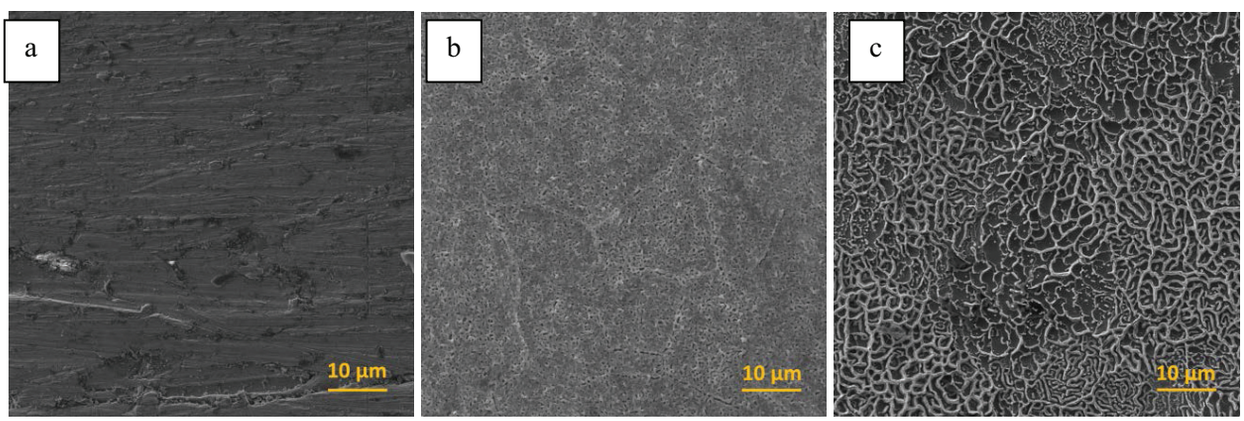

Fig. 1. SEM images of VT1-0 samples: (a) untreated; (b) P_{rf} = 1200 W; (c) P_{rf} = 1600 W

The resulting surface topology exhibited characteristic sizes of pores in the 0.1–5 μm range depending on parameters of irradiation regime. Such surfaces could be relevant for applications in implantology and for biofilm preparation.

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EFFECT OF IRRADIATION WITH A HIGH-INTENSITY PULSED ELECTRON BEAM ON MECHANICAL PROPERTIES AND STRUCTURAL STATES OF COATINGS FORMED BY PLASMA SPRAYING[1]

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Combined technologies based on the use of concentrated energy fluxes, in many cases, allow a multiple increase in physicomechanical and tribological properties of materials [1, 2]. The purpose of the present studies is to develop a method for modifying steel surfaces, which combines plasma spraying of a powder coating and the subsequent irradiation with an intense pulsed electron beam. Plasma spraying of a steel powder of the system Ni-Cr-B-Si (PGSR-4 with a fraction of 80-100 μm) onto the surface was carried out with the use of an original apparatus equipped with two plasma generators [3]. Irradiation of the modified layer of steel was carried out with an intense pulsed electron beam on the installation SOLO [4]. An investigation of the phase and elemental composition, as well as the state of the defective substructure, was carried out using X-ray diffraction analysis methods, scanning and transmission electron diffraction microscopy; mechanical properties of the irradiated surface were characterized by microhardness; tribological properties by wear resistance.

It is shown that plasma spraying of the powder coating leads to formation of a high-relief surface containing micro- and macropores (Fig. 1, a, b). The subsequent electron-beam treatment of the modified surface in the mode of the surface layer melting with an electron beam is accompanied by smoothing of the coating surface (Fig. 1,c), saturation of the crystalline lattice of the surface layer of steel with Ni, Cr, B, and Si atoms, formation of dendritic crystallization cells of submicron sizes (Fig. 1, d), release of nanosized particles of the second phase, and formation of the quenching structure. Together, this has allowed a multiple increase in the hardness and wear resistance of the surface layer of steel.

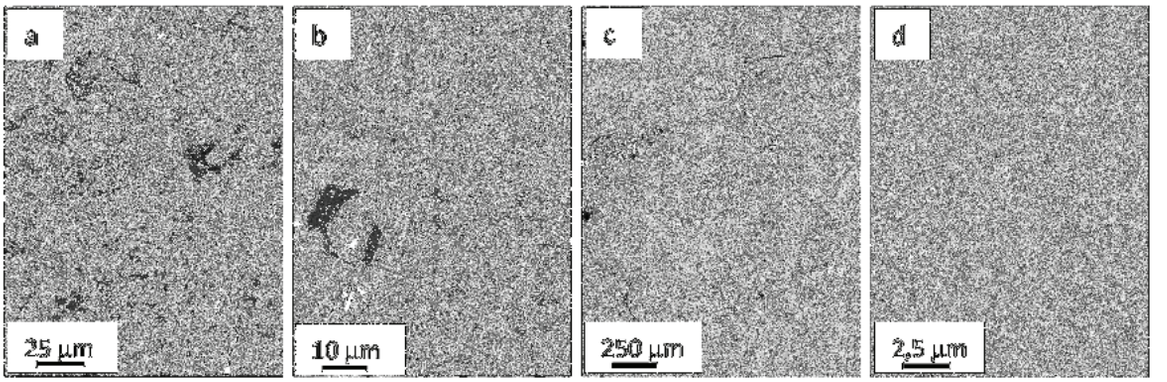

Fig. 1. Surface structure of steel samples subjected to plasma spraying of a powder coating (a, b) and the subsequent irradiation with an intense pulsed electron beam at a pulse duration of 200 μs, an electron beam energy density of 40 J/cm^2, and a pulse number of 10. Scanning electron microscopy.

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TRANSLATIONAL NONIVARIANCE OF THE MODIFIED LAYER FEV - N TREATED WITH A HIGH POWER PULSE ION BEAM[1]

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The distribution of elements in the surface layer of nitrided ferrovanadium after treated of high power pulse ion beam (accelerator TEMP-4M) was studied . Irradiation of (FeV – N)- samples (samples are produced of the SHS-method) was carried out H^+ and C^+ : the energy of single - charged ions-250 Kev, pulse duration-100 ns, dose -10 pulses at a flux density of 10^{14} ion/ cm^2. Surface relief and element composition of(FeV-N)-materials after ion irradiation were studied by means of SEM. In the time of ion irradiation in (FeV-N)-materials are created an internal heat source due to the effect of Bragg. After the termination of ion pulse radiation asymptotically unstable system-solid / liquid- are situated. At ultrafast solidification of the melt, a dissipative structure with a volume content of the elemental composition fundamentally different from the original one arises are formed. Cracking the cooling of the melt takes place mainly on the old craters formed in the early stage of cooling fluid. New craters are smaller in size and appear from the porridge-like state of the melt. Concentration profiles of elements of the selected spaces in the images of SEM do not have translational invariance for the analyzed volumes. After exposure to HPIB on the surface (FeV-N) - sample new nonequilibrium phases, coarse carbide Fe_{23} C_6 and local x-ray amorphous carbon clusters (two – three of cubic micrometer) are formed.

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THE STRUCTURE OF CRATERS ON THE SURFACE OF STAINLESS STEEL AFTER THE HIGH-POWER PULSED ION BEAM [1]

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The most characteristic feature of the surface relief of samples treated with a powerful pulsed ion flux is the presence of defects in the form of craters. Craters arise as a result of complex physical processes, but the mechanism and causes of crater formation are not clear.

Therefore, in this paper, the topography of the surface of 12Kh18N10T steel (321 AISI) after exposure to a high-power pulsed ion beam was investigated by scanning electron microscopy. The samples made from stick strain of 12Kh18H10T before irradiation were polished to high luster. The flows ions of pulse beam ions (H+ and C+ ; accelerator TEMP-4, energy ion 250 keV , J_i = 100 – 300 A/cm^2). A thin foil of the crater cross section was prepared using a focused ion beam in a column of a two – beam electron-ion microscope. The microstructure and elemental composition of the cross-section were studied by transmission electron microscopy Tecnai G2 F20. On the fig.1 shown microstructure of crater.

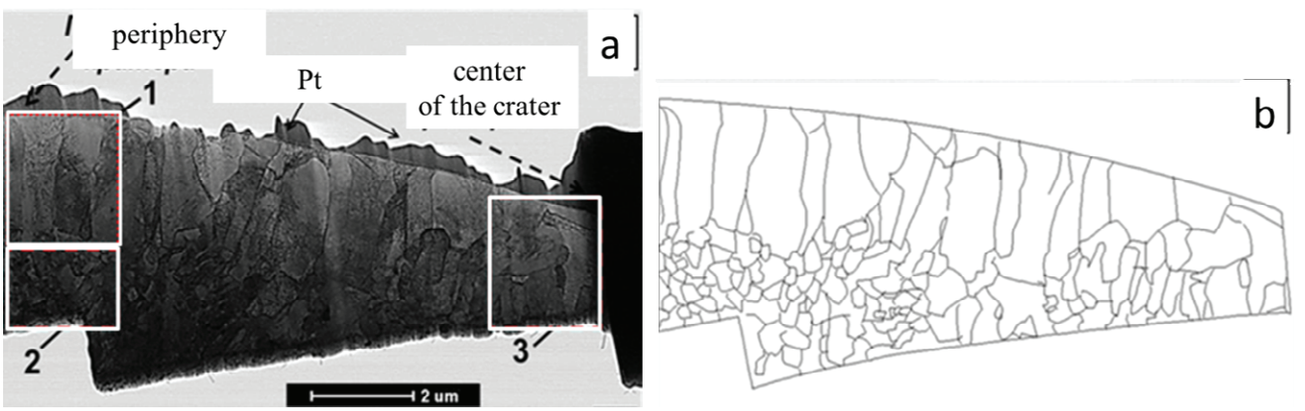

Fig.1. Microstructure of crater cross section (a) and its schematic drawing (b).

The surface layer of the crater consists of elongated towards the surface of columnar grains with an average size (length) of ~ 2 microns (coefficient of grain nonequilibrium>3) (area 1). Under the layer of elongated crystallites in the crater is an area with a submicrocrystalline (SMC) grains, the average size of which is 250 nm (area 2).

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[1] This work was supported by the program of Russian Academy of Science «Fundamentals of high-current pulsed emission electronics»

COMPLETE IMPREGNATION OF CONIFEROUS WOODS UNDER THE INFLUENCE OF HIGH-FREQUENCY CURRENTS

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Wood is a universal building material with many useful properties. Wood has good thermal insulation, high mechanical strength, aesthetics, and is also easy to install and handle. However, the use of wood in construction is limited by its susceptibility to destruction by bio-organisms, fire, weather conditions and mechanical deterioration.

In construction, coniferous wood is one of the most widely used types of wood.

Treatment of the wood surface with protective liquid compositions does not provide sufficient protection of the material. To increase the required qualities of wood, complete impregnation is used. All methods of impregnation are divided into four groups – capillary impregnation, diffusion impregnation, impregnation under pressure, a centrifugal impregnation method [1]. Complete impregnation of coniferous species in accordance with the technology developed for hardwoods does not provide impregnation of the material throughout the entire volume of the billet due to differences in the internal structure of the fibers.

In this work, we carried out the studies of the effect of high-frequency currents in complex with technologies of complete impregnation developed for hardwoods on the depth of impregnation of coniferous woods.

Samples of wood were cut into two halves. One half was used in the experiment without a high-frequency generator, the other with a connected high-frequency current generator.

Samples were placed in a sealed chamber. Inside the chamber a pin electrode was located. In all cases, before impregnating the samples, a vacuum of 10^4 Pa was maintained in the chamber for 30 minutes. The chamber is then filled with an impregnating composition and the pressure increased to 10^6 Pa. At this pressure, the samples were kept in solution for 2 hours. In this work, an aqueous solution of the dye was used as the impregnating composition for a visual determination of the impregnation depth. For the experiment with a high-frequency generator, simultaneously with pressure impregnation, HF currents (90 kHz) were fed into the chamber through the electrode, and all other parameters remained unchanged.

The oscillograms of the acting currents are shown in Fig. 1

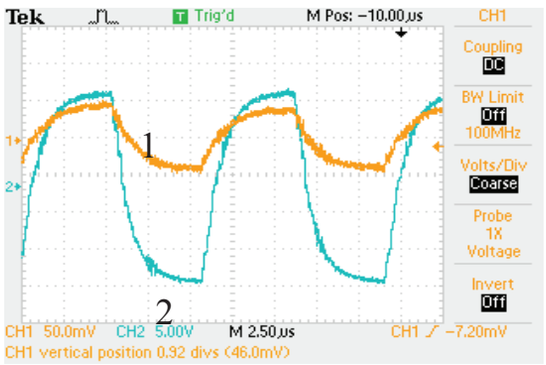

Fig. 1. Oscillograms of 1-voltage and 2-current

Our experiments showed that the usage of high-frequency currents allows impregnating coniferous wood (pine) to a greater depth, relative to impregnation without the influence of high-frequency currents.

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THE PHASE COMPOSITION OF TOOL STEELS AFTER NITRIDING BY GLOW DISCHARGE IN THE MAGNETIC FIELD.

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A detailed study of the phase composition of the surface and the zone of internal nitriding was carried out using X-ray diffraction analysis. The experiments were carried out on samples of steels P6M5 and X12, as a working gas a mixture of nitrogen, argon and acetylene (N2 70%, Ar 25%, C2H2 5%) was used. Preliminary ion cleaning was performed at P = 10 Pa during cathode sputtering, the surface temperature did not exceed T = 250 ° C. The cleaning time was 15 minutes. The samples were subjected to ion nitriding in a glow discharge with a magnetic field, at P = 40-80 Pa, the surface temperature of the samples treated was T ≈ 500 ° C. The treatment was carried out for t = 4 hours.

Analysis of diffraction patterns from X12 steel (Fig. 1a, 1b) shows that nitriding at P = 44 Pa, and U = 580 V, results in notable changes of phase composition in the surface region.

On the surface of the samples after ion nitriding by glow discharge reflexes detected nitrides and carbonitrides of alloying elements (CrN, Cr (C, N)), and carbides ((Cr, Fe) 7S3, Fe3C). It is known [1] that the iron nitrides have higher heat capacity as compared with iron, thus creating favorable conditions for preventing the temperature at the tool surface flares. The high hardness of tool surface without changing when heated to 500 ° C, determined by the presence of nitrides and carbonitrides of chromium (CrN, Cr (C, N)), and the wear resistance and low tendency to pilling - carbides (Cr, Fe) 7S3, Fe3C [2,3].

The diffractogram taken from the surface of steel X12 after ion nitriding is distinguished by the blurring of interference maxima, due to the formation of nitride and carbonitride phases in the near-surface layer

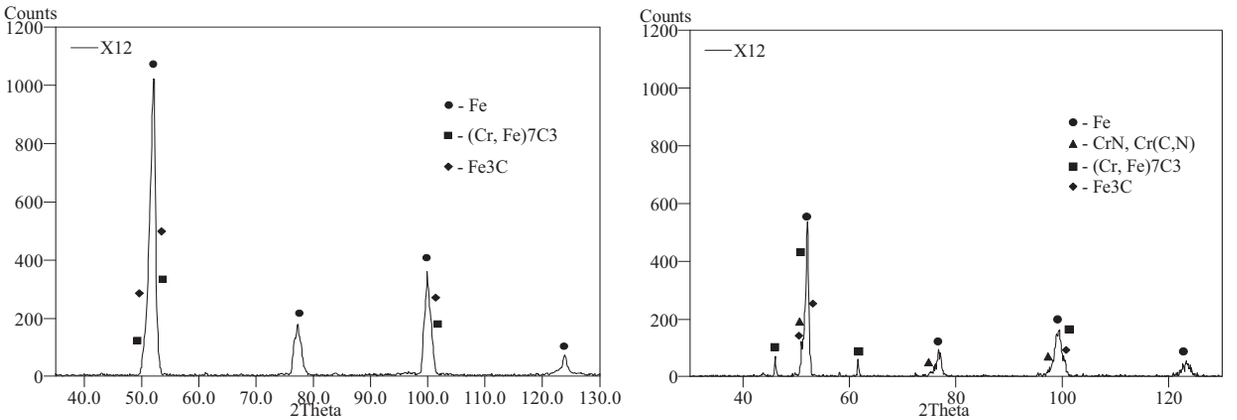

Fig. 1. Diffractograms of X12 steel samples: (a) - before nitriding after hardening and tempering,

(b) - after ion nitriding in a glow discharge at P = 44 Pa, t = 4 hours, U = 580 V.

A layered X-ray diffraction analysis gives a typical picture of the phase composition change in depth. In the near-surface zone, there is a nitrided layer consisting of an α-phase - a nitrogenous ferrite with distributed fine-dispersed nitride and carbonitride particles (CrN, Cr (C, N)). The diffraction maximum of α-Fe shifts towards large reflection angles and approaches the value corresponding to the initial state at a depth of 50 μm, and at a depth of 100 μm actually corresponds to the initial state.

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EFFECT OF NITRIDING IN A GLOW DISCHARGE WITH A MAGNETIC FIELD ON THE MICROHARDNESS OF 08X18H10T STEEL.

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The influence of the magnetic field on the microhardness and thickness of the diffusion layer of 08X18H10T steel is studied. The effect of pretreatment on the diffusion of nitrogen during ion nitriding in a glow discharge is studied. The microhardness distribution curves for depth are obtained at various temperatures. It has been established that the use of a magnetic field during nitriding leads to an increase in the microhardness of 1.5 ... 2 times and 1.5 times the thickness of the diffusion layer. Application of finishing treatment diamond smoothing before ion nitriding in a glow discharge increases the diffusion of nitrogen deep into the processed material, due to the formation of a deformed structure.

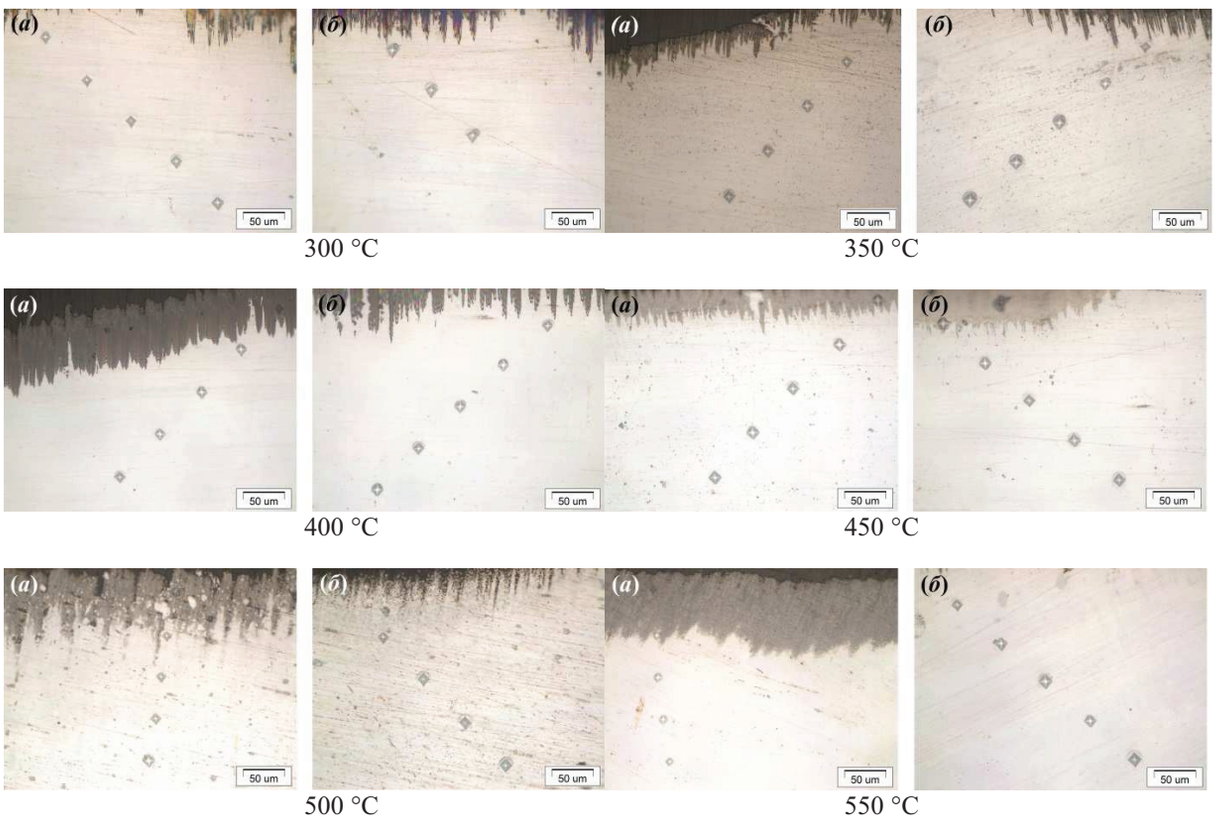

Fig. 1. Optical photographs of microhardness measurements of samples with diamond thinning (a) nitriding with a magnetic field (b) nitriding without a magnetic field (t=6 h, p=50 Pa).

Analysis of the results of microhardness measurements of samples with diamond smoothing showed that a 1.5-fold increase in the thickness of the diffusion layer occurs in the magnetic field. This is due to the increase in the density of the ion current, due to the retention of the magnetic field of electrons in the processing zone, which in turn increase the number of ionization events.

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IBUPROFEN CONTROLLED RELEASE FROM E-BEAM TREATED POLYCAPROLACTONE ELECTROSPUN SCAFFOLDS[1]

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Biodegradable polymers are considered to be a highly suitable candidate as materials for the fabrication of targeted drug delivery devices [1]. The fine-tuning of the amount of a drug in the human body delivered to a specific site is possible by varying the size, thickness and shape of the device. Moreover, physicochemical properties of polymeric materials can be also altered with the employment of various surface modification techniques in order to achieve the desired dosage and release rate of a drug [2].One of the most important aspects in scaffolds utilization is the control of their degradation rate which must correlate with a speed of tissue regeneration. Moreover, control of the degradation rate is also critical for drug release profile. In order to control the rate of degradation and drug release from polymer scaffolds number of physico-chemical surface modification methods have been proposed [3]. One of the advanced methods, which allow obtaining required properties of the polymer, is the pulsed electron beam treatment.

The study was focused on the investigation of ibuprofen-loaded electrospun fibrous scaffolds. The sustained release of the model drug over a period of one day was demonstrated. Electron beam irradiation treatment in a pulsed mode was employed to increase polymer surface energy, reduce polymer molecular weight and subsequently modify the release kinetics of ibuprofen.

Electron beam irradiation of PCL scaffolds was conducted using pulsed e-beam accelerator TEA-500 with range of absorbed dose of 25 kGy. Poly (ε-caprolactone) (PCL) (MW 80-90 kDa) was purchased from Purac (Netherlands). Hexafluoroisopropanol (HFIP) and ibuprofen were obtained from Ekos-1 (Russia) and Pharmstandard (Russia), respectively. All the materials were used without further purification. PCL scaffolds were produced by electrospinning a 7% (wt./wt.). PCL solution in HFIP using a NANON-01A machine (MECC Co., Japan). For the drug release study, ibuprofen was added to PCL solution prior to the electrospinning process, and the ratio of the drug to PCL was 1:10 (10 wt.%) and 1:20 (5 wt.%) (wt./wt.). Scaffolds surface morphology was investigated employing scanning electron microscopy (Quanta 400 FEG) with pre-coating of the samples with a thin layer of gold. Untreated and e-beam treated electrospun PCL scaffolds, both with the incorporated drug, were immersed in Phosphate Buffer Saline (PBS, pH 7.4) at 25 ºC with three replicates for each scaffold. The amount of released ibuprofen was determined using UV-vis spectroscopy.

The results of scanning electron microscopy showed no significant changes in surface morphology of scaffolds and fibers radii distribution after the e-beam irradiation. The treatment allowed for an increased drug amount to be delivered in a shorter time period compared to the released drug amount from the untreated samples. Release tests results demonstrated the significant rose in release rate of the loaded drug after treatment. In case of 5 wt.% PCL scaffolds the cumulative amount of released ibuprofen after an hour of observation was only 0.5 wt.%. After scaffolds surface modification this value increased more than fifteen-fold up to 7.8 wt.%. The change of released drug amount in case of 10 wt.% ibuprofen samples in the PCL scaffolds was less than in 5 wt.% samples but still significant (from 0.9 wt.% before and 2.3 wt.% after the irradiation).

Thus, it has been demonstrated that the modification of ibuprofen-loaded PCL scaffolds by pulsed electron beam treatment leads to an increase in drug release rate and can be useful and effective method for controlling drug release.

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CHANGES IN THE STRUCTURAL PROPERTIES OF NB AS A RESULT OF IRRADIATION BY A PULSED ION BEAM[1]

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One of the most pressing problems in developing structural materials for nuclear power applications is to control and limit irradiation induced defects in refractory materials. Materials interacting with ionizing radiation may experience thermal peaks that lead to the changes in structural properties, increase defects formation and reduces mechanical strength properties. Thus, there is a need in materials with excellent radiation resistance, good thermal conductivity, low sputtering speed, good adaptability with plasma for use. Here we study irradiation effects on structural materials by investigating the effect of a pulsed ion beam irradiation on structural properties and corrosion resistance of niobium films. The 80 ns beam consisted of carbon ions (C^{n+}) and protons with a ratio (C^{n+}/H^{+} = 85/15). Samples were irradiated by 3 and 10 pulses of the beam with energy density 1 J/cm^2 per pulse. We found irradiation induced change in morphology due to recrystallization processes caused by short high intensity ion beams. Increasing number of pulses leads to the surface formation of crater-like structures with small carbon impurities due to recrystallization. We demonstrated that samples irradiated by pulsed ion beam with controlled specific parameters are less susceptible to corrosion processes, as a result of formation of a modified surface layer preventing rapid heat induced oxidation of samples surface.

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PULSED ION BEAM MODIFICATION OF SILVER NANOWIRES[1]

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Transparent conducting films are essential for modern optoelectronic devices such as flexible solar cells, flexible organic light-emitting diodes, touch screens, transparent heaters etc. [1, 2]. Currently, indium-doped tin oxide (ITO) has been widely used as a conductive optically transparent material. However, indium limited supply, high-cost and the need for high vacuum deposition and high-temperature annealing cause very high cost of ITO coating production. Alternative materials such as carbon nanotubes [3, 4], graphene [5, 6], metal nanofilms, metallic nanowires [7, 8], etc. are intensively investigated to produce transparent conductive coatings with good mechanical flexibility and plasticity. Silver nanowires (AgNW) based transparent coatings are promising because of their high electrical conductivity, high transparency and high ductility of the conductive layer [1, 9, 10]. Since AgNW coating consists of disordered network of nanowires, there is a significant contact resistance effect of nanowire-nanowire junctions on the coating conductivity. To increase the conductivity of the coating additional processing is required. The traditional method of AgNW based coatings post-treatment is annealing at the temperatures of about 200-300 ^{o}C [11, 12]. However, high temperature processing is not permissible for flexible substrates that are mostly made of polymers.

We applied an intense pulsed ion beam of nanosecond duration to modify AgNW based coatings. Main parameters of a pulsed ion beam used in the experiments are following: accelerating voltage is 200 kV, total beam current is 10 kA, the beam pulse duration is 80 ns (at half-height). The beam mainly consisted of carbon ions (C^+, C^{++}) and protons in approx. ratio C^+/H^+ = 85/15. A beam current density was varied in the range from 0.5 to 10 A/cm^2 while the accelerating voltage and composition of the ion beam remained unchanged. Each sample was irradiated by only one pulse of ion beam. We found that pulsed ion beam of nanosecond duration is an efficient low-temperature tool for AgNW welding. Moreover, changing the beam current density various structures and modifications of AgNW may be obtained in controlled way.

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INFLUENCE OF AL ADDITIONS ON MICROSTRUCTURE OF MG-6 WT.%Y ALLOY AND SYNTHESIS OF MG-10 WT.%AL_2Y MASTER ALLOY

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In this work, the influence of Al additions on microstructure of as-cast Mg-6Y alloy has been investigated, meanwhile Mg-10Al2Y master alloy was synthesized. The results show addition of Al into Mg-6Y cast alloy dramatically refine the grain size, and the finest grains of 57 μm were obtained at 1 wt.% Al addition level. The active nucleation Al2Y particles were reproducibly observed at the centres of fine grains. But continue to add Al, the grain size is obviously coarsening. Two types of Al2Y phase were discovered in the Mg-6Y-*x*Al (*x*=1~4) alloys. One is the granular pre-precipitated phase and the other is irregular eutectic phase, however act as a heterogeneous nucleation sites is the pre-precipitated Al2Y. The YA65 alloy can be regarded as a grain refiner of Mg with pre-precipitated Al2Y particles and application prospect has high expected.

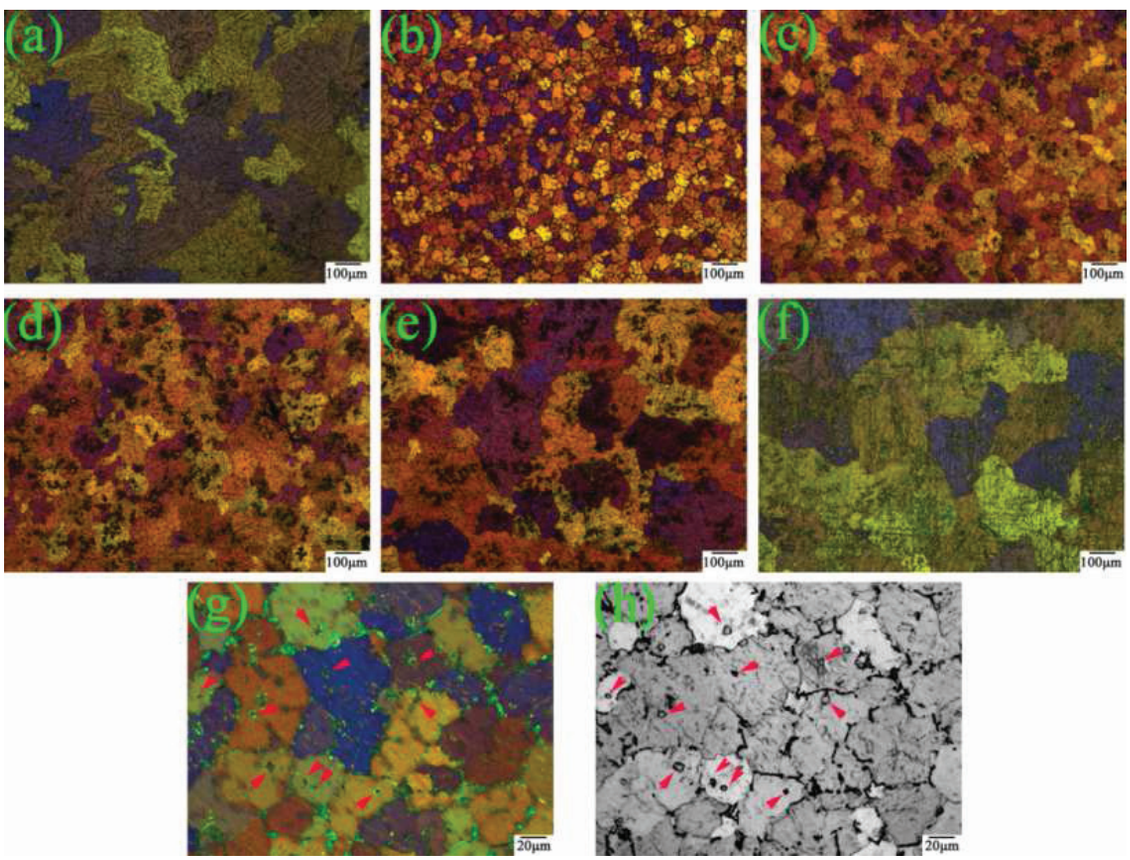

Fig. 1 Optical micrographs of as-cast Mg-6Y alloys with the different addition Al contents:

(a)0.5%; (b)(g)(h)1%; (c)2%; (d)3%; (e)4%; (f)5%

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THE PHYSICAL AND MECHANICAL PROPERTIES OF COATINGS BASED ON INTERMETALLICS OF TI-AL SYSTEM SYNTHESIZED IN A OXYGEN ENVIRONMENT BY VACUUM ARC PLASMA

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The last time in the aircraft engine industry is along the way of increasing the characteristics of the engine by the toughening requirements imposed on the structural materials. For this, new materials with improved physical and mechanical properties are created [1]. Because of this, the requirements for metal-cutting tools are increases. One of the most promising ways to solve the problem is the creation of new wear-resistant composite coatings for metal cutting tools [1]. It is also promising to create multi-layer composite coatings by alternating layers with different physical and mechanical properties. Research in this area shows that multilayer composite coatings have higher performance properties than single-layer coatings.At present, the coatings based on intermetellides of the Ti-Al system attract much attention [2,3]. This coating has an increased physical and mechanical properties compared to other coatings, since it has a higher resistance to oxidation at higher temperatures and retains its properties up to the melting point. However, there is little in the scientific literature devoted to the study of the physical and mechanical properties of coatings based on intermetallides of the Ti-Al system synthesized in the envoirenment of oxygen by vacuum arc plasma.

The purpose of this article is to study the physical and mechanical properties of coatings based on intermetallides of the Ti-Al system synthesized in the environmebt of oxygen by vacuum arc plasma.

To study the physicomechanical properties, H10 F hard alloy samples were deposited by coatings based on intermetallides of the Ti-Al system synthesized in the environment of oxygen. Microhardness, wear resistance, coefficient of friction, adhesion strength, scratch test of coating were studied on the obtained samples. Influence of the deposition conditions on the content of intermetallic compounds in the coating was established by X-ray diffraction methods. The results of investigations showed that the main physico-mechanical properties of coatings based on intermetallides of Ti-Al system synthesized in the environment of oxygen were increase compared to the coatings synthesized in the environment of argon or nitrogen.

The results of the research broaden the knowledge of the physical and mechanical properties of coatings based on intermetallides of the Ti-Al system synthesized in the environment of oxygen. Thus, the coatings obtained allow us to expand the field of application of coatings based on intermetallic compounds of the Ti-Al system. To confirm the results of the studies, production tests were carried out of conical end mills with obtained coating (Fig. 1).

Fig 1. conical end mills with obtained coatings based on intermetallides of the Ti-Al system synthesized in the oxygen environment by vacuum arc plasma.

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INVESTIGATION OF THE DEFORMATION STRUCTURAL FEATURES UNDER INDENTATION OF NANOSTRUCTURED TI-AL-SI-CU-N COATINGS

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The combination of strength and plasticity of the coatings, which necessary for operating conditions, can be obtained on the basis of the results of studying the features and mechanisms of structure modification under various conditions of specific mechanical loading, in particular, indentation. In this connection, it should be noted that, in view of the low plasticity of the nitride coatings, the main mechanism of their deformation when indenting on soft substrates is the formation of various kinds of cracks - radial, inclined, edge, and shear slip along the boundaries of columnar crystals [1]. The data on the modification of the defective microstructure in the indentation region are few and contradictory [2, 3], but allow one to assume the possibility of initiating the plastic deformation of the coating by changing the level of the stresses acting under indentation with a change in the strength of the substrate or by forming a heterophase nanocrystalline structure with substantially different strength properties of the constituent components. In this connection, the task this work was to obtain more complete data on the nature of deformation and fracture, the modification of the microstructure of single-layer columnar and nanostructured gradient, gradient-layered coatings of doped titanium nitride in indentation zones. The electron-microscopic dark field analysis of the bend-torsion of a crystal lattice was used as an investigation method, which makes it possible to determine the features of the defective microstructure of nanostructured coatings.

Coatings of the Ti-Al-Si-Cu-N system were obtained by reactive magnetron sputtering of Ti targets (VT1-0 alloy), $Al_{60}Si_{40}$ alloy and technical grade copper in argon and nitrogen medium under conditions of assisting two sources of gas (nitric) plasma on hard-alloy substrates T15K6 at their planetary rotation. The deposition of the coatings was carried out at a pulsed potential bias of -200 ÷ -300 V and a substrate temperature of ~ 150 °C, the nitriding of the titanium sublayer was performed with a negative bias 6 kV on the substrate. The sputtering power of the targets of Al-Si and Cu alloy was varied with a corresponding increase in the rate of nitrogen leakage during the growth process for the synthesis of gradient and gradient-layered coatings. The thicknesses of the coatings obtained are in the range 0.8-2.2 μm.

Investigations of the deformation behavior of coatings with various structures (with grains in the size range from submicro to nanometer), including gradient-layered, in the indentation zones were established that regardless of the structural state under the indentor tip, the plastic behavior of the coating material is observed without its cracking. For the coatings and their individual layers with a columnar structure and submicron grain sizes, their residual shape change is associated with the slope, shape change, and also the increase in density, disorientation values, and nonequilibrium state of the interfaces. It is shown that the deformation of a nanocrystalline gradient coating with a high copper content is expressed in the formation of bands of localized deformation with increasing crystal sizes in them and a decreasing a bend-torsion of crystalline lattice and the associated stresses, which are in (1.5-2) lower in comparison with the state after deposition. For coatings with a graded-layered structure, the role of the interfaces of the layers is determined, which prevents the coating from peeling off from the substrate, as fracture propagation sites; an increase in the bend-torsion values of the crystal lattice under the top of the imprint across the entire thickness of the coating, as well as a change in the shape and dimensions of the crystals (coherent scattering regions) in the surface nanocrystalline region were established. The obtained data on the structure modification are discussed on the basis of model ideas corresponding to the scheme of the indentation stress state and deformation mechanisms in the nanocomposite system.

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THE STUDY OF CONDITIONS OF Al_2O_3 COATINGS DEPOSITION BY REACTIVE EVAPORATION OF ALUMINUM IN THE DISCHARGE WITH HOLLOW ANODE

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Low-temperature deposition of nanocrystalline Al_2O_3 coatings by vacuum plasma methods is implemented in a certain range of values of current density and ion energy on the growing coating surface [1]. For the high-rate deposition (~ 10 μm/h) the considerable intensity of the ion assistance is require. Such requirements are met by the arc evaporation method, which, however, doesn`t provides the deposition of the Al_2O_3 coatings with a high content of thermostable α-phase at temperatures below 700 °C [2]. An alternative approach used in the present work is based on the high-density discharge with self-heating hollow cathode, anode-crucible and additional hollow anode providing an increase in the degree of ionization and dissociation of oxygen. This type of discharge is burning steadily in a wide range of discharge current ($1\text{-}10^2$ A) and gas pressure (0.01-0.1 Pa) in an argon flux and oxygen filling the anode part. It allows heating the crucible up to high temperatures and thus ensuring rapid growth of Al_2O_3 coatings.

The aim of this work was to determine the parameters of ion flow generated in the high-current discharge with self-heating hollow cathode, anode-crucible and additional hollow anode; to deposit Al_2O_3 coatings by reactive anode evaporation of aluminum with intensive ion assistance; to determine the range of current density and ion energy, within which α-Al_2O_3 coatings are formed.

It has been shown that double electric layer and a stream of accelerated ions are being formed near the output aperture of the hollow anode, which leads to a two-times increase in the density of ion current on a remote collector in comparison with the value achieved in the mode with a flat anode. Discharge with the hollow anode is characterized by a high concentration of atomic oxygen resulting from electron impact dissociation of molecular oxygen in the hollow anode. According to assessments based on data about reaction rates in Ar-O_2 plasma [3], the concentration of atomic oxygen in the discharge with hollow anode increases more than 10^2 times.

We have deposited Al_2O_3 coatings on metallic substrates with an isostructural sublayer of chromium oxide at a temperature of 600 °C and deposition rate up to 6 μm/h. It has been shown that amorphous-crystalline coatings with the dominant phase γ-Al_2O_3 are formed in the discharge with the flat anode in a wide range of ion current densities and bias voltage. The proportion of crystalline phase in coatings reaches 0.8, their hardness is ~ 22 GPa. The formation of single-phase α-Al_2O_3 coatings requires discharging current range of 4-28 A and negative bias voltage 25-200 V in the mode with hollow anode.

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INVESTIGATION OF REFURBISHING TECHNIQUE FOR DIAMOND COATED TOOLS

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In recent years, diamond coated tungsten carbide (WC-Co) tools are widely utilized for their benefits in machining of non-ferrous alloys and polymer composite materials, especially carbon fiber reinforced plastics (CFRP). Refurbishing of such coated tools is important in economical aspect, due to their high cost and short tool life. One of the key point of the refurbishing is effective removal of diamond film from tool substrate. Another key point is deposition of diamond film on the treated tool substrate again. In present time, only few approaches were reported regarding refurbishing of diamond coated tools, but commercial cost-effective method is not found yet [1-2].

This study investigated effects of removing CVD diamond films by ion beams [3] in terms of substrate damage, adhesion, and subsequent deposition of CVD diamond films for WC-Co tools with complicated tool shape. Figure 1 shows surface conditions of WC-Co drill after key steps of the refurbishing.

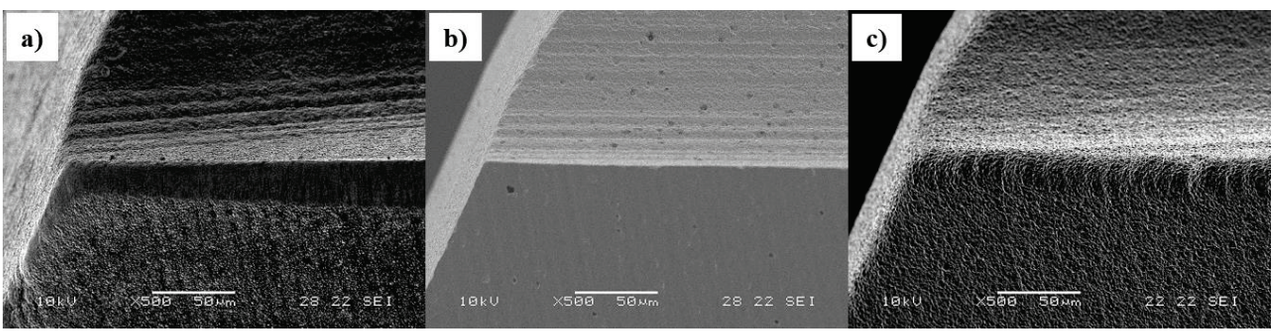

Fig. 1. The micrographs of WC-Co drill tip: a) before refurbishing, b) after removing diamond film and c) after deposition CVD diamond film.

Then cutting performance of refurbished tools was confirmed by cutting tests with drilling of series of consecutive holes in CFRP workpiece. Cutting test results shown in the Figure 2 indicate comparable cutting performance of refurbished tool to new one. Therefore, it is concluded that this technique is suitable for practical application.

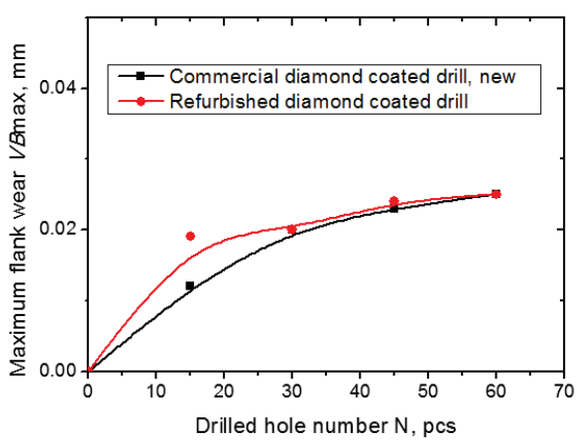

Fig. 2. Maximal flank wear as a function of drilled holes number (Drill Ø7.95, V=60 m/min, n=2400 rpm, f=0.04 mm/rev).

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PECULIARITIES OF METAL COATINGS DEPOSITION USING MAGNETRON SPUTTERING SYSTEMS WITH HOT AND EVAPORATIVE TARGETS

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Our research shows that the use of magnetron sputtering systems (MSS) with highly heated and evaporative metal targets gives possibility significantly (by one or two orders of magnitude) increase the deposition rate of coatings in comparison with conventional magnetrons. For this, special designs of the cathode assembly must be used, in which the target is thermally insulated from the cooled magnetron body.

We developed mathematical and numerical models of thermal and emission processes in cathodes with thermally insulated targets. It was taken into account that the target substance undergo melting and evaporation. A methodic for calculating the flux density of deposited particles and the growth rate of coatings and a structure of energy balance on the substrate are proposed. A program code for calculating the substrate temperature is developed too.

Calculations on the developed models in combination with experimental studies have made it possible to reveal the features of the mechanisms of atomic emission and transfer of erosive matter from the target to the substrate during operation of direct current and high current pulsed magnetrons, depending on their power and a number of other parameters. Cases of liquid-phase targets placed in crucibles (copper in molybdenum and graphite), hot solid targets from metals, having a high rate of sublimation (chromium, titanium), as well as deposition of coatings from ferromagnetic materials (nickel) were considered. Power ranges of MSS were determined, in which the evaporative component is dominant. The possibilities of modern MSS for increasing the deposition rate of coatings from various metals have been studied. For example, the productivity of copper coatings deposition during the operation of MSS with evaporation of the target can be increased by 10–100 times. The results of our calculations are in good agreement with the experimental data.

The combination of calculations and experiments revealed factors that affect the rate of erosion of targets and the deposition of metal coatings. The most important are the emissivity of the crucible surface, binding energy of the target atoms, dependence of the saturated vapor pressure of the target material on temperature, configuration of thermal conductive isolation of the target. For pulsed MSS, it is clarified that the target temperature and evaporation rate practically do not change between the current pulses. Here, the evaporation component of the erosion flow is determined by the average power of the MSS source averaged over the period. Due to evaporation, the rate of deposition of metal coatings during the operation of high current pulsed magnetrons is not less than that of a direct current MSS with the same value of the power averaged over the period.

The possibility of transferring MSS with the evaporating copper target to the self-sputtering mode without sputtering gas was studied. The parameters of MSS are found when MSS functions stably at a pressure in vacuum chamber of 0.01 Pa. The influence of the target temperature and atoms emission rate on the development of self-sputtering was investigated. It is found that the erosion coefficients of metal targets of MSS in the case of evaporation reach several tens of atoms per an ion. This is approximately an order of magnitude higher than the sputtering yields. Due to this fact, the coatings deposition during the operation of MSS with the evaporation of targets under self-sputtering conditions takes place practically without reducing the deposition rate in comparison with the MSS modes in which the sputtering gas is supplied.

We undertook a study of influence of the deposition conditions with targets heating and evaporation on the properties of the formed coatings. It turned out that it is ambiguous. Using copper, chromium and nickel coatings as an example, we revealed that surface morphology, growth directions, mechanical properties and adhesion depend in a complex way on the combination of the heat flux on the substrate and the flux density of the deposited atoms.

The study was supported by the Russian Foundation for Basic Research (Project No. 18-08-00454). Calculations are carried out at Tomsk Polytechnic University within the framework of Tomsk Polytechnic University Competitiveness Enhancement Program grant.

TRIBOLOGICAL PROPERTIES AND FEATURES OF DESTRUCTION OF ANTIFRICTIONAL MAGNETRON-PLASMA COATING Ti-C-Mo-S ON STEELS AND TITANIUM SUBSTRATES UNDER DIFFERENT LOAD-SPEED REGIMES

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In this work authors investigated the effectiveness of Ti-C-Mo-S antifriction coating deposited by a magnetron-plasma method [1] on samples from different materials under different load-speed friction regimes. The titanium alloy VT6, stainless steel 20X13 and steel 40X were chosen as the materials of the samples. Friction tests were carried out according to the scheme pin-on-disk in two regimes: the sliding speed of the indenter on the sample surface 50 cm / s at load 1 N and 5 cm / s at load 5 N. The friction coefficient changing by the number of disk rounds and character of the coating wear was investigated. Tracks of wear of the coating after friction tests were investigated by optical and electronic scanning microscopy.

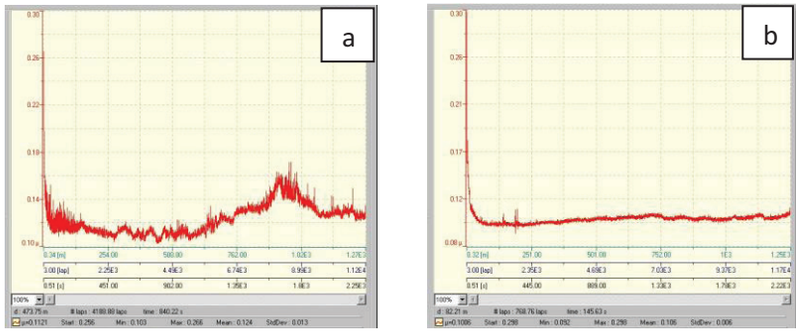

Fig.1. The value of the friction coefficient μ depending number of rounds on substrates of 20X13(a) and 40X(b) steels. Load 1 N, sliding speed 50 cm / s.

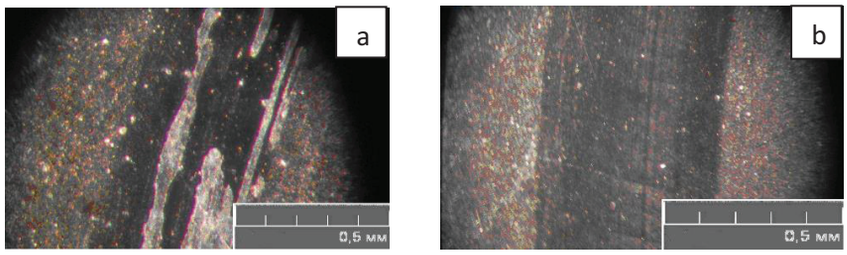

Fig.2. Wear of the coating on substrates of 20X13(a) and 40X(b) steels after friction tests. Load 1 N, sliding speed 50 cm/s.

1. The low value of the friction coefficient during tests for both load-speed regimes is observed: for the load 1 N in order 0.11 ÷ 0.12 and for the load 5 N in order of 0.07 ÷ 0.075.

2. The difference in the values of the friction coefficients and the wear characteristics of the coating at different friction regimes is due to the difference in the mechanisms of formation of the "third body" [2] between interacting materials and a more pronounced effect of removing particles of the coating from the wear zone at high friction speeds.

3. The difference between the frictional interaction and the character of the coating wear on a steel 20X13 in comparison with titanium alloy and steel 40X is described. This is due to the different adhesion strength of the coating with the selected materials of the samples.

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INFLUENCE OF NITROGEN CONCENTRATION ON STRUCTURE, COMPOSITION AND PROPERTIES OF MONOLAYERED NITRIDE COATINGS DEPOSITED BY VACUUM ARC PLASMA-ASSISTED METHOD

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Nitrides of transition metals are widely used as wear-resistant hard coatings on surface of materials and products for multiple increase of their main characteristics [1]. DC vacuum arc discharge (VAD) has been used for the coating deposition technologies for more than 30 years [2]. In present work vacuum arc plasma-assisted method is used for deposition of nitride coatings.

The experimental works are carried out on original automotive ion-plasma QUINTA set-up (IHCE SB RAS, Tomsk, Russia) [3], which is equipped by two type plasma generators: arc evaporators based on self-sustained discharge with cathode spot and non-self-sustained arc discharge with combined thermionic and hollow cathode [4].

The purposes of the work are synthesis of monolayered nitride coatings by vacuum arc plasma-assisted method with different concentration of nitrogen and investigation of influence of nitrogen concentration on its structure, composition and properties. To vary the nitrogen concentration of coating the share of nitrogen ion in gas-metal plasma will be varied by change of parameters of gas-discharge plasma at constant working gas pressure and arc current of arc evaporator.

The results of investigations show the increase of nitrogen concentration in coating composition at increase of arc current of gas-discharge source and the increase of the share of nitrogen ion in gas-metal plasma at constant working gas pressure and arc current of arc evaporator. In addition, phase composition, structure and properties are significantly changed.

In the work new quickly-response method for change of nitrogen concentration in coating composition and its properties and structure is showed. That can be used not only for deposition of monolayered nitride coatings, and for that of multilayered nanosized metal/ceramics and gradient (with variable composition on the thickness) coatings.

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DIAGNOSTIC SYSTEM «YUNA» FOR DESPERSE PHASE PROPERTIES CONTROL IN PLASMA AND LASER POWDER DEPOSITION PROCESSES[1]

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The paper presents the possibilities of the original optical complex «Yuna», designed for diagnostics of the dispersed phase characteristics in powdered flows in plasma and laser coating processes. The diagnostic complex is an automated system consisting of an optical registration unit, a synchronization module, a positioning module and control software on operating at a personal computer. The optical registration unit includes a brightness channel based on a digital video camera with a narrow-band optical filter installed in its optical channel and a spectral channel based on an optical range photospectrometer. The both channels have common field of view. The brightness channel allows to record images of individual moving of the dispersed phase particles - tracks, and the spectral channel - to register the total radiation spectrum of the two-phase flow, which is formed due to the thermal radiation of the ensemble of powder particles and the intrinsic radiation of the plasma. Software processing of data allows to determine the spatial distribution of particles in a two-phase flow, the statistical distributions of the magnitude and direction of their velocity. The temperature of the emitting particles is measured using a new method of spectral brightness pyrometry (SBP) [1]. The positioning system of the optical unit allows scanning of a two-phase flow and obtaining maps of the distribution of particle characteristics in a region up to 300 mm in size with a spatial resolution of down to 20 μm. The diagnostic complex allows to observe particles with a size of 15 μm and above, the range of measured velocities is 1-1000 m/s, measured temperatures from 1500 K and above. For diagnostics of gas-powder flows with cold particles, a pulsed LED illumination system is used.

Examples of the application of the diagnostic system in the technologies of thermal synthesis of products: plasma [2, 3] and plasma-arc spraying [4], laser cladding, as well as testing of high-temperature materials are presented.

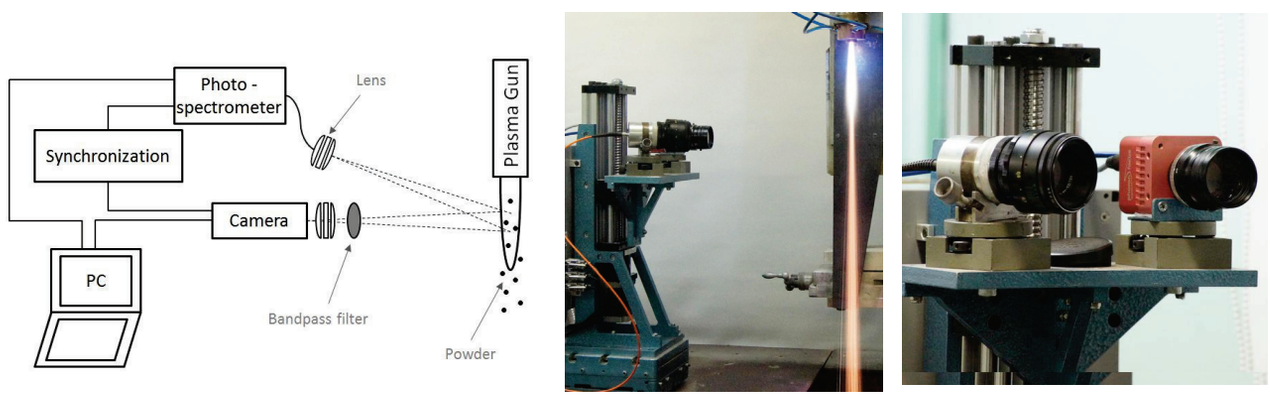

Fig. 1. Schematics of «Yuna» setup (left), its operation during plasma spray diagnostics (center) and close-up view of the optical unit.

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ZINC SUBSTITUTED HYDROXYAPATITE COATINGS' STRUCTURE MANIPULATION BY VARIATION OF RF MAGNETRON SPUTTERING PARAMETERS [1]

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A nosocomial infection during a dental or endoprosthesis replacement is still one of the significant problems for healthcare. In some of the cases, bacteria become antibiotic resistant. It is further lead to aseptic loosening of an implant. In order to overcome this problem, calcium phosphate coatings with antibacterial agents deposited on the implants are suggested [1].

Zinc ions are well-known not only for its antibacterial properties but as a component of many enzymes. It is an essential trace element for tissue regeneration [2]. Therefore, Zn-substituted hydroxyapatite (HA-Zn) coatings as a thin osteoconductive bactericidal coating are proposed as the promising material1. Moreover, with an RF magnetron sputtering method it is possible to deposit not only amorphous but crystalline coatings [3]. Control over the coatings crystalline state will allow to manipulate the coatings biodegradation rate.

In this work nanocrystal, equiaxed grain and amorphous HA-Zn coatings were deposited on the commercially pure titanium substrate by the RF magnetron sputtering method. Several regimes of the coating deposition with the variation of the substrate temperature during the sputtering process were performed. Cytotoxicity tests were performed using the mouse myoblasts C2C12 cell line.

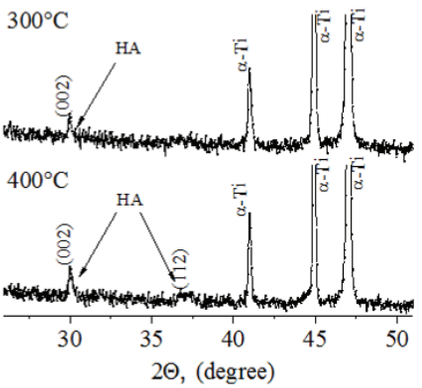

Fig. 1. XRD diffractograms of the HA-Zn thin films deposited under 300°C and 400°C substrate temperatures.

As we previously showed [3], it is possible to deposit nanocrystalline equiaxed grain structured HA-Zn coatings. However, for the clinical practice, the deposition of crystalline columnar-like structured HA-Zn is also of interest. In our work, we employed the fact that the coating deposition under elevated substrate temperatures leads to increased adatoms energy and movement. It also may result in partial crystallization of the coating. As it is seen in figure 1, starting from the temperature of 300°C coatings' recrystallization with texturization and preferential orientation of HA crystals in the (002) plane occurs. The effect is more pronounced at the temperature of 400°C and additional growth orientation for HA crystals in the (112) plane. Coatings of HA-Zn deposited on the Ti substrate under evaluated temperature have a crystalline structure. Biological tests showed the absence of the toxic effect of the HA-Zn coating to the C2C12 cell line.

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[1] This work was supported by the state program of fundamental research of Russian Academy of Science for 2017–2020, No.III.23.

THE FORMATION OF THE STRUCTURE, PHASE COMPOSITION AND PROPERTIES OF THE ELECTRIC EXPLOSIVE WEAR – RESISTANT COATING AFTER ELECTRON BEAM PROCESSING[1]

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One of the main factors limiting the service life of the machine parts of different purposes is their mechanical wear. The failure of machine parts is mostly caused by the local wear of working surfaces in the sites of their intensive interaction with the working environment or mating part. Creation of new bulk three – dimensional composite materials being the main way of the increase in reliability and durability of mechanism and machine parts is becoming a more problematic one because of the deficit and high price of the composite additives. It increases their cost. In these conditions it is economically and technically profitable to develop a radically alternative approach to the creation of the materials at which the mechanical strength of the part is ensured by the application of economic substrates and the special surface properties – by the continuous or local formation of the composite coatings on it, whose properties are consistent with the service requirents. The economy may reach 90% at this approach. The expert estimates substantiate this tendency. One of the main directions in condensed – state physics is the development of methods of increase in service characteristics of different materials. In view of this fact the hardening of the part surface working in the conditions of intensive mechanical wear is the topical problem of new modern technology development. Composite materials of TiC-Mo, TiC-Ni, TiB_2-Mo and TiB_2-Ni systems possess a high wear resistance. The coatings of this composition can be formed by method of electric explosion spraying (EES) making it possible to obtain the pore – free coating of high quality. The purpose of the research consisted in the modification of electric explosion composite coatings of TiC-Mo, TiC-Ni, TiB_2-Mo and TiB_2-Ni systems on Hardox 450 steel by high intensity electron beam and the investigation of their structural phase state and properties.

It is established by SEM that after EES of coatings of the systems under study a number of morphological relief features are formed on the surface. Among these are the numerous deformed crystalized microglobules with representative diameters from 1 to 50 μm falled out on it from the rear of the jet and distributed nonuniformly. The microglobules are formed by the foil metal and powder particles used for EES. The influxes caused by the radial flow of metal from the center of spraying spot to the periphery, microcraters, microcracks. In the present research, the coatings of TiC-Mo, TiC-Ni, TiB_2-Mo, and TiB_2-Ni systems were applied to the surface of Hardox 450 steel by the method of electrospray coating. After that, the electron-beam processing of the coatings was carried out. It is established that after electro-explosive spraying of the coatings of the systems under investigation, a number of morphological features of the relief are formed on the surface: the deformed crystallized microglobules, influxes, microcraters, microcracks, layers. After the electron-beam processing of the coatings, the microglobules, microcraters and microcracks disappear on their surfaces, a polycrystalline structure is formed, in the bulk of which the structure of cellular crystallization is observed. The roughness of the coatings after electron beam processing is 1.1 ... 1.2 μm. It is established that the thickness of the layers of electrically explosive coatings modified by an electron beam, depending on the surface energy density, is linear. Its maximum value is observed for the TiB2-Mo system, the minimum one – for the TiC-Ni system, which is explained by their thermophysical properties. In the coatings the following substructures are revealed: cellular, band, fragmented, subgrain, and also the grains with chaotically distributed dislocations and the dislocations that form nets. The electron beam processing of coatings leads to the formation of a composite filled structure throughout the entire section of the remelted layer, the formation of a more dispersed and homogeneous structure in it than in the coatings without electron beam processing. The dimensions of titanium carbide or titanium diboride in inclusions in a molybdenum or nickel matrix are reduced by a factor of 2-4 compared with their dimensions immediately after electroexplosive spraying. Particles of the second phases are found in the volume of molybdenum or nickel grains and at the boundaries: titanium carbide or titanium diboride.

[1] This work was supported in part by a grant of the President of the Russian Federation for state support of young Russian scientists having candidate of sciences degrees (project no. MK-1118.2017.2) and by the Russian Foundation for Basic Research (projects no. 16-32-60032 mol_a_dk, 18-32-00075 mol_a).

STRUCTURE OF SnO_2-Ag COATING FORMED ON COPPER BY ELECTROEXPLOSION METHOD[1]

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On evidence derived from the International association "Interelectromash" the segment of failures in the work of electroequipment for the reason of contact instrument failure ranks first among the other troubles and it amounts to 26%. To reestablish the work the contact is substituted for the new one. The combination of various and simultaneously incompatible requirements are characteristic of the electric contact's materials. For example, they need to have a high hardness, the melting temperature, electro-and heat conductivity, electrocorrosion and corrosion resistance combining with the absence of welding and bridge-formation. The application of the powder metallurgy methods enables to realize in one material the diverse and contradictory complex of properties that should be possessed by the electrocontact material. Nowadays, a large number of electrocontact material are developed for application in different service conductions. Their composition includes, as a rule, a matrix possessing a high electric conductivity and a high-melting component (filler) with high wear-and electroerosion resistance. The composite materials based on silver, copper copper-nickel, aluminum matrix and a high melting filler are the most promising ones for the manufacture of contacts. The following systems belong to them: W-Cu, Mo-Cu, W-C-Cu, Mo-C-Cu, Mo-C-Cu, Ti-B-Cu, Ti-B-Cu, TiB_2-Cu, TiB_2-Al, W-Ni-Cu, Mo-Ni-Cu, Cr-C-Cu, Cr-Cu, CdO-Ag, SnO_2-Ag, W-Ag, Mo-Ag, W-C-Ag and Mo-C-Ag and others.

According to the expert estimates the volume of the word composite, materials market in 2016 amounted to nearly 17 mln Toms. In the structure of the word consumption of the composite materials and products from them according to the sectors of economy share of the composite materials consumed by electronics and power industry amounts to 21% among other fields of industry and it is the main driver of growth. The volume of domestic production of arc-resistant electric contacts from the composite materials amounts of 18 mld rubbles. If it is remembered that in fact, the wear of the contact before its failure and substitution for the new one does not exceed 50% then nearly 9 mln rubles are spend in vain only in the Russian Federation.

As the process of the material's failure begins with its surface, for a number of practical applications, for example, the hardening of contact surfaces of medium and heavy duty switches and commutation apparatuses, it is promising to from the protective coating because in this case it is important to have electroerosion resistance only on the surface of the contact instead of bulk. Economically and technically practical to develop the approach to the creation of the materials wherein the mechanical strength of the part is achieved by the use of the economic substrates and the special properties of the surface – by the continuous or local formation of the composite coating on it whose properties correspond to the service requirements. The economy in this approach may reach 90%. The expert estimates substantiate tendency. One of the primary directions of condensed state physics is the development of the methods of the increase in the service characteristics of different materials. With regard to it, the surface hardening of arc-resistant electric contacts in the topical problem of development of the new modern technologies.

The studies of the phase and elemental composition of the surface layer copper electric contact of KPV-604 contactor subjected to electroexplosion spraying of the composite SnO_2-Ag-system coating were done by the method of transmission electron microscopy. The scale of the elemental structure of the coating's surface varies within a very wide interval after the electroexplosion spraying – from hundreds of micrometers to tens of hundreds of nanometers. According to the morphological feature two layers may be distinguished in the coating's volume: the coating proper and the thermal effect layer smoothly transferring to the bulk of the sample. The nanocrystalline structure was detected. The main phase of the coating are SnO_2, Ag_3Sn, $Cu_{10}Sn_3$, Cu_3Sn, Cu_6Sn_5, Ag_4Sn and CuO. The volume of copper adjacent to the coating has a structure indicative of the high level of deformation of the sample's surface layer in the electroexplosion method of the coating's formation.

[1] This work was supported in part by a grant of the President of the Russian Federation for state support of young Russian scientists having candidate of sciences degrees (project no. MK-1118.2017.2) and by the Russian Foundation for Basic Research (projects no. 16-32-60032 mol_a_dk, 18-32-00075 mol_a).

DEVELOPMENT OF ROBOTIC MICROPLASMA SPRAYING TECHNOLOGY FOR APPLYING BIOCOMPATIBLE COATINGS ON MEDICAL IMPLANTS[1]

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The paper presents the main results of development of a new technology for applying two-layer titanium and hydroxyapatite coatings on medical implants made of titanium alloy using a robotic complex of microplasma processing.

The synthesis of hydroxyapatite powders suitable for applying biocompatible coatings on medical implants has been carried out. A wire from titanium of BT-1-00 (GOST 19807-91) of 0.3 mm in diameter has been used for the titanium coatings application. Microplasma spraying of the coatings from titanium wires and hydroxyapatite powders onto substrates made of medical titanium alloy has been applied on microplasma processing areas based on the industrial robot Kawasaki RS-010LA (Kawasaki Robotics, Japan) in D.Serikbayev East Kazakhstan State Technical University with the use of microplasmatron MPN-004 manufactured by E.O. Paton Electric Welding Institute (Ukraine). Grade 5 ELI titanium alloy samples produced by Ust-Kamenogorsk Titanium and Magnesium Plant JSC (JSC UK TMP) have been used as substrates for microplasma spraying. New software has been developed to solve the problem of providing the desired trajectory of the robot-manipulator with a microplasmatron maintained on.

X-ray structure phase analysis, transmission and scanning electron microscopy and metallographic analysis have been used to study the structure-phase composition of coatings and substrates. The influences of the main parameters of the microplasma spraying on morphology and structure-phase transformations in coatings have been studied.

The study has been carried out with the use of the metallographic microscope Olympus BX-51 (Japan), the transmission electron microscope JEM-2100 (JEOL, Japan), the X-ray diffractometer X'Pert PRO (PANalytical, the Netherlands), the scanning electron microscope JSM-6390LV (JEOL, Japan) with energy dispersive analysis by INCA ENERGY (Oxford Instruments, UK), The scanning electron microscopy images of coatings cross-sections have been processed using ZAF/PB, Micro Capture, Atlas computer-aided programs to evaluate the porosity of the coatings.

The composition and regimes of microplasma spraying of the two-layer coatings for titanium implants, including a sub-layer of a porous titanium coating with a thickness of 200 ... 300 μm with a pore size of 150 ... 300 μm, and an upper layer of hydroxyapatite with a thickness of up to 200 μm with a high crystallinity (88-98%), controlled by changing the spraying regime have been developed. Recommendations for the preparation of the surface of Grade 5 ELI titanium alloy for microplasma spraying of the coatings from biocompatible materials, including gas-abrasive treatment regimes, have been elaborated.

Technological guidelines and software have been developed enabling to implement robot-aided microplasma spraying of coatings from biocompatible materials on medical implants. In the future, this will allow implementing new technologies for the production of various types of high-quality and affordable medical implants made of titanium alloys produced in the Republic of Kazakhstan

[1] This work was supported by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan within the framework of program-targeted financing for 2017-2019 on the scientific and technical sub-program 0006 / PCF-17 "Manufacture of titanium products for further use in medicine"

INVESTIGATION OF A COATINGS BASED ON INTERMETALLICS OF TI-AL SYSTEM ALLOYED WITH CHROMIUM BY VACUUM-ARC PLASMA

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Cutting properties of the instrument are determined by a complex of factors. That are depend on the chemical composition, structure and crystal-chemical structure of the tool material and determine the most important operational properties of the tool - microhardness, heat resistance, thermal conductivity, strength, toughness [1]. To increasing the life of tools with coated that working at shock loads is paid particular attention. The directions for improving the life-cycle of cutting tools with coating are the development and improvement of the multilayer coating design, the improvement of coating technology and the development of new coating compositions [2]. So, multilayer coatings are characterized by a smooth transition of physical, mechanical and thermal properties of layers from the tool base to the upper boundary layer of coatings. Recently, coatings based on intermetallides of the Ti-Al system are of great interest, due to their high physical and mechanical properties [3]. Therefore,in this paper is proposed to apply composite multilayer coatings based on intermetallides of the Ti-Al system alloyed with chromium to improve the performance properties of the grooving tools working at shock loads.

In the work, the influence of technological parameters on the chemical composition of coatings alloyed with Cr was studied. The mechanical properties of alloyed coatings based on intermetallics of the Ti-Al system depending on the percentage contents of Cr are studied . For to determine the coefficients of friction and wear resistance were carried out Tribological tests by the Nanovea Tribometr equipment. Based on the results of the studies, the pilot lot of the slotting tool (fig. 1 a) was processed and production tests were carried out. The results of the production tests (fig. 1 c) showed an increase in tool life of 10 times compared to the original tool without coating.

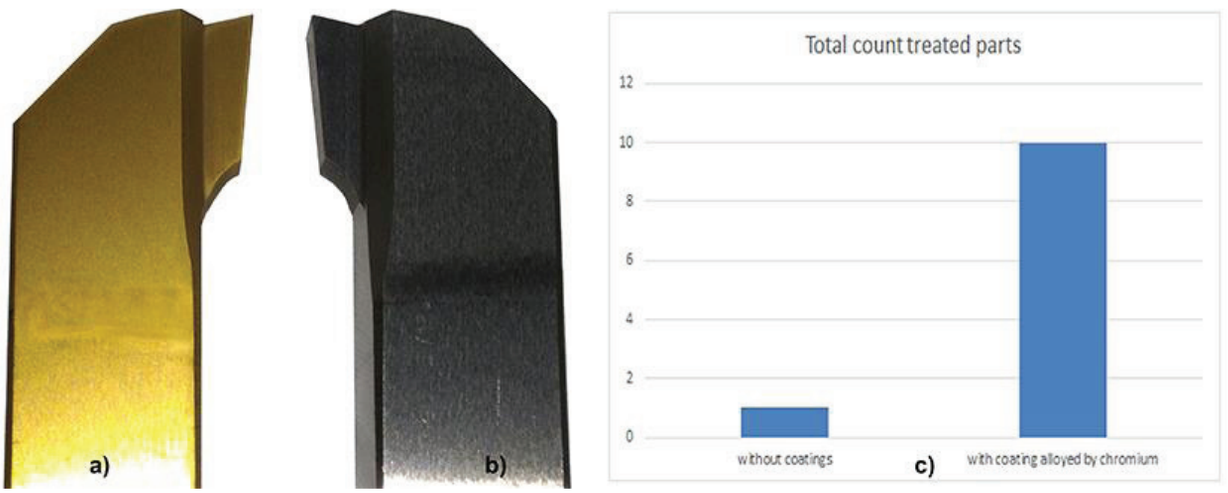

Fig. 1. Slotting tool:

a) with coating alloyed by chromium; b) without coatings; c) results of compared production tests.

The results of the production tests (fig. 1 c) showed an increase in tool life of 10 times compared to the original tool without coating.

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MATHEMATICAL MODEL OF DEPOSITION PROCESS OF COMPOSITE COATINGS BASED ON INTERMETALLIC TI-AL SYSTEM BY VACUUM ARC PLASMA

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This work is devoted to mathematical modeling of the process of deposition of composite coatings based on intermetallics of the Ti-Al system from vacuum-arc plasma, considering diffusion processes between alternating layers in order to predict the phase composition of the coating [1,2,3]. The physical and chemical processes occurring during layer-by-layer deposition of multilayers coatings based on intermetallics of Ti-Al system by vacuum arc plasma in thin films were investigated. The processes occurring in the deposition of coatings based on intermetallic Ti-Al system by vacuum arc plasma were mathematically described. Diffusion processes were simulated using the finite element method. Diffusion processes occurring between layers of Ti and Al were modeled using Fick equations. Software based on mathematical model that allows to calculate the optimal deposition modes of coatings based on intermetallic Ti-Al system was developed. Influence of the deposition conditions on the content of intermetallic compounds in the coating was established by X-ray diffraction methods.

The results of the calculations showed (fig. 1) that when the location of the design point in the vacuum chamber changes from the center of the working table (r0 = 0) to the point (R = 22cm) located close to the arc-evaporators, the widths of (a, c) regions of deposition of pure Ti or Al are identical and remain Equal to 1200. While, the region (b) in which the simultaneous deposition of titanium and aluminum occurs is reduced from 600 (r0 = 0) to 0 (at R = 22cm), since. The angle between the normal vector of the surface being processed and the normal vectors of the cathodes of electric arc evaporators is increased.

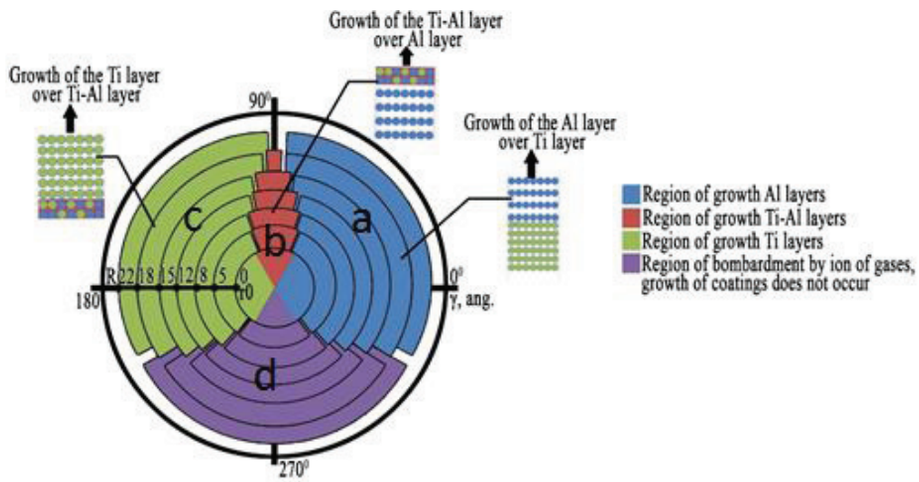

Fig. 1. Graphical representation of growth areas of Al, Ti-Al, Ti layers.

Results of mathematical model of deposition multilayer coatings considering diffusion processes were compared with experimental data. It is established that in the layer-by-layer deposition of titanium and aluminum, there are two mechanisms for the formation of intermetallic phases: due to diffusion processes and chemical reactions of samples running on the surface. A correlation between the percentage content of intermetallic phases in the coating and the diffusion processes between titanium and aluminum was established.

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DEPOSITION OF SILICON CARBONITRIDE COATINGS IN THE PLASMA OF HIGH-CURRENT DISCHARGE WITH SELF-HEATED HOLLOW CATHODE[1]

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The results of study of the influence of discharge burning and current modes, contents and pressure of gas environment, as well as sample position relative to the vapors sources and sources of plasma, on the properties and composition of coatings obtained by usage of silicone precursor (hexamethyldisilazane) are reported in the present work. On the surface of samples made of quartz glass and stainless steel, silicon carbonitride (SiCN) coatings up to 8 μm thick with hardness up to 14 GPa were obtained during 2 hours at 200ºC. Coatings compositions were analyzed by FTIR method that showed that main absorption bands of SiCN(H) system were present in all the spectra (Fig. 1). When the discharge burns in nitrogen flow with growing N_2 content, the intensities of absorption peaks 2950 cm^{-1} (C-H), 2200 cm^{-1} (Si-H) and 1150 cm^{-1} (Si-NH) increase, that can explain coatings hardness decline, as in [1].

It is shown that the coatings adhesion and homogeneity can be affected not only by value and type of bias voltage applied to the samples, but also by high-current discharge burning mode. Coatings obtained in plasma of continuous discharge are characterized by better adhesion and homogeneity than those got in pulse plasma.

The increase of discharge current leads to growth of the rate of coatings deposition and their thickness that is probably caused by growth of the degree of decay of hexamethyldisilazane molecules and drop of the content of hydrogen-containing molecular fragments that substantially reduce the mechanic properties of SiCN-coatings [2]. It was also shown that samples position relative to the vapors sources and sources of plasma affects thickness, composition and hardness of the coatings. When the distance from the atomizer is 2 times increased, the rate of coatings deposition drops only by 20-30%, though vapors concentration is 4 times lower. This fact can be explained so: the samples near the atomizer form their coatings from the initial precursor molecules, whereas the distant samples “catch” already “activated” vapors that have crossed thick plasma flow.

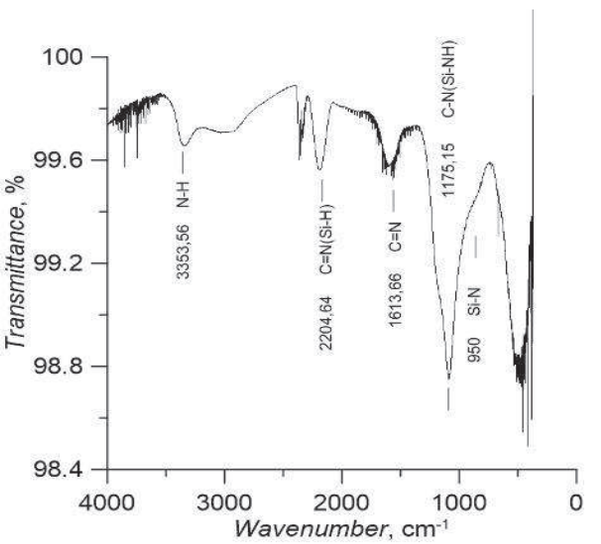

Fig. 1. IR-spectrum of the coating on steel 12X18H10T. Current 20 A, pressure N_2+HMDS 1 mTorr (Q_{N2} : Q_{HMDS} = 3:1).

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HIGH THERMAL STABILITY OHMIC CONTACTS TO NITRIDE SEMICONDUCTORS WITH REFRACTORY METAL SIDEWALL DIFFUSION BARRIER DEPOSITED BY MAGNETRON SPUTTERING[1]

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GaN HEMTs are generally promising candidates for switching power transistors due to their high breakdown strength and the high current density giving a low on-resistance [1]. Low-resistance ohmic contacts are needed to reduce losses and self-heating [2]. Conventional Ti/Al/Ni/Au based ohmic contacts require the high annealing temperature (>800 °C). These temperatures can also melt Al, creating unwanted surface roughness of the contact. Thus making it difficult to implement gate-first process, which is useful for the self-aligned process development. Alternative ohmic metals for gate-first GaN process are being sought using Mo, Hf or Ta, for example, which can be annealed at lower temperatures. In work [3] are presented ohmic metal contact scheme based on Ta and Al only. Overall, the minimum contact resistance found for Ta/Al/Ta based ohmic contacts were 0.28 Ohm mm after annealing at T = 550 °C. However Ta-based ohmic contacts have the some disadvantages. At the first, there are sold state phases of Al film observed after low temperature annealing at T < 660 °C. At the second, Aluminum can oxidize at ohmic sidewalls and can react with wet chemicals. At the third, GaN HEMT use Au based interconnects. Once Au can diffuse to Al-based ohmic contacts at sidewalls to make the high resistivity intermetallic compounds.

In this study, we report the Au-free low temperature Ta/Al based ohmic contacts with the sidewall refractory diffusion barrier deposited by magnetron sputtering to improve ohmic performance and prevent Au sidewall diffusion into Al based ohmic from interconnects.

That have been shown, the Ta/Al ohmic contacts fabricated with the Ta sidewall diffusion barrier have the lower contact resistance (0.18 Ohm mm) and the better surface morphology after the high temperature annealing (Fig.1). Peculiarities of the improving low temperature Ta/Al based ohmic contact technology and mechanisms responsible for discovered phenomenon has been discussed.

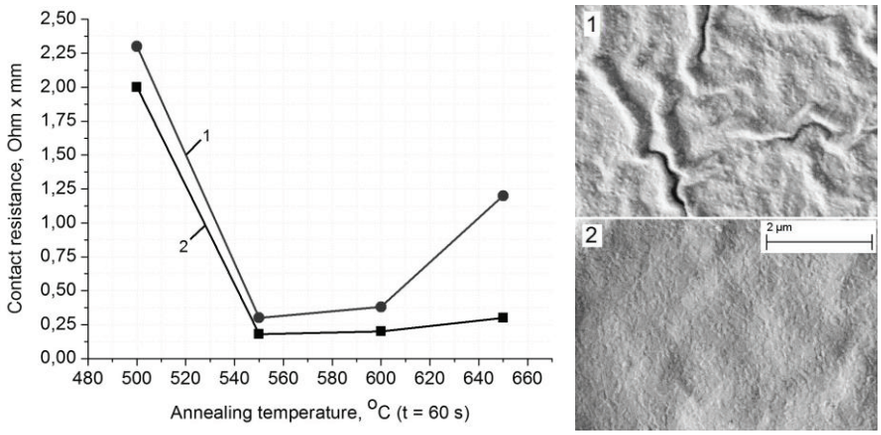

Fig. 1. The contact resistance of Ta/Al/Ta ohmic contacts without (curve 1) and with Ta sidewall diffusion barrier (curve 2) versus annealing temperature in nitrogen and SEM images of contact morphology after annealing at T = 650 °C during t = 60 s.

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MINIMIZATION OF THE ARC ENERGY IN HIGH CURRENT IMPULSE MAGNETRON SPUTTERING

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High current impulse magnetron sputtering (HIPIMS) is a relatively new technology of ionized physical vapor deposition (I-PVD), which provides plasma parameters commensurable with cathode arc evaporation and RF sputtering, without their fundamental drawbacks. One of the important problems of HIPIMS is the formation of electric arcs during coatings deposition. The transition of a magnetron discharge into an arc leads to the formation of defects in the deposited coating, due to local melting of the target and micro-droplets formation.

High pulsed power density on the target (up to several kW/cm^2) increases the probability of arcs formation during HIPIMS in contrast with conventional magnetron sputtering in DC mode. To prevent arcing, the pulse duration or the amplitude of the discharge current is reduced. These measures help to reduce the frequency of arcing events, but do not guarantee their complete elimination, especially in reactive processes (R-HIPIMS). Therefore, one of the main tasks of the HIPIMS power supply system is to detect an arc fast enough and limit the amount of energy release to prevent particles formation from an arc once it occurs.

In this paper, we present the results of an analytical and experimental study of various methods for arcs detecting and suppressing in the HIPIMS process. It is demonstrated that the effective reaction of the power supply system allows significantly reduce the arc energy (see Fig. 1). In addition to the arc detection rate and the delay in triggering the arc protection system, an important role is played by the possibility of energy recuperation stored in the output circuit of the power supply system.

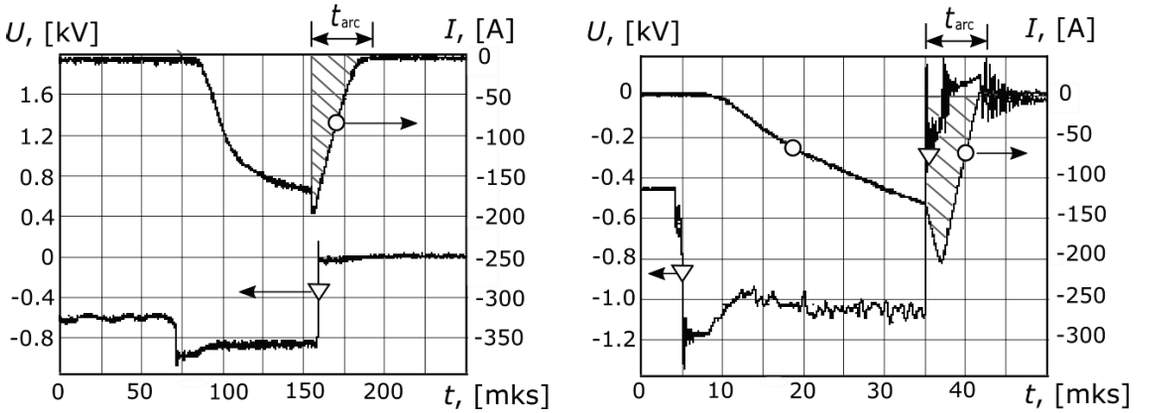

Fig. 1. Form of current and voltage at the output of the power supply when an arc arises in the HIPIMS process: a) slow response of the arc suppression system (t_{arc} = 30 μs; E_{arc} = 250 mJ), b) fast reaction (t_{arc} = 7 μs; E_{arc} = 50 mJ).

PULSE FORM MODIFIER FOR DUAL MAGNETRON SPUTTERING

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Dual magnetron sputtering (DMS) systems are widely used for the deposition of dielectric coatings for industrial applications, especially for the treatment of large area substrates, because they provide high productivity and stability of the deposition process. DMS allows cardinally solve the "disappearing anode" problem and prevent the formation of electric arcs [1]. Despite the presence of several great advantages, dual magnetron sputtering systems also have some drawbacks, which are connected with the periodic principle of operation and the presence of a magnetic field at the anode surface. DMS have a lower coating deposition rate and a higher value of the impulse current, relative to a single magnetron sputtering systems at the same average discharge power [2]. To compensate for these effects, it is necessary to further increase the operating power and use more expensive semiconductor elements, which leads to an increase in the cost of the power supply system.

An alternative solution to this problem is the use of an adapted impulse power supply. The idea is to modify the shape of the rectangular voltage pulses to transform the form of the current pulses from triangular to trapezoidal, as shown in the diagrams in Fig. 1. As a result, the maximum impulse current and, accordingly, the load on the semiconductor elements decrease. To solve this problem, manufacturers of power supplies use pulse formers with inductive energy storage devices and special snubber circuits [3,4].

In this work, a voltage pulse generator providing the complex form of voltage pulses for DMS systems based on a constant voltage source (non-inductive storage) and an additional pulse form modifier (PFM, Fig. 1) was realized. A description of the new device and the V-I characteristic of the DMS discharge is given. The obtained characteristics are compared with the characteristics obtained without PFM.

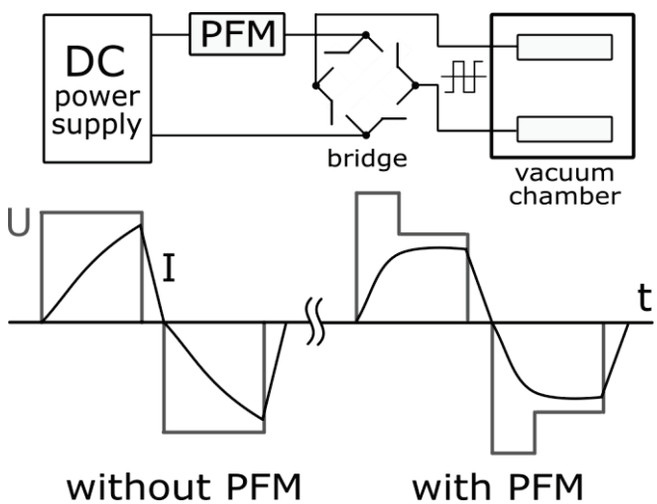

Fig. 1. Circuit of bipolar power supply for DMS, current and voltage pulse diagrams with PFM and without PFM.

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PROPERTIES OF THE POLYSILOXANE FILMS PRODUCED IN DISCHARGE INITIATED BY RUNAWAY ELECTRON BEAM[1]

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The results of experiments on the deposition of organosilicon films on plane surface of titanium and quartz are presented. The deposition of the films was carried out by PVCD method in a plasma of a pulsed-periodic runaway electron preionized diffuse discharge (REP DD) at atmospheric pressure in a nitrogen flow and hexamethyldisiloxane (HMDSO) vapor, which was used as a precursor.

Substrate surface was treated by plasma during 5 minutes for adhesion increasing. The process of film deposition by plasma lasted 30 minutes, then the substrate with the formed polymer was annealed at a temperature of 300° C. As a result, polymer films of 2-3 μm thickness, 2 GPa hardness and 70% absorption of radiation in the UVB-UVC region of the spectrum were formed on the surface. In the IR spectrum, bands corresponding to stretching vibrations of the Si-O-Si groups and a bands corresponding to deformation vibrations of the $Si-CH_3$ group are observed.

[1] The work is performed in the framework of the Russian Science Foundation (the project #17-72-20072)

INVESTIGATION OF THE FORMATION FEATURES OF GRADIENT STRUCTURES AT THE INTERFACE OF THE TIN COATING AND A-TI SUBSTRATE OBTAINED BY PLASMA-IMMERSION ION NITRIDING

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Currently, titanium and its alloys are promising construction materials, which have a complex of unique properties, including high specific strength, low density and high corrosion resistance. A factor that largely limits using of the titanium-based alloys is the low level of tribological properties due to the low hardness and tendency of titanium to adhere when working in friction pairs. One of the effective solutions to this problem is based on the formation on the titanium goods of a combined structure of a high-strength surface layer and a coating with high wear resistance. The durability of this structure is determined by the adhesive strength associated with the characteristics of the defective microstructure and phase composition, which can be controlled by changing the synthesis conditions. In this connection the task of this work is examination the features of the formation of gradient structures in the interface region of the TiN coating and the α-Ti substrate obtained in various regimes of plasma-immersion ion nitration and magnetron sputtering.

To elucidate the features formation of the structural-phase state surface layer of α-Ti substrate, a study of the depth variation of its elemental composition with the determination of the distribution profile and maximum values of the concentrations of the alloying elements has been made. The type and characteristics of the defective substructure and the second phases, crystallography and the level of local stresses at the interface of the coating-substrate were shows by methods of the transmission electron microscopy. To establish the physico-mechanical properties of the combined structure the measurement of nanohardness and experimental study of adhesive and cohesive strength with using the scratch testing methods were carry out. The characteristic features of deformation and fracture of the coating and substrate have been revealed in these tests. A discussion about relationship between the conditions of obtaining, deformation, fracture, and properties and structural-phase state in the interface of the coating and the modified substrate was made on the based of results obtained for the combined structure.

OPTICAL AND STRUCTURAL PROPERTIES OF NITROGEN-DOPED TITANIUM DIOXIDE THIN FILMS DEPOSITED BY MAGNETRON SPUTTERING

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During the last decades, titanium dioxide TiO_2 thin films have drawn lots of attention as they have potential applications in different areas [1,2]. Among the applications, we are presently most interested at nanostructured TiO_2 thin films, used for biomedical and photocatalytic purposes [3]. Besides, the self-cleaning function of implants, especially the stents, is also of great interest. Deposition of TiO_2 thin film has been proven to be capable of effectively improving the implants clinical properties; meanwhile based on the high photocatalytic activity of TiO_2 film, the self-cleaning of coated implants can be realized. Nitrogen-doping using different methods has been extensively investigated and shown to improve the photocatalytic performance of TiO_2 film in visible light region [4]; meanwhile in [5] has been found that N-doping may lead to anatase-rutile transition in TiO_2 film, which makes it possible to tailor film structure and red-shift film absorption edge at the same time. In this paper we present the results of studying the structure and properties of N-doped TiO_2 thin films, deposited onto biased substrate with different N_2/O_2 flow ratio.

Materials and methods. The N-doped TiO_2 thin films were deposited onto different substrates by means of a reactive magnetron sputtering system. Titanium target was sputtered in a mixed argon-oxygen-nitrogen atmosphere with different N_2/O_2 flow ratio: 0.3, 0.7, and 2.5. The Ar flow was kept as 1.4 mL/min. The total pressure in chamber was 0.1 Pa and power was 1kW. The substrate-target distance was 10 cm, films were deposited with substrate bias of –200 V. Thickness (*Th*) with measurement deviation (*q*), refractive index (*n*), extinction coefficient (*k*) and dispersion curve of N-doped TiO_2 film have been obtained using the spectroscopic ellipsometry method. Crystalline structure of films was determined from XRD and Raman spectroscopy data.

Table 1. Thickness, refractive index, extinction coefficient and deposition rate of N-doped TiO_2 films.

N_2/O_2 flow ratio	*Th*, nm	*q*, %	*n* (632.8 nm)	*k* (632.8 nm)	*v*, nm/min
0.3	188.6	5.8	2.25	0	3.14±0.10
0.7	219.9	9.8	2.14	0	3.67±0.16
2.5	203.8	8.4	2.11	0	3.40±0.14

As shown in Table 1, the maximum film thickness, and thus deposition rate (*v*), was found for N_2/O_2 flow ratio of 1-1. Extinction coefficients remained constant and almost equal to zero at different wavelength. The refractive index of N-doped TiO_2 films at 623 nm decreased from 2.25 to 2.11, as N_2/O_2 flow ratio was increased from 0.3 to 2.5. The data shows the normal dispersion behavior for deposited films and is consistent with other works [6]. The XRD data indicates that the films have amorphous structure, instead of mixed anatase-rutile structure observed in the films deposited in oxygen-nitrogen atmosphere without substrate bias [5] and with bias of –100 V.

Conclusion. In the present paper we have investigated the optical and structural properties of N-doped films, deposited onto biased substrate with different N_2/O_2 flow ratio. The structure of films was found to be amorphous, which may be attributed to the high bias. The increase of N_2/O_2 flow ratio led to decrease of film refractive index. The maximum thickness was observed for film deposited with N_2/O_2 flow ratio of 0.7.

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MAGNETRON SPUTTERED LSC THIN FILMS FOR SOLID OXIDE FUEL CELL APPLICATIONS[1]

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Mixed ionic-electronic conductors, such as $La_{0.6}Sr_{0.4}CoO_{3-\delta}$ (LSC), show high electrocatalytic activity towards oxygen reduction and are used as a cathode of solid oxide fuel cells (SOFC) [1]. Conventional SOFC cathodes are fabricated by screen-printing teqnique followed by high-temperature sintering. Yoon et al. [2] showed that LSC thin film cathode fabricated by pulsed laser deposition (PLD) technique improves SOFC performance significantly. In this work, LSC layers with the thickness of several hundred nanometers were deposited on Si wafers and SOFC electrolyte membranes by magnetron sputtering of LSC target. The effect of O_2 flow rate and post-annealing temperature on a crystalline structure of the LSC films was studied. Post-annealing was performed in air at temperatures in the range of 400–1000 °C. The phase composition, crystalline structure and surface morphology of the films were determined using X-ray diffraction, scanning electron and atomic force microscopy, respectively. To study the electrochemical characteristics of the deposited films anode-supported SOFCs with bi-layered thin-film yttria-stabilized zirconia (YSZ) / gadolinium-doped ceria (GDC) electrolyte have been fabricated. LSC films served as interlayer between the YSZ/GDC electrolyte and the LSC cathode contact layer. The performance of fabricated single cells was compared to that achieved the cells without the thin-film LSC interlayer. It was shown that the LSC interlayer could be used as a transition layer that improves adhesion and relieves both thermal stress and lattice strain between the cathode and the electrolyte. Our results demonstrate that magnetron sputtering provides a low-temperature synthesis route for realizing thin LSC films for intermediate- or low-temperature solid oxide fuel cells.

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STRUCTURAL FEATURES, PHASE COMPOSITION AND PROPERTIES OF N-CONTAINING TITANIUM DIOXIDE THIN FILMS DEPOSITED BY MAGNETRON SPUTTERING[1]

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The biocompatibility of the surfaces of medical implants is an urgent problem of medical materials science. This problem is especially critical in vascular surgery. Recently, the application of ultra-thin coatings on medical implants becomes a trend in implantology. Plasma technologies make it possible to obtain coatings with a unique structure and properties, which can be controlled by their atomic structure and chemical composition. Combined nitrogen-containing titania (Ti-N-O) films are promising coatings for vascular stents. Formed by high-tech process, such films combine the properties of two components: titanium oxide and nitric oxide (NO). Over the last two decades, NO has emerged as one of the most diverse and important ubiquitous biological mediators. It plays a functional role in processes ranging from neurological function and vascular tonicity to pathogen eradication [1]. Consequently, the search for donors of NO is very urgent.

The structural features of Ti-N-O films grown in magnetron discharge plasma suggest that they can serve as depots of nitrogen oxides directly in the pathology region. In this case, it is possible to predict the following mechanism of interaction of the Ti-N-O coating with the biological system: *i*) titanium oxide increase the corrosion resistance of implant; *ii*) titanium oxide inhibits the electron transfer from fibrinogen to the surface, reducing platelet aggregation and fibrinogen coagulation; *iii*) nitric oxide (NO) performs the necessary biological functions, promotes endothelialization. The structure and properties of thin-film coatings grown by magnetron sputtering system (MDS) depend on deposition regimes, in particular, on the composition of the working gas, power, and the magnitude of the electrical displacement on the substrate. The present work aims at studying the influence of the above factors on the structure, phase and chemical composition of the films.

In this study, titanium target was sputtered in a mixed argon-oxygen-nitrogen atmosphere with different N_2/O_2 flow ratio. The data X-ray diffraction (a) illustrate the changing of the crystal structure of the films as a function of the N contents. All films consist of the anatase and rutile mixture with tetragonal structure. Regardless of the grows of nitrogen content in working gas, there is no presence of TiN phases in deposited films. This leads to transition from anatase to rutile crystal phase. The calculation of the crystal size showed the reduction of crystallites from 15 to 8 nm with the N increase. The amount of anatase decreases from 60% to 22% while as the amount of rutile increase from 38% to 68% with the nitrogen increasing. Thereby, the nitrogen increase in gas mixture led to the structural changes of the films. Complex analysis of the films structure and phase composition demonstrates its significant changes due to nitrogen content. The grain size decreases and reaches units of nanometer with increase of nitrogen concentration. This allows supposing the nitrogen intergranular position in form of nitric oxide two-dimensional layer located at the TiO_2 grains boundary. That reduces the surface of TiO_2 grains, grains boundary migration, and limits grains coarsening. Epitaxial growth of individual crystallites is interrupted by sequence of renucleation. The data of high-resolution XPS spectra demonstrate appearance of chemisorbed nitrogen and nitric oxide that are located at the TiO_2 grain boundaries. Increase of nitrogen in reactive gas leads to predominant formation of rutile phase and inhibits the growth of TiO_2 anatase phase in deposited films. In addition, the growth of nitrogen ratio in plasma leads to reduction of film's structural elements up to 8 nm. The modified Structure Zone Model is revealed, in which the boundaries between zones are determined by content of nitrogen in the plasma. The nitrogen occupy the intergranular positions in the TiO_2 crystallite lattice in form of nitric oxide as well [2]. That limits grains coarsening. The anatase-rutile phase ratio in the film can be controlled by the reactive magnetron sputtering process through nitrogen addition in the working gas. It can allow producing the Ti-N-O films as depot of nitric oxide and promote their applications in biomedicine.

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FORMATION OF SUPERHARD AND WEAR-RESISTANT NBC BASED COATINGS ON HARD ALLOY TOOLS BY CATHODIC ARC DEPOSITION

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In the proposed study, a prospective approach is considered for increasing the wear resistance of a carbide tool of wide application consisting of the formation of a layered architecture of high-hard NbC based coatings by the ion-plasma method. The change in the phase-structural state, elemental composition, and microstructure in the depth of the cross-sectional pattern of tool samples with synthesized coatings, their hardness, adhesion, and wear depending on their vacuum-arc deposition regimes are studied. The peculiarity of the variation of the ion processing and deposition regimes consists in changing of ion treatment time by niobium ions and bias voltage on the hard alloy tool.

It has been established that the effect of high-density niobium ion fluxes and Nb plasma flows in a methane medium allows synthesis of layered $(Nb,W)C_{0.7}$, NbC coating with a hardness of 40-70 GPa on a hard alloy tool. Synthesis of an adhesively strong sublayer made of $(Nb,W)C_{0.7}$ with a hard alloy occurs after niobium ion bombardment and heating of the surface of the hard alloy tool at temperatures of 1200-1400 ° C. With the help of sliding X-ray diffraction by the Halder-Wagner method it is determined that the macro-stresses in these coatings do not exceed 1 GPa. The coatings peeling on the tool was determined by the method of sclerometry. It has been shown that for single-layer carbide coatings, the critical peel load does not exceed 40 N, the macrostresses has a value of 4-5 GPa. Layered $(Nb,W)C_{0.7}$, NbC coatings have a critical peel load of at least 140 N. These layered coatings on hard alloy cutting tools allow to reduce the specific volumetric wear during dry friction of diamond on the tool surface up to 10 times in comparison with the tool without coating.

COMPARATIVE STUDY OF THE RESISTANCE OF THE PROTECTIVE NANOCOMPOSITE AL-SI-N AND IN-SN-O COATINGS TO THE SHOCK IMPACT OF SOLID MICROPARTICLES

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Currently, there is an active exploration of space, which requires the development of spacecraft capable of operating in extreme operation conditions of outer space, which in turn demands the development of new functional materials and technologies for their production. The spacecraft surface erosion and its local destruction can be caused by the collisions with micrometeoroids. Different optical elements of space vehicles (i.e. windows, lenses, mirrors) suffer the most from impacts of micrometeoroids.

The most promising method to prolong the durability and improve the efficiency of glass elements is to protect them with coating produced by magnetron sputtering technique. Magnetron sputtering method allows to obtain dense and uniform coatings with high adhesion.

As protection for optical systems can be used transparent and stable under specific conditions coatings, for example one based on Al-Si-N and In-Sn-O system. There are several works dedicated to obtaining using magnetron sputtering method and studying Al-Si-N [1] and In-Sn-O [2] coatings. According to them Al-Si-N coatings possess a high degree of transparency within the visible light spectrum and a high level of mechanical properties, especially microhardness and elastic recovery. In-Sn-O coatings also have high transparency within the visible region, however, in contradistinction to Al-Si-N coatings, they have lower mechanical properties.

This paper presents the results of investigations of the structural and phase conditions, mechanical, optical and protective properties of Al-Si-N and In-Sn-O coatings obtained by magnetron sputtering on K-208 glass substrates. Structural and phase state coatings were determined by X-ray diffraction and transmission electron microscopy. It was established that Al-Si-N coatings have highly disperse structure and contain hexagonal AlN, α- and β-Si_3N_4 phases. In-Sn-O based coatings are characterized by columnar microstructure and consist of cubic In_2O_3 phase. Nanoindentation showed high values of hardness for the Al-Si-N coatings (31 GPa) and much less (5,7 GPa) for In-Sn-O coatings. Al-Si-N-based coatings are characterized by more transparency (≈ 80 %) within the visible light spectrum compared with In-Sn-O coatings (≈ 65 %) according to spectrophotometry measurements. The protective properties of the coatings were evaluated by the impact of high-speed (5-8 km/s) flows of iron microparticles. It was shown that deposition of protective coatings contributes to significant decrease in surface density of craters. On the surface of samples with coatings having different mechanical properties, due to the impact of the flow of microparticles, craters are formed, the sizes of which are in different ranges.

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PROPERTIES OF THIN TANTALUM FILMS DEPOSITED IN DIFFERENT MODES OF MAGNETRON SPUTTERING[1]

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Ta thin films were grown on Si and stainless steel substrates using direct current (DC) magnetron sputtering and high power impulse magnetron sputtering (HIPIMS). Samples of tantalum thin films were also deposited using a hybrid process combining HiPIMS and DC magnetron sputtering techniques. The structure of the as deposited films was investigated using X-ray diffraction (Fig. 1), scanning electron and atomic force microscopy, while a four-point probe setup was used to measure the resistivity. Increased degree of ionization of the sputtered material in HIPIMS mode allowed for better control of the energy and directionality of the sputtered species, and consequently for improved properties of the deposited films.

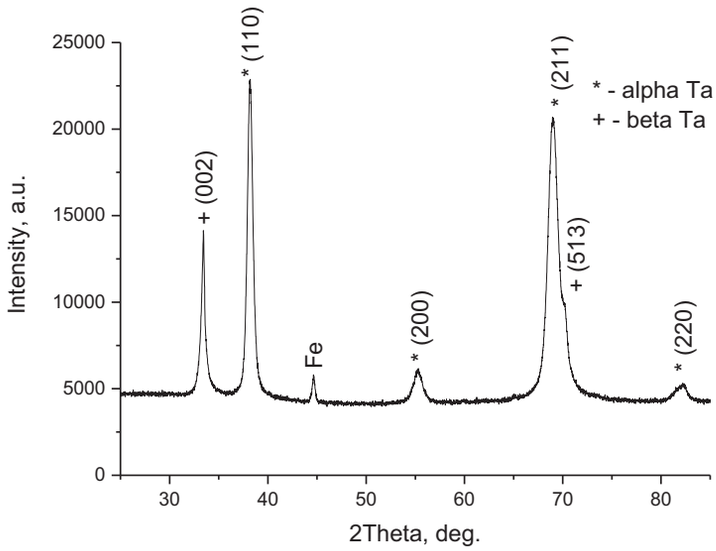

Fig. 1. Typical X-ray diffraction pattern of Ta film with α and β phases on stainless steel substrate.

It was shown that by using HIPIMS mode of sputtering, control of the alpha and beta Ta phase formation could be obtained. The formation of low-resistivity bcc-phase (α-phase) could be understood in light of the high ion flux from the HIPIMS discharge.

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INVESTIGATION OF PARAMETERS OF PLASMA GENERATED BY HIGH POWER IMPULSE MAGNETRON SPUTTERING (HIPIMS) OF GRAPHITE

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High-power impulse magnetron sputtering (HiPIMS) systems operate at pulsed power densities on the cathode from several hundred to several thousands of W/cm^2. As the amplitude of the discharge current increases to several hundred amperes, the density of the generated plasma and the degree of ionization of the sputtered material increase to 10^{13} cm^{-3} [1] and 70–90% [2], respectively.

HiPIMS of graphite is used for deposition of hard and wear-resistant amorphous carbon films [3], because tetragonal diamond-like carbon (DLC) bonds produced under subplantation mechanism [4] at negative substrate bias voltage more intensive in the case of high-density plasma. In [5] it is shown that DLC films are more hard (37 GPa) in the case of short (7 μs) magnetron discharge pulses comparing to long (50 μs) ones (17 GPa) in HiPIMS of graphite. Authors of the present paper also confirmed deposition of harder amorphous carbon films (18 GPa) in the case of short-pulsed (50 μs) HiPIMS of graphite compared to long-pulsed (100 μs) one (13 GPa) [6]. Likely, it is connected with denser plasma (10^{12} cm^{-3}) and more effective carbon subplantation in the case of short discharge pulses compared to long ones. To confirm this, it is necessary to investigate dependencies of plasma parameters (ion concentration, electron temperature, floating potential, and plasma potential) generated by HiPIMS of graphite on discharge pulse width.

The magnetron was mounted in stainless-steel vacuum chamber with dimensions of 600 × 600 × 600 mm. The chamber walls served as the anode of the magnetron discharge. The main magnetron units were the sputtered cathode (disk with a diameter of 90 mm and thickness of 5 mm, pressed from of pyrolytic conductive graphite granules) and a system of permanent NdFeB magnets. The magnetron was connected to the HiPIMS power supply with the following parameters: voltage – up to 1 kV, current – up to 1 kA, pulse frequency and width – 0.1 - 15 kHz and 3 – 250 μs. We used voltage pulses with the magnitude of 595-825 V, width of 15-100 μs and frequency of 2 kHz. The working gas was argon, and the pressure in the chamber was maintained at a level of 0.14 Pa. Langmuir probe was made of 0.5-mm-diameter and 5-mm-length nichrome wire and was situated at 10 cm away from the graphite target surface, where NdFeB magnetic field influence can be neglected. It was shown that electron temperature increased from 5.55 to 9.54 eV and plasma density from $4.5\cdot10^{11}$ to $2\cdot10^{12}$ cm^{-3} while pulse width decreased from 100 to 15 μs. It is coincides with results in [7] and can be explained by growth of plasma disequilibrium since pulse current and power density increased to 250 A and 3 kW/cm^2, respectively.

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PROCESS STABILITY OF REACTIVE MAGNETRON SPUTTERING OF Ce/Gd TARGET [1]

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One of the main problems of reactive magnetron sputtering is to stabilize the sputtering process in the transition mode between the 'metal' and 'compound' sputtering mode, because of hysteresis. Stoichiometric coatings with high deposition rates can be deposited in the transition mode. [1,2].

Most commonly used methods for stabilization of reactive magnetron sputtering process in the transition mode are based on partial pressure control by adjusting reactive gas flow rate. They are relatively expensive and have a low response rate. For many reactive sputtering processes, the time taken to obtain a feedback signal and process it should not exceed several tens of milliseconds. Long delays lead to oscillations between high and low partial pressures. Thus, high-speed and cost-effective methods to stabilize reactive magnetron sputtering process in the transition mode are needed.

In this study, a cost-effective approach to stabilize reactive magnetron sputtering process in the transition mode was proposed. It was based on the use of pulse parameters of the power supply as feedback and control signals. It was shown that the changes in the state of the target surface conditions causes almost instantaneous changes in the pulse parameters, such as amplitude and shape of the current and voltage pulses. A converter that transfers electrical energy to the magnetron sputtering system (MSS) acts as a regulator. It allows to adjust the discharge power of the MSS by regulating the pulse frequency in order to control sputtering rate of the target surface. Thus, compensating the changes in the reactive gas partial pressure.

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SYNTHESIS OF MULTILAYERED COATINGS OF THE METAL/CERAMICS SYSTEM BY VACUUM ARC METHOD WITH PLASMA ASSISTANCE

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Modern methods of hardening and modifying the surface of materials and products are increasingly used in advanced industries. The most promising way to harden their surface is to apply nitride wearresistant coatings of various compositions, for example, TiN, TiCuN, TiAlN, etc. [1, 2]. Due to a wide range of properties of obtained coatings, it becomes possible to use the parts and products from cheaper materials in different production areas. Details with applied coatings have a high (2-3 times) strength, a lower (to 0.1) coefficient of friction, 3-5 times increase in wear resistance and other properties in comparison with the characteristics of the initial part without coating. Particularly worth noting is the prospects for the formation on the surface of materials and products of multilayered functional coatings, which have a number of wellknown advantages over monolayer coatings. When they are created, it becomes possible for a smooth transition through the hardness and the expansion coefficient from the bulk of a part or tool to its surface through several layers with gradient properties.

In this paper, the possibility of obtaining multilayered metal/ceramics coatings by the original method is presented. The originality of the method consisted in the deposition of multilayered metal/ceramics coatings with relatively sharp boundaries. For this purpose, the deposition was carried out at a constant working pressure and the ratio of arc current of gas discharge source and arc current of the evaporator. A transition from the metallic to the nitride layer was carried out by changing the plasma parameters of a non-selfsustained arc discharge with a combined hot and hollow cathode (increasing the arc current of a gasdischarge plasma source, and hence the increase of fraction of nitrogen ions). That is low-inertia method, therefore it is possible to increase the repeatability of the thickness and composition of the layers. It also allows to deposit the coatings with denser packaging and lower porosity at a lower operating gas pressure.

The result of the present work is multilayered (30 layers) coatings of a Ti/TiN system with a thickness of each layer of ~ 100 nm deposited by an original low-inertia method in low-pressure arc discharge plasma. The maximum microhardness of the multilayered coating was about 23 GPa.

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THERMO-BARRIER NANOSTRUCTURE MICROPLASMA COATINGS OF ZRO_2

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Development of thermo-barrier nanostructure coatings, which are capable to maintain thousand-degree temperature drops without destruction, for space technologies is an urgent problem of present days. Formation of such coatings is possible due to combination of well-known methods.

Researches in the field of synthesis of functional ceramic coatings have developed a technique for formation of a unique thermo-barrier nanoporous layered oxide ceramic coating on a copper substrate.

Titan is sprayed on a copper substrate (PVD method), and then zirconium is sprayed (magnetron deposition) step by step. Then the sample is exposed to microplasma influence in an electrolyte solution (sodium metasilicate - 70 g/l and alkali – 6 g/l. Process time varied from 6 to 10 minutes As a result, zirconium transforms into zirconium dioxide, and the porous ceramic coating characterized by high adhesion and containing mainly of zirconium dioxide and compounds of elements of material substrates (zirconium) and elements of electrolyte is formed on the surface.

The phase composition of the coating was studied with the scanning raster microscope. The coating composition contains monoclinic and tetragonal zirconium oxide. Silicon oxide is contained in the x-ray amorphous state in the coating.

The samples were analyzed for thermal stability. The obtained samples were heated to 1000 degrees, then quickly cooled to room temperature The coating becomes dark after the first cycles (regardless of the time of the microplasma process). The uniformity of the coating remains until the 40th cycle (treatment time – 10 min). Small parts at the coating after the 44th cycle start to peel; there are copper sites after the 58th cycle. Further there are no visible changes until the 63rd cycle; at the 70th cycle the number of copper sites increases. As a result, more than 50% of the coating preserves at the 95th cycle.

The sample 2 showed lower results (microplasma treatment time - 6 min). The uniformity of the coating remains until the 10th cycle. Copper appears on the 13th cycle. More than 50% of the coating is destroyed by the 70th cycle.

Thus, the combination of three processing methods of surface treatment (vacuum plasma spraying, magnetron deposition, and microplasma oxidation) has allowed creation of the new unique oxide ceramic coating with the raised thermal stability owing to formation of a nanoporous layer structure. Similar coatings can be used on an interior sheeting of a nozzle in explosive motors for the space industry.

APPLICATION OF A FILM DEPOSITION REGIME FROM THREE MAGNETRONS ON A CYLINDRICAL SURFACE TO CREATE A MULTILAYER COATING

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The essence of the method of magnetron coating is the deposition of target atoms on the substrate, which is atomized in the plasma of a magnetron discharge. This method allows to film films with an accuracy of tens of nanometers on a wide class of substrates and is therefore widely used in various fields of science and technology. So, for example, the effectiveness of modifying the surface of metallic materials by mixing the near-surface layer with an ion beam directly depends on the quality and uniformity of the previously deposited films of the alloying elements. The method of magnetron sputtering allows for multicomponent doping of substrates, since the deposition of films is not a thermally activated process.

The paper presents the results of experiments performed on the ILUR-03 installation, which is a system for complex ion beam treatment of long cylindrical samples. The work of the magnetron system of the installation for ion beam treatment of long-length cylindrical products ILUR-03 is analyzed. The ranges of operating parameters that ensure the effective deposition of films of alloying elements from the Al, Fe, Mo, Mg, Cr series for subsequent alloying of the surface of the samples in the ionic mixing regime are determined.

Modified layers with a content of alloying elements of Fe, Mo, Y, Cr, Ni, Al, Mg up to (1-5) at% were obtained under the influence of a radial beam of Ar^+ ions in the polishing and ionic mixing mode of multilayer films near the surface of cylindrical samples of the E110 alloy maximum penetration depth up to 1-2 microns.

INFLUENCE OF ULTRA SHORT WAVES ON NANORELIEF AND PRECIPITATED NANOPARTICLES METALS

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The modification of the metal surface due to the compacting of the deposited nanoparticles by picosecond compression pulses is considered. The impact of a shock wave is often used to compact the nanopowder. In the case of nanoscale thicknesses of plates and metal particles, it is possible to use molecular-dynamic modeling. The effect on the front surface of the test sample is carried out by successive pressure pulses. The interaction of the shock wave with the deposited nanoparticles on the substrate leads to plastic deformation in the surface layer of the metal. The presence of precipitated nanoparticles on the rear surface increases the threshold value of the amplitude of the shock wave, which causes the rear splitting. As a result of the action of successive pulses, nanoparticles are compacted into a nanocrystalline coating.

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INFLUENCE OF MICRO-ARC OXIDATION TIME AND APPLIED VOLTAGE ON FORMATION OF STRONTIUM- AND SILICON-INCORPORATED BIOCOATINGS [1]

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The influence of the process time and pulsed voltage on the formation of the strontium- and silicon-incorporated calcium phosphate (Sr-Si-CaP) biocoatings during the deposition by the micro-arc oxidation (MAO) was investigated. The coatings were deposited on the Ti substrates in the anodic potentiostatic mode under the following parameters: the pulse time of 100 μs, the frequency of 50 Hz, the process time up to 10 min, and the pulsed voltage from 200 to 370 V. The electrolyte consisted of the phosphoric acid, the calcium carbonate, and nanopowder of Si- and Si-substituted HA ($Ca_{9.5}Sr_{0.5}(PO_4)_{5.5}(SiO_4)_{0.5}(OH)_2$).

It is seen in Figure 1, during the MAO process under the applied voltage of 200 V the current density decreases from 0.27 to 0.02 A/cm^2 due to the growth on the Ti substrate of the dielectric CaP coating up to 50 μm. It should be noted, the MAO time does not influence on the coating characteristics such as roughness and apparent density, morphology, phase composition and microstructure.

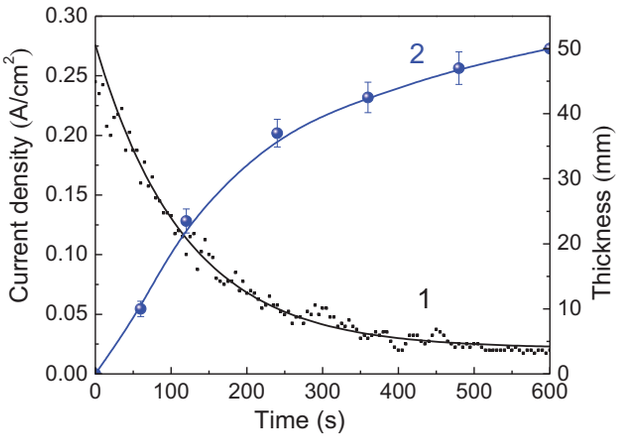

Fig. 1. Graphs of the current density (curve 1) and the coating thickness (curve 2) against the MAO processing time (at the applied voltage of 200 V).

Whereas, the increase in the applied voltage leads to the significant linear increase of the coating thickness and roughness, and decrease of the coating apparent density and adhesion strength. All the formed Sr-Si-CaP coatings have a complex porous structure including the inner thin oxide layer (TiO_2 anatase) at the interface coating/substrate and the main CaP layer containing numerous branched pores through the thickness and structural elements (spheres) with open pores through the coating surface. With increasing voltage, the structural elements grow in sizes and are destructed partially, and new plate-like crystals grow inside the destroyed hemispheres. In this case, the coating structure transforms from X-ray amorphous state to the amorphous-crystalline state with increasing degree of the crystallinity up to 60 vol.%. The amorphous-crystalline coatings includes the following phases: $CaHPO_4$, β–$Ca_2P_2O_7$, and TiO_2 (anatase). The structured-phase transitions and the changing in the coating microstructure, morphology and composition with increasing applied voltage due to the increase in the intensity of the micro-arc pulsed discharges and, as consequently, the increase of the temperature inside the micro arc channels.

Thus, the work results demonstrated that the applied voltage has the greatest influence on the coating morphology, microstructure and composition, and physico-chemical properties during the coating deposition by the MAO method.

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THE EFFECT OF FIBERS AVERAGE DIAMETER PLLA SCAFFOLD ON THE DEPTH OF PENETRATION TITANIUM OBTAINED BY THE DC MAGNETRON SPUTTERING[1]

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One promising synthetic polymer suitable for use in tissue engineering is poly-(l-lactic) acid (PLLA). PLLA is a bioresorbable polymer with a unique combination of physical and chemical properties. Currently, it is used for producing resorbable bone implants and stents, artificial vascular grafts [2] and as a stent coating [3]. Main disadvantages of PLLA-based medical devices are low degradation rate and high hydrophobicity [4]. Since electrospinning allows the morphology of the PLLA scaffolds to be controlled in a wide range DC the magnetron sputtering composition and properties of the deposited coatings, their comprehensive use for tissue engineering applications is promising. However in this moment there is no information in literature on the joint use of the methods of electrospinning and plasma modification for morphology changing and biocompatibility of polymer PLLA scaffolds.

The samples were produced using a NANON-01A electro-spinning setup (MECC Co. Ltd., Japan) from a 5, 9 and 14 % (mass) spinning solution of PLLA PL-38 (Purac, Amsterdam, Netherlands) in trichloromethane ($CHCl_3$) (Ekros, Russia). The distance between the needle and collector was 110 mm. The flow rate of the spinning solution was 4 mL/h, and the voltage was 22 kV. Before modification of the electrospun scaffolds, they were exposed to vacuum at a pressure of 10^{-2} Pa and 100˚C for 10 h to remove the residual solvent.

The coating deposition was performed using the magnetron plasma discharge occurring during sputtering of the metal target made of chemically pure (99.99%) titanium under a nitrogen atmosphere. The area of the sputtered target was 224 cm^2. For coating deposition, the universal magnetron sputtering system was used, which has been described previously [5]. To reduce the destructive influence of plasma on PLLA electrospun scaffolds, the modification was conducted in a cyclic mode: 1 min of plasma treatment was followed by 3 min of magnetron cooling. The following technological parameters were used for coating deposition: the distance between the target and scaffold was 33 mm, preliminary pressure of 3×10^{-3} Pa, operating pressure of 0.4 Pa and nitrogen (N_2) as the working gas. The samples were treated at 1, 2, 4, 6 and 8 min at a constant discharge power of 88 W. The temperature in the sample holder region was controlled by a copper thermocouple (TCM9623; Etalon, Russia) and did not exceed 43˚C for the 8-mincoating deposition cycle with the selected processing parameters. Thus three groups of samples were formed – PLLA 5%, PLLA 9% and PLLA 14%.

In this study we demonstrated the possibility of modification of the surface PLLA scaffold by reactive magnetron sputtering of a titanium target under a nitrogen atmosphere. The influence of exposure time and average fiber diameter on the morphological and physico-chemical properties of these materials was studied. In the course of the research it was found that the DC magnetron modification method allows the formation of a coating whose penetration depth into the volume of the material depends on the mean diameter of the PLLA graft fibers. The wettability studies of the obtained materials showed that the value of the contact angle also depends on the average diameter and concentration of the deposited coating.

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DEPOSITION OF CALCIUM PHOSPHATE COATINGS USING RADIO FREQUENCY MAGNETRON SPUTTERING OF SUBSTITUTED B-TRICALCIUMPHOSPHATE TARGETS[1]

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The problem of low biological compatibility of titanium implants is usually solved by deposition of calcium phosphate (CaP) coatings, which are not rejected by the body. One of the most frequently used deposition technologies is radio frequency magnetron sputtering (RFMS). The main advantages of this method are high adhesion strength, the ability to vary the chemical composition of coatings by changing parameters of the sputtering and chemical composition of targets, wide range of sputtered materials. Its main disadvantages are poor effectiveness and low deposition rate. According to Ozeki and coauthors, type of CaP material, used for coatings deposition on implants, affects the deposition rate [1]. β-tricalciumphosphate (β-TCP) was chosen for our study because of its high deposition rate and good biological properties. It should be noted that the occurrence of bone defects is often associated with the presence of bones diseases, such as osteoporosis. It was proved that Mg and Sr substitutions affect regeneration process positively [2,3].

The influence of Mg and/or Sr substitutions in the structure of β-TCP targets, deposited by the RFMS method, on the deposition rate and coatings properties was studied.

Four different types of targets based on synthesized in Riga Technical University (Prof. Janis Locs) β-TCP were sputtered: pure β-TCP; Mg-substituted β-TCP (β-TCP+Mg, Mg content was 1,53±0,01 wt.%); Sr-substituted β-TCP (β-TCP+Sr; Sr content was 3,39±0,09 wt.%); Mg- and Sr-substituted β-TCP (β-TCP+Mg+Sr, Mg and Sr contents were 1,18±0,22 wt.% and 3,68±0,06 wt.% respectively). Coatings deposition was carried out using the custom-made magnetron device developed in TPU. Sputtering parameters: preliminary pressure in the chamber was 10^{-3} Pa, working pressure (Ar) was 0.5 Pa, distance between the sputtered target and the substrate was 40 mm, discharge power was 1.5 kW, coating formation time was 21 hours, and reflected power was 400 W. The thickness of the formed coatings was determined using the contact profilometry (Talysurf-5, Taylor&Hobson, UK). The elemental composition of the CaP coatings was evaluated by X-ray fluorescence (XRF) analysis (XRF 1800, Shimadzu, Japan). The study of coatings roughness was performed by atomic force microscopy (AFM) (Solver-HV, NT-MDT, Russia). The mechanical properties were tested using a nanohardness tester (NanoScan-4D, TISNCM, Russia). The wettability and the surface free energy (SFE) of coatings were measured using the "sitting drop" method (Easy Drop, Krüss Optronic, Germany). The release of the elements in normal saline was investigated using an atomic-emission spectrometer with inductively coupled plasma (iCAP-6300 Duo, Thermo Fisher Scientific, USA).

It was determined that Sr substitutions in the sputtered targets structures (β-TCP+Mg+Sr, β-TCP+Sr) increase deposition rates of coatings by 1.5 times in comparison with pure β-TCP. According to the XRF-analysis, there were significant differences in the elemental compositions of coatings and targets. All coatings had a lower Ca/P ratio than targets. It was found that light elements were sputtered more intensively than heavy ones. Using the AFM it was determined that coatings deposited by sputtering of substituted targets demonstrate lower roughness than the β-TCP group. There were no significant differences in the SFE. It can be concluded that the presence of Mg dopes in CaP coatings decreases its hardness. Coatings containing Mg dopes demonstrate lower Ca and P release in normal saline than β-TCP. Sr dopes, on the contrary, increase Ca release.

Thus, it has been proved that it is possible to increase the deposition rate of coatings using ionic substitutions and to overcome the main disadvantage of the RFMS method. Varying the type of substitution elemental composition of coatings, its roughness, hardness, and solubility can be changed.

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EVALUATION OF THE TEMPERATURE OF MELTING OF MULTI-ELEMENT COATINGS[1]

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Already in the middle of the last century it became clear that one should not go along the path of creating new heat-resistant alloys, but create coatings that lead to an increase in heat resistance and heat resistance. At present, there are not even high-quality models showing in what cases an increase in heat resistance can be expected. Therefore, the search for heat-resistant coatings is still conducted intuitively and by trial and error. In this work we attempt to predict such coatings that would show increased heat resistance.

The dependence of the microhardness of the deposited coating on its thickness is described by the formula [1]:

$$\mu = \mu_0 \cdot \left(1 - \frac{d}{h}\right), \quad (1)$$

where μ is the microhardness of the deposited coating; μ_0 is a massive sample; h is the thickness of the coating. The parameter d is related to the surface tension σ by the formula:

$$d = \frac{2\sigma\upsilon}{RT}, \quad (2)$$

where σ is the surface tension of a massive sample; υ is the volume of one mole; R is the gas constant; T is the temperature.

In the coordinates μ ~ 1 / h (1 / h is the inverse thickness of the deposited coating), a straight line is obtained, the tangent of the slope angle which determines d, and the surface tension of the deposited coating is calculated from formula (2). Table 1 gives the values of σ for the coatings studied by us.

Table 1 - Surface tension of multielement coatings obtained in an argon medium

Coating	σ, J/m^2	Coating	σ, J/m^2
12Cr18Ni10Ti+Zr	0,970	12Cr18Ni10Ti+Zn-Al	1,098
12Cr18Ni10Ti+Zn-Cu-Al	1,093	12Cr18Ni10Ti+Al	1,144
12Cr18Ni10Ti+Fe-Al	1,292	12Cr18Ni10Ti+Cu	1,445

The melting temperature of the coating can be estimated from the formula [2]:

$$T_m = 1{,}4 \cdot 10^3 \cdot \sigma \text{ (K)}. \quad (3)$$

The corresponding estimates are given in Table 2.

Table 2 - Melting temperature of multielement coatings obtained in argon

Coating	T_m, K	Coating	T_m, K
12Cr18Ni10Ti+Zr	1358	12Cr18Ni10Ti+Zn-Al	1537
12Cr18Ni10Ti+Zn-Cu-Al	1530	12Cr18Ni10Ti+Al	1602
12Cr18Ni10Ti+Fe-Al	1809	12Cr18Ni10Ti+Cu	2023

The melting temperature of steels depends on their chemical composition, but lies within the limits (1450-1520) K. As can be seen from Table 2, the coatings 12X18H10T + Al, 12X18H10T + Fe-Al and 12X18N10T + Cu, obtained in argon medium, significantly exceed the melting point all have become.

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SURFACE ENERGY OF PLASMA COATINGS

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The experimental determination of the surface energy (surface tension) of solids is complicated by the fact that their molecules (atoms) are unable to move freely. An exception is the plastic flow of metals at temperatures close to the melting point, when the mobility of atoms on the surface becomes noticeable. In the papers [1. 2] we proposed a method for determining the surface tension of plasma coatings. The method is based on the dimensional dependence of some physical property of these coatings. Table 1, by way of example, shows the surface tension of nitrides of some refractory metals.

Table 1 - Surface tension and properties of nitride coatings

Nitride	Temperature melting coatings, ^{0}C	Micro-hardness coatings, GPa	Electrical Conductivity coating, μOhm^{-1} m^{-1}	Surface tension of coating, J/m^2
TiN	2945	20,0	40	2.062
ZrN	2955	16,0	18	2.069
HfN	3330	22,0	32	2.331
NbN	2320	14,0	78	1.624
TaN	3360	17,5	180	2.352

That gives the knowledge of the surface energy of the coating? Let us consider this in more detail.

Within the framework of the molecular theory, the work of frictional forces is:

$$A = \int_L FdL \approx \sigma \cdot L, \quad (1)$$

where σ is the surface tension, L is the length of the traversed path.

The greater the surface tension, the greater the frictional force.

Work on the destruction of a solid is proportional to the newly formed surface S:

$$W = \sigma \cdot S. \quad (2)$$

The melting point of the coating T_m is:

$$T_m = 1.4 \cdot 10^3 \cdot \sigma. \quad (3)$$

The higher the melting point, the higher the heat resistance of the coating:

$$\chi = \tilde{N}_1 \cdot \sigma. \quad (4)$$

and corrosion resistance of the coating:

$$\varsigma = \tilde{N}_2 \cdot \sigma. \quad (5)$$

C_1 and C_2 are constants. Equations (1) - (5) allow us to predict the operational properties of functional coatings.

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PHYSICAL PROPERTIES OF FLUORIDES BARIUM AND CALCIUM NANOPOWDERS PRODUCED BY THE PULSED ELECTRON BEAM EVAPORATION METHOD[1]

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The mesoporous nanocrystal powders BaF_2 and CaF_2c with a specific surface up to 34.8 and 88.7 sq.m/g, respectively, are produced by the pulsed electron beam evaporation method in vacuum [1]. Was investigated influence of thermal annealing of nanoparticles on air in the range of temperature from 200 to 900°C on the size, morphology of particles and change of their magnetic and luminescent properties.

Was revealed the essential stoichiometric impurity (overage of metals), significant growth in a specific surface of nanopowders (NP) BaF_2 and CaF_2 after annealing at the temperature of 200°C.

It is established that the synthesized NP BaF_2 is a paramagnetic while initial material in the bulk state is diamagnetic. After annealing at 900°C appears the small ferromagnetic contribution at NP BaF_2.

It is assumed that transformation of a magnetic state is connected with formation of radiation and structural defects in the course of synthesis by method of pulsed electron evaporation. Feature of received NP is a large amount of structural defects of various types and also targeting by the X-rays radiation of various radiation defects.

Defects transformation depending on hold time is confirmed by essential reduction of paramagnetic susceptibility and ferromagnetic contribution after storage of samples on air within several weeks.

After annealing of NP BaF_2 at the temperature 200°C was revealed increase in intensity of a red band of photoluminescence by 2,1 times after annealing due to increase in number of F-centers of the 1 - type in ~3 times and the 2 - type in ~4.3 times. Was set noticeable (~50 nm) red offset of the intensity maximum pulsed cathode luminescence (PCL) at the synthesized NP and after annealing at the temperature 200°C.

Produced NP CaF_2 showed ferromagnetic behavior. In literature there is no information about the ferromagnetism of CaF_2. Appearance of the ferromagnetic response can be explained with formation of structural and radiation defects (F-centers, etc.).

At the same time annealing on air in case of 200°C led to increase in amplitude of the ferromagnetism almost ten times that confirms the defective nature of the magnetism. More high-temperature annealing (in case of 900°C) led to loss of the ferromagnetism that confirms that it is connected to CaF_2 nanostatus.

The analysis of PCL and magnetization curves of samples CaF_2 allows to draw conclusions about their connection. After annealing at the temperature 900°C there was a sharp reduction of green band intensity with the maximum in case of ~ 500 nm, connected to F-centers, and appearance of a new intensive band in the red area of PCL range in case of wavelength ~ 700 nm.

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PHYSICOCHEMICAL CHARACTERIZATION AND ANTIOXIDANT PROPERTIES OF CERIUM OXIDE NANOPARTICLES[1]

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Research of cerium oxide nanoparticles (CONPs) biological activity show that this compound exhibits antioxidant, antitumor, antimicrobial and antiviral properties, indicating its prospects for use in pharmaceuticals development [1-3].

Cerium dioxide in nanocrystalline state is characterized by a change in the oxygen non-stoichiometry associated with the formation of oxygen vacancies in the crystal lattice due to an increase in the surface of the crystal and, as a result, a change in the Ce3+/Ce4+ valence ratio. The oxygen vacancies cause active participation of CONPs in oxidation-reduction reactions and, as a consequence, the unique biological activity of the compound. The Ce3+/Ce4+ ratio on the surface of nanoparticles largely determines the possibilities of using the material and depends on a variety of factors (particle size and shape, dopants, stabilizers and production method). The purpose of the work is to study the electronic structure of cerium dioxide nanoparticles by spectroscopic methods for determining the Ce3+/Ce4+ valence ratio, and to establish a correlation between the physical and chemical properties of cerium dioxide nanoparticles and their biological activity.

CONPs was obtained by a pulsed electron beam evaporation in the low pressure gas on installation NANOBIM-2 [4]. The method allows us to obtain CONPs of 3-5 nm with a specific surface ~ 190 m2/g [5]. In addition, highly nonequilibrium conditions of synthesis lead to the formation of highly defect structures, which can lead to an increase in the Ce3+/Ce4+ ratio and, as a result, enhance the degree of their biological activity. It should be noted that this ratio can be changed by irradiating nanopowders with ionizing radiation [6].

Methods of X-ray photoelectron spectroscopy, optical and luminescence spectroscopy were used to determine the Ce3+/Ce4+ valence ratio of cerium dioxide nanoparticles. Measurement of XPS spectra was carried out using the ESCALABMKII electronic spectrometer. Luminescent investigations of CONPs were carried out by excitation with a laboratory source of ultraviolet radiation; spectra were recorded with MDR-23 monochromator and FEU-106 photoelectric multiplier. The optical absorption spectra were measured with a 9423UVA1002E Helios Alpha spectrophotometer. An analysis of interaction of nanoparticles suspensions stabilized with sodium citrate with hydrogen peroxide was made to study the enzyme-like activity of CONPs.

The results of spectroscopic studies show the presence of cerium ions in two valence states. Moreover, measuring the XPS spectra under the influence of ion bombardment of inert gases (ion etching) leads to an increase in the Ce3+/Ce4+ ratio. Luminescent and optical measurements also show the presence of cerium ions of different valences. An investigation of the CONPs interaction with hydrogen peroxide shows the active participation of Ce3+ ions in this process, which is confirmed by a change in the optical absorption spectra of nanoparticles suspensions after the oxidizer addition. Probably, such activity causes the observed antioxidant properties of nanoparticles [3].

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PLASMA SYNTHESIS OF Al_2O_3-TiO_2 FROM RELATED NITRATES

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Oxide systems based on Al_2O_3-TiO_2 are actively used for the application of resistant coatings [1], the production of catalysts [2]. The most valuable properties are nanoscale particles obtained by coprecipitation [3], hydrothermal synthesis [4], self-propagating high-temperature synthesis [5], sol-gel technology [6] and other methods.

The report considers the preparation of Al_2O_3-TiO_2 oxide system obtained by the plasma method. The experimental facility consists of the 6 kW air single-phase AC plasma torch and the supply system for liquid precursors. The aqueous solution was prepared from aluminum nitrate ($Al(NO_3)_3 \cdot 9H2O$) and titanium nitrate ($Ti(NO_3)_2$ 2H2O). The concentration of the solution for each substance was 0.1 mol/l. The solution was fed through a nozzle into the plasma torch. This process involved water evaporation, the decomposition of nitrates to the corresponding oxides, and the formation of particles of the Al_2O_3-TiO_2 oxide system. The resulting material was cooled without water condensation and investigated by X-ray diffraction analysis, IR spectroscopy, X-ray fluorescence analysis, differential thermal analysis and scanning electron microscopy. However, nitrogen dioxide formed during operation of the plasma torch insignificantly reacts with the cooled oxides to form nitrates. Later, this problem is planned to be solved using a non-air plasma torch.

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MODIFICATION OF POLYMER TEMPLATE FOR IMPROVING OF STRUCTURE AND PROPERTIES OF THE ELECTROLESS DEPOSITED COPPER NANOTUBES [1]

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Polymeric track-etched membranes (TeMs) with embedded copper nanotubes (CuNTs) have gained much attention past decade due their application in various fields of material science. Copper extensively used because of its physical and chemical properties, thus copper-based nanomaterial's has a long range of applications in fabrication of the electronic nanodevices, as antibacterial agent, sensors and catalysts [1-2].

This study is dedicated to the electroless synthesis and structure elucidation of the CuNTs composites prepared in etched and oxidized PET TeMs template as well as comparative testing of the catalytic activity.

The carboxylic group functionality was enriched by successive oxidation by hydrogen peroxide and UV [3] and its concentration was significantly improved. Usually prior electroless plating of copper, the TeMs is treated during sensitization and activation processes to create catalytically active Pd seeds [3]. The influence of Pd seeds on the plating effectiveness was also investigated and PET TeMs templates after single and double pre-plating treatment were prepared. The effects of template modification on structural properties of electroless deposited copper nanotubes were investigated by scanning electron microscopy (fig.1), X-ray diffraction measurements.

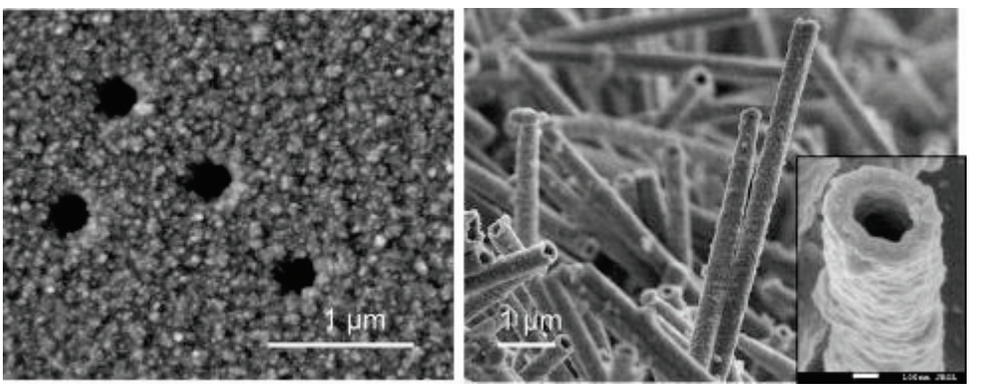

Fig. 1. Surface and cross-sectional view of composite membranes with embedded copper nanotubes

"As-prepared" membrane composites were elucidated for catalytic activity using reduction of 4-nitrophenol in the presence of sodium borohydride as a benchmark reaction. The reusability of tested composites was monitored for 5 consequent cycles and decreasing of catalytic ability after third cycle was observed.

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LARGE SCALE PRODUCTION OF CARBON NANOTUBES AND GRAPHENE USING NITROGEN PLASMA JET SYSTEM: SYNTHESIS, CHARACTERIZATION AND GAS PHASE KINETICS[1]

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The possibility of large scale production of carbon nanotubs and graphene during its synthesis in a plasma jet of nitrogen has been studied. The plasma jet reactor was used on the basis of a powerful dc plasma torch with an expanding anode channel [1-2]. The precursor of carbon in the form of hydrocarons was introduced into the plasma torch simultaneously with a plasma-forming gas. Hydrocarbon decomposes both in the arc discharge region and in the plasma jet to form a vapor-gas mixture, which, as it cools condenses in the form of solid products in the collector. Pressure in the reactor was 77-750 Torr. The current of the arc was maintained constant in the experiment for tens of minutes and was equal to 300-350 A. Power of the plasma torch reached 40 kW. Methane, mixture of propane-butane and acetylene were used as hydrocarbons.. The rate of hydrocarbons and nitrogen varied independently of each other. Synthesized materials are characterized by the method of scanning electron microscopy, X-ray photoelectron spectroscopy and thermogravimetric analysis. The spectra of plasma were measured on three-channel fiber-optic spectrometer AvaSpec 2048 with a spectral resolution of 0.1 ÷ 0.4 nm and a spectral range 200 ÷1100 nm. The temperature of plasma was determined using the method of the relative intensities of the lines.

The synthesized graphene was presented by flakes containing from 2 to 5 layers of graphene. Flakes have a lateral dimension up to 200 nm. The possibility of doping graphene during its synthesis in a plasma jet of nitrogen has been studied. Study of the thermal stability of N-graphene system shows that the nitrogen impurity significantly increases the temperature stability limit compared to pure graphene. Photoelectron spectroscopy has shown that in the synthesized N-graphene, pyridine nitrogen, which is not an electron donor, predominates. The maximum degree of nitrogen doping of graphene was obtained at decomposition of acetylene.

Carbon nanotubes (CNT) synthesized at atmospheric pressure has a diameter of up to 20 nm. The ratio of length to diameter does not exceed 100. At a pressure of 100 Torr, filamentous CNTs are formed assembled into tangles with diameter of 20 to 50 nm. The ratio of length to diameter of these CNTs is more than 1000.

The chemical kinetic model was proposed, which describes the processes initiated by thermal plasma of N_2-CH_4 mixture. The initial composition of plasma was determined by the code for calculation of thermodynamically equilibrium composition. To determine the mixture composition in different temperature ranges in the gap between the reactor nozzle and the material collection point, the own software complex RADICAL [3] and the extended scheme of chemical reactions involving 753 reactions between 120 components. The calculation of the N_2-CH_4 mixture composition during the conversion process of hydrocarbons has been carried out. The results are given for different pressures and exponential temperature change along the flight of the reaction products. Three temperature ranges were identified with radically different mixture composition. The important role of reactions involving particles C and C_2 with N_2 and N has been shown. The presence of molecular nitrogen and methane leads to the rapid formation of CN and C_2 at a high temperature of ~ 10000 K, while the concentration of N and C atoms decreases by several orders of magnitude. High CN concentration and low N atoms concentration are confirmed by the emission spectra obtained in the experiment. This distinguishes these results from those obtained in other studies with mixtures without nitrogen, containing an inert gas, or in works with low temperature conditions where high C and N concentrations were obtained.

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EFFECT OF MULTI-LAYER COATINGS ON SHOCK RESISTANCE OF OPTICAL GLASS

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The impact of the flow of high-speed meteoroids and space debris on the windows of spacecraft illuminators and photovoltaic solar cells leads to a degradation of the optical and mechanical characteristics of these structures and their failure.

The report presents the results of the investigation of the formation of multilayer optically transparent nanocomposite coatings on quartz (KV) and optical glasses (K208), used respectively for the manufacture of a windows of space vehicles and solar batteries, and ways to protect these glasses from the impact of high-speed microparticles.

The protective coating is a complex hierarchical structure formed on a glass substrate by high-energy ion implantation and pulsed magnetron deposition from heterogeneous nitride and oxide layers. The principle of formation of structural-phase states of a multilayer nanocomposite should provide not only high values of adhesion of the coating to the substrate, cohesive strength of the material and its transparency for visible light, but also implies the coordination of the physical-mechanical characteristics of all its functional layers to ensure high effect of scattering of a shock wave as it passes through the coating.

The interlinks of magnetron sputtering regimes of composite targets and high-energy ion doping of the substrate and functional coating layers and interfaces on the protective ability of the nanocomposite coating against the impact of microparticles of classified iron powder, moving with close to the first cosmic velocity, is investigated.

It is shown that the process of formation of craters on glass surface with a multilayer coating under impacts of solid microparticles differs significantly from the analogous process on glass with a single-layer coating. The change in thickness and the period of modulation of the layers in the coating affects the process of crater formation. The intensity of this process depends to a large extent on the structural-phase state of both the individual functional layers of the coating, and on the number of layers, their chemical composition and the type of structure of the coating as a whole. The influence of the substrate material on these processes is also analyzed.

Structural studies were carried out using high-resolution transmission electron microscopy, X-ray diffraction analysis, scanning electron microscopy with X-ray microanalysis of the elemental composition of local microzones along the coating thickness, and other methods of modern physical material science.

SYNTHESIS OF SILICON CARBIDE NANORODS IN THE ATMOSPHERIC DC ARC DISCHARGE PLASMA [1]

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Silicon carbide characterizes a number of important properties for science and technology: high thermal conductivity, superhardness, radiation resistance, resistance to chemically active media [1]. There are many approaches to the production of silicon carbide, one of which is the electric arc discharge method [2]. Traditionally, in a sealed reactor, a DC arc discharge is ignited in the presence of silicon and carbon, which are often included in the discharge circuit electrodes. In the present work, a vacuum-free method for obtaining nanosized silicon carbide rods in a plasma of a DC arc discharge initiated in an open air is realized.

An experimental setup was assembled and a series of experiments were carried out to implement the method at the Tomsk Polytechnic University. The product synthesis was analyzed by X-ray diffractometry and transmission electron microscopy method. The possibility of implementing such a vacuumless method is being actively used to produce carbon nanomaterials [3]. Its implementation is possible due to the generation of the CO atmosphere at burning an arc discharge on graphite electrodes in the air [4]. According to X-ray diffractometry, the product is identified with a cubic modification of silicon carbide, as well as initial reagents in the form of graphite and silicon. According to the data of high-resolution transmission electron microscopy HRTEM (Fig. 1), the product contains nano-sized rods, the core of which consists of cubic SiC, and the shell consists of amorphous SiO_x. The length of the rods is on average up to 1 μm, the cross section is about 30-60 nm. Silicon carbide particles have a typical structure for nanoscale rods, described in the world scientific practice [5]. The measured interplanar distance by the lattice image was 2.52 Å.

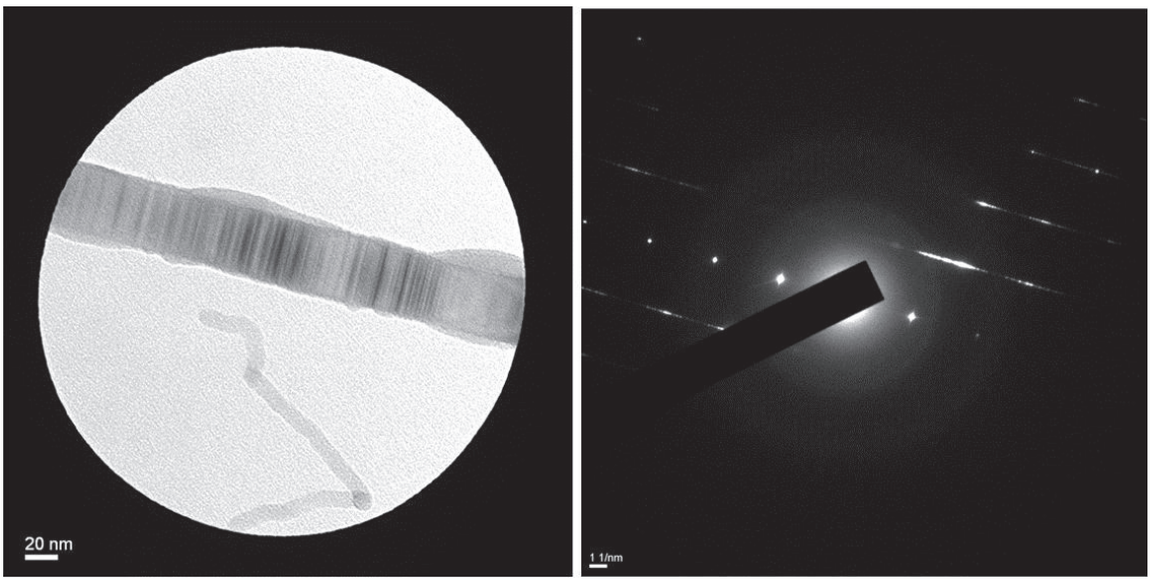

Fig. 1. HRTEM-images of obtained product (Silicon Carbide)

In the present work, the information of the possibility of obtaining nanosized silicon carbide rods in a plasma of a DC arc discharge is presented and initiated in an open air environment. The obtained silicon carbide particles have a typical structure for nanoscale silicon carbide rods.

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STRUCTURE OF ZINC OXYDE NANOCRYSTALS IN TRACK TEMPLATES[1]

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In this report, we present the results on the study of ZnO nanoclusters obtained by electrochemical deposition of zinc in track template *a*-SiO_2/Si – n.

The structure of a-SiO_2/Si-n was prepared by thermal oxidation of a silicon substrate (Si-n type) in an atmosphere of moist oxygen at 900 °C. The thickness of the oxide layer was 700 nm according to ellipsometry. The samples were irradiated at a DC-60 accelerator with 200-MeV xenon ions, up to a fluence of 108 ions / cm^2. Chemical etching of Si/SiO_2 samples in 1% HF, m (Pd) = 0.025 g, 18 ± 1C. Electrochemical deposition of Zn in template SiO_2/Si was carried out in the potentiostatic regime at a voltage range (1.5-1.85) V, and pH = 3.

The surface of the precipitated samples was examined using a scanning electron microscope JSM 7500F. X-ray diffraction analysis of the samples was carried out on a D8 ADVANCE ECO X-ray diffractometer using an X-ray tube with ∝ Cu-anode in the range of angles 2θ 30° - 110° in 0.01° increments. To identify the phases and study the crystal structure, the software BrukerAXSDIFFRAC.EVAv.4.2 and the international ICDD PDF-2 database were used.

In the electrochemical deposition of zinc in the track template *a*-SiO_2/Si – n, nanocrystals of zinc oxide were obtained in three crystalline phases: wurtzite, sphalerite, and rock salt structure. It should be noted that the type of structure depends on the voltage applied to the electrodes. The optimum ECD regime was established, with obtaining the most widespread phase of ZnO, wurtzite.

[1] This work was supported by grant AP05134367 of MES RK

PLASMADYNAMIC SYNTHESIS OF POWDERS OF THE TI-B SYSTEM AND THEIR SPARK PLASMA SINTERING[1]

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A synthesis of new material is the problem in the 21th century. Titanium boride (TiB) and titanium diboride (TiB_2) can be added in material and the properties of material increase. It has a lot of properties, such as high melting point, good wear resistance, high electrical conductivity and high hardness, so it can be used in metallurgy, medicine and aerospace industry [1, 2]. Moreover, ceramics based on nanopowders of Ti-B system shows better hardness and ductility. There are some methods of synthesis TiB_2 nanopowders: sol-gel reduction, self-propagating high-temperature synthesis, high energy ball milling [3-5].

This work shows a new way of production TiB_2 nanopowders – direct plasmadynamic synthesis using coaxial magnetoplasma accelerator [6]. This method used a capacitor banks with stored energy 68 kJ. The advantage of this method is that the formation of the final product takes a short time (about 1 ms).

The purpose of the work was to synthesize nanopowders with high content of TiB_2 and obtain ceramics based on it. According to the purpose three experiments with different ways of discharge initiations were implemented: 1) with titanium conductors; 2) with carbon fibers; 3) with graphite aerosol (graphitization). Synthesized powder products were analyzed without any pretreatment by X-ray diffraction (XRD), transmission electron microscopy (TEM). Qualitative X-ray analysis was made by PowderCell. The analysis showed that the maximum content of titanium diboride was in experiment using graphitization 93.2 %. It can be explained by the different nature of the transition of precursors to the plasma state: from the abrupt and short process in experiments with Ti-conductors and carbon fibers to a smooth and long process in the experiment using graphitization.

Ceramics was obtained on the synthesized product by spark plasma sintering method. It takes only a few minutes to complete a sintering process. Figure 1 shows that the hardness and density of the obtained ceramics depends on the method of initiation of an arc discharge. We can see that density and hardness grows with better experimental conditions. The best value of hardness 30.3 GPa shows the experimental with graphitization. The obtained ceramics showed high values of hardness, depending on the phase composition of the initial powder which is determined by discharge initiation method.

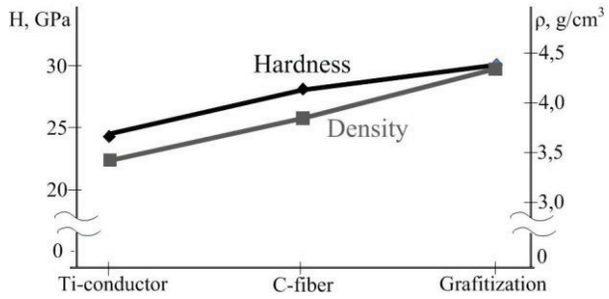

Fig. 1. Dependence graph of hardness and density of ceramics from the method of initiation of an arc discharge

Nanopowders of Ti-B phases were obtained by the synthesis in a hypersonic plasma jet. The most successful result was obtained using graphitization 93.2 % TiB_2. Ceramics based on it showed the best value of hardness 30.3 GPa.

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LIGHT-INDUCED SEDIMENTATION IN NANOLIQUIDS

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Processes of sedimentation are ubiquitous in nature and important for science and technology. Gravity settlers are commonly used to separate particles from waste streams and, in lab practice, analytical ultracentrifugation is a common tool to separate or characterize particle size distribution. Besides their practical interest, sedimentation studies on model systems have also provided fundamental information on the structural properties of colloidal suspensions [1].

In a gravitational field, only sufficiently large particles that are not subject to thermal (Brownian) motion are capable of precipitating. The steady-state deposition rate of the particles depends on the mass, size and shape of the particles, viscosity and density of the medium. In this case, the larger the mass and the particle size, the higher the settling velocity. For smaller particles, for example, molecules of natural and synthetic polymers, centrifugation is usually used [2].

Separators, working on the basis of the above methods, are quite bulky (large-sized) in the execution of the design. We suggest using light pressure forces for the sedimentation of nanoparticles in a liquid. These forces have a sufficiently large value, providing a sedimentation rate comparable with centrifugal methods.

This work is devoted to the model of sedimentation of nanoparticles by a light field, which is an alternative to the above methods, which makes it possible to create compact separators of small particles.

Consider the liquid phase medium with the nanoparticles (dispersed phase) which is under the influence of the reference laser beam with a uniform intensity profile I .

Balanced one-dimensional equation describing the dynamics of the concentration of the nanoparticles in a liquid phase medium with diffusion [3]:

$$\frac{\partial C}{\partial t} = D\nabla^2 C - V\nabla C \,, \tag{1}$$

here $C(z,t)$ - volume concentration of particulate matter, axis z is aligned with the reference beam I , D is diffusion coefficient; particle velocity:

$$V = \left(64\pi^4 a^5 n_1 \left(m^2 - 1\right)\left(m^2 + 2\right)^{-1}\left(9c_0\lambda^4\eta\right)^{-1}\right) I \,, \tag{2}$$

where $m = n_2 / n_1$, n_1 , n_2 - the refractive substance indices of the dispersion medium and the dispersed phase respectively, c_0 - velocity of light, a - particle radius, λ – light wavelength, η is viscosity of the fluid.

The relevant boundary conditions:

$$-D\nabla C + \vec{V}C = 0 \text{ , when } z = 0 \text{ and } z = l \,, \tag{3}$$

where l - the height of the cell along the propagation of the reference beam.

Initial conditions:

$$C = C_0 \text{ , when } t = 0 \,. \tag{4}$$

The exact solution of the equations (1)-(4) is due to the strong dependence (the radius of the 5th degree) of the deposition rate of the particle radius, which we believe can afford much more effectively diagnose polydisperse particle mixtures.

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PLASMODYNAMIC SYNTHESIS IN THE SI-C-N-O SYSTEM

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Nowadays, much attention is paid to materials combining different chemical, physical and mechanical properties. There are many works on binary inorganic compounds. A new departure was the study of materials produced from ternary systems. The Si-C-N system has some advantages with binary Si-C system: mechanical strength and high hardness, resistance to chemical influences, and high thermal conductivity [1]. Presumably, such properties are acquired due to the presence of a bond between all three atoms of the system.

The basis of methods for the production of ternary systems Si-C-N and others is deposition from a complex gaseous medium [2]. Such methods are toxic and explosive. The possibility of synthesis of the Si-C-N triple system is investigated in the work, using the method of direct dynamic synthesis in a coaxial magnetoplasma accelerator (CMPA) [3].

The series of experiments were conducted with a different gas atmosphere of the reactor-chamber (series 1-air, series 2-argon and air, series 3-argon). Power to the accelerator was supplied from a capacitive energy storage device. The results of experiments were the production of powdered products, which were studied by X-ray diffractometry (XRD) and transmission electron microscopy (TEM).

Structural-phase analysis of the products of series 1 showed that the powder consists of one phase – silicon dioxide, which gives on the diffractograms amorphous reflexes. The diffraction patterns of the second series includes the reflections of cubic silicon carbide phase and cubic silicon, and also amorphous silicon dioxide. Series 3 is distinguished by a high content of the phase of cubic SiC.

TEM-images confirm XRD-analysis. In Fig. 1 shows the accumulation of particles of the synthesis product obtained in the series 1. The formed spherical particles correspond to the morphology of silicon dioxide. The set of TEM-images shows that the presence of air particles of amorphous silicon dioxide in the reactor-chamber are dominated

The result of the work is the following: the preparation of Si-C-N system is not feasible in the air atmosphere, due to the oxidation of the phases entering into the product. Further research will be directed to the study of the formation of silicon carbonitride in the nitrogen atmosphere of the reactor-chamber.

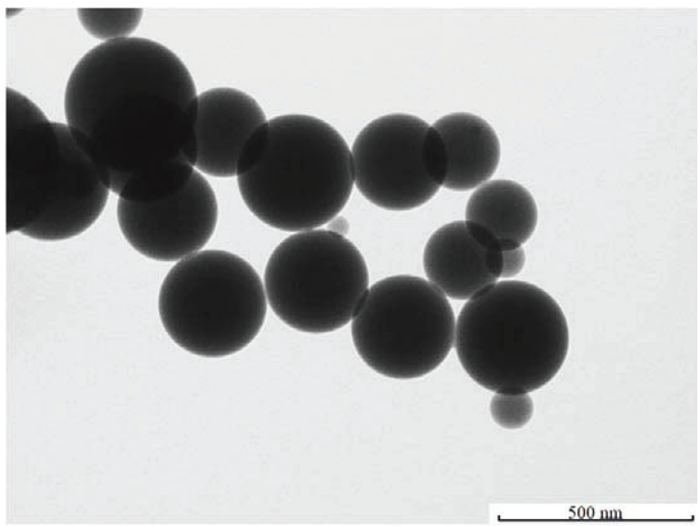

Fig. 1. TEM-image of the product, obtained in series 1

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SYNTHESIS OF AMORPHOUS CARBON NANOFIBERS ON A SURFACE OF COMMERCIAL CHLORINATED POLYMERS UNDER THE ACTION OF A HIGH POWER ION BEAM OF NANOSECOND DURATION

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The formation of layers of nanostructured carbon on the surface of polymeric materials is of great interest for the production of various elements for mobile electronics devices. A thermally stable polymers (polyimide, phenolic resin) is usually used as the substrate. An infrared laser (10,6 μm) is used to transform the surface layer of polymer into nanostructured carbon [1,2].

In the present work, as a polymer, we use inexpensive commersial chlorpolymers - chlorinated polyvinyl chloride and polyvinylchloride. A high power ion beam of nanosecond duration was used to affect the surface layers of polymers. Inorganic iron compounds ($FeCl_3 \cdot 6H_2O$ и $Fe(NO_3)_3 \cdot 9H_2O$) were used as the catalytic additive in the polymer providing growth of carbon nanofibers.

The irradiation was performed on a Temp accelerator by the ion beam (70% C^+ and 30% H^+) with energy E ≈ 200 keV, duration τ = 60 ns, and a current density range of 20–150 A/cm^2. The residual pressure in the accelerator chamber was $5 \cdot 10^{-3}$ Pa. Scanning electron microscopy (SEM, JSM-6610LV JEOL with an Inca-350 energy dispersive analyzer) and transmission electron microscopy (TEM, JEM-2100 JEOL with an Inca-250 energy dispersive analyzer, operated at 200 kV) were used to investigate the morphologies and structure of the polymer samples. Room temperature Raman spectra of the polymer samples were recorded on a DXR Smart Raman spectrometer (Thermo Fisher Scientific) using a 632.8 nm wavelength laser.

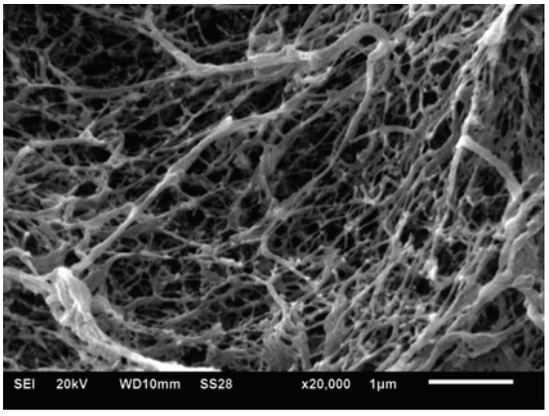

Fig. 1. SEM images of PVC surface after single HPIB irradiation with j=150 A/cm^2

We have found that inorganic iron salts can be used as a catalytic addition for synthesis of amorphous carbon nanofibers on a surface of commercial chlorinated polymers under the action of a high power ion beam of nanosecond duration. In this case, the irradiated polymer layer has a higher porosity, which is associated with the removal of water from these hydrated iron salts under high power ion beam irradiation. The most probable nanofiber diameter was in the range from 70 nm to 90 nm and the fiber length does not, usually, exceed 2 μm (Fig.1). A possible mechanism of the observed phenomenon is discussed.

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LONG TERM OPERATION OF 100-M DIFFERENTIALLY HARDENED RAILS: EVOLUTION OF DEFECT SUBSTRUCTURE

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In the modern conditions of high loads on the axis and movement speeds the surface layers of rails undergo the intensive plastic deformation leading to the damages in long-term operation, it may be the cause for the withdrawal of rails. One of the most important directions of development of notions of structural phase transformation is the determining of corresponding quantitative regularities along the rail cross-section.

As the production of 100-m differentially hardened rails by compressed air began comparatively recent the determination of nature and evolution regularities in long-term operation of fine structure in the head of these rails is of current concern and has the scientific and practical importance. The purpose of the research is the analysis of fine structure being formed at long-term operation of DT 350 rails by methods of layer-by-layer transmission electron diffraction microscopy.

The test materials were the samples of differentially hardened rails DT 350 passed tonnage of 691.8 mln t brutto in the process of testing on proving ground at experimental ring LTD «VNIIZhT». The investigation of phase composition and defect substructure of rails was carried out by methods of diffraction electron microscopy. The tests foils were manufactured by methods electrolytic of thinning of plates cut by electrospark method at 0, 2 and 10 mm distance from the tread surface along the central axis.

The following structure components were detected in the rail head along the central axis: the colonies of lamellar pearlite (fractional content ≈0.7), the grains of ferrite-carbide mixture (≈0.25) the grains of structurally free ferrite (≈0.05).

The operation of rails is accompanied by the essential transformation of material's defect substructure. It is clearly seen that the value of dislocation density reaches the maximum magnitude in the surface layer. As the distance from the tread surface increases the dislocation density decreases, in this case the type of dislocation substructure is practically unchangeable. The structure of dislocation chaos or ball-cellular dislocation substructure is present in the ferrite component of pearlite colonies, in the grains of structurally free ferrite and in the grains of ferrite-carbide mixture.

The steel structure formed in the process of long-term operation is in the elastic-stressed state. This fact is detected by the presence of bend extinction contours on the structural images. The presence of bend extinction contrours in electron microscope images is indicative of the elastic-stressed distorsions of the material's crystal lattice and may be caused by the mechanical effect on the rail metal in the process of operation. The stress concentrators of the test steel are the intraphase (the interphase of ferrite grains and pearlite grains belong to term) and the interphase (interphase of ferrite and cementite) interfaces.

All morphological constituents of steel (the lamellar pearlite grains, the ferrite-carbide mixtures grains and the grains of structurally free ferrite) undergo the essential transformation in long-term operation of rails. At 10 mm distance from the tread surface the relative content of grains of structurally free ferrite amounted to 5 % (note that the relative content of ferrite grains is practically independent of the distance to the tread surface); the grains of ferrite-carbide mixture -5%; the balance-pearlite grains. At 2mm distance from the tread surface relative content of ferrite-carbide mixture grains increased up to 10%; in the surface layer(the layer adjacent to the tread surface) measured 35%. It is evident that these transformations of steel structure take place at the expense of failure of lamellar pearlite grains. The performed studies of morphology of rail surface layer structure retained amounted to 25%; the balance – the pearlite grains in which the cementite plates are cut by gliding dislocations into separately located particles. These particles have globular shape, with their average dimensions being 30-50 nm.

For bulk hardened rails the formation of nanodimentional particles of carbide phase in steel ferrite constituent is observed after long-term operation. They are detected both in pearlite grains and in ferrite-carbide mixture grains and in grains of structurally free ferrite.

MODIFICATION OF TERAHERTZ PROPERTIES OF GRAPHENE/POLYMER MULTILAYERS BY ION BEAM OF NANOSECOND DURATION[1]

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Due to its unique properties, graphene has been a subject of intensive interest since it was successfully isolated. Its material parameters including mechanical strength and elasticity, very high electrical and thermal conductivity, and others suggest that graphene could replace various materials in existing applications and could also enable development of new technologies [1]. In particular, the high electron mobility and unique energy band structure provide a great potential for development of high-speed electronic device operating in microwave[2] and terahertz (THz) frequency range [3]-[4].

Recently, we demonstrated [3] that free standing multilayered structure consisting graphene monolayer separated by 700 nm thin polymer slabs can absorb up to 50% of incident radiation. The ideas of using carbonaceous sandwich structure as the main working components of different THz devices, such as filters, polarizers, collimators has been proposed theoretically and proved experimentally [5].In this communication, we continue our work and the dependence of THz properties of graphene/PMMA multilayer on the pulse ion radiation has been studied. The graphene sheet was synthesized by chemical vapor deposition (CVD) at 1000 ^{o}C in methane atmosphere on a copper foil and was covered by 600-800 nm thick PMMA layer. After desiccation of PMMA layer copper substrate was wet etched in ferric chloride. The obtained PMMA film coated with graphene sheet was washed in distilled water and placed on quartz substrate (0.5 mm thickness). The same procedure was repeated several times and allowed us to fabricate multilayer sandwich containing several graphene sheets. Ion irradiations were performed on a TEMP high-current pulsed accelerator, whose design and operating principle are described in [6]. In our experiments the 80 ns beam composed mainly of carbon ions (70%) and protons (30%) with energy of 300 keV and current density 10 Acm^{-1} was used. The measurements of complex transmission in the frequency range from 100 Ghz up to 1.4 THz before and after ion irradiation were performed using the time domain terahertz spectrometer (EKSPLA, Vilnius Lithunia) based on femtosecond laser (wavelength 1 μm, pulse duration less than 150 fs) and GaBiAs photoconductive switch as THz emitter and detector.

Although graphene was proved to be radiative resistant [7], its supports used in our experiments, i.e. PMMA and SiO2, having small penetration depth for studied ions, influence negatively to the radiation tolerance of overall graphene/polymer sandwich structure due to (i) destructive contribution of low-energy recoil atoms coming back to graphene from the side of dielectric support and (ii) gas bubbles formation on the graphene/polymer/SiO2 interface and their explosions leading to snapping off upper graphene layers. To explain the experimental results the morphology of graphene/PMMA layers before and after irradiation as well as ion induced defects in graphene layer was analyzed using Raman spectroscopy, SEM microscopy. Our experimental results were supported with STRIM simulation.

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PLASMA CHEMICAL TECHNOLOGY FOR PROCESSING RARE EARTH ELEMENTS IN THE PRODUCTION OF NANO- POWDER[1]

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Nowadays, rare earth metals (REMs) are actively used in a number of advanced high-tech industries, including electronics and radio equipment, instrumentation, semiconductor materials in the nuclear power industry, as well as composite materials.

Plasma chemical synthesis methods that enable production of highly dispersed and high-purity materials of desired composition with uniform distribution of components have become the most common practice of synthesizing powdered materials based on REMs and their oxides. The plasma chemical process does not require chemical reagents for sedimentation and separation of sediments from mother solutions, as well as such labor-intensive operations as drying and baking. This method reduces a number of process stages, eliminates processing of waste solutions, and minimizes their volume to the level not exceeding initial solutions. In addition, waste solutions can be recycled for preparing starting reagents. The plasma chemical method allows materials to be synthesized in the shortest possible time (10^{-3} – 10^{-1} sec).

The proposed method is based on the following processes:

1) plasma thermal destruction of metal salt solutions to oxides in a plasma stream carried out in the working space of the plasma chemical facility;

2) plasma thermal denitration of aqueous metal salt solutions followed by conversion to oxide powders in a plasma stream carried out in the working space of the plasma chemical facility.

The electron microscopy studies of produced oxides demonstrate that the basic morphological components of polycrystalline powders include polycrystalline hollow spheres and their fragments – transparent polycrystalline films and irregularly shaped particles. Solid spherical single crystal structures of metal oxides (opaque irregularly shaped particles) occurred less frequently. The specific surface area of powders was measured using the ID 188 device by the low-temperature nitrogen adsorption method. The X-ray phase analysis was performed by the DRON-UM1 unit with filtered copper radiation. Radiographs taken in accordance with were used to determine the quantitative phase composition, calculate lattice parameters and size of the coherent scattering region.

Furthermore, large variation of particle sizes (from 50 to 700 nm) was observed in all experimental powders, while the size of crystallites in polycrystalline particles does not exceed 20 - 30 nm. Depending on the salt concentration in the solution, proportions of powder particles with different morphology changed. With increasing a salt concentration in the solution, the specific surface area of particles was continuously increasing. Such changes in the specific surface area could be associated with both changes in the particle size and changes in quantity of its transparent and opaque particles depending on a salt concentration in the solution. A higher salt concentration in the solution is correlated with a greater number of powder particles in the form of hollow spheres and, therefore, higher specific surface area.

The report provides overview data, data from technological and analytical equipment, experimental methods, analytical data, general conclusions, conclusions about the prospects for using these technologies, and a list of references. The physicochemical and mathematical model of the process is shown.

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ATMOSPHERIC ARC PROCESSING OF FOOD POLYMERS IN NANOSIZED CARBON POWDER [1]

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The volume of polymeric wastage daily grows in the modern world. Therefore it is required to development of methods of it utilization. One of such way is plasma processing of polymer by plasma torch equipment [1] including in the air environment [2]. Carbon nanosized particles are possible useful product of that process [3].

Arc installation of a direct current which generates the arc discharge on graphite electrodes in the air atmosphere is developed in Tomsk Polytechnic University. Such arc systems of a direct current are rather simple, cheap and effective that plasma torch systems [4]. The air DC arc discharge plasma systems are considered as the modern effective technique for carbon nanomaterials synthesis [5].

We have made a series of experiments on a research of applicability of this system to a problem of processing of polymeric materials in carbon nanosized particles. As initial raw materials the PET polymer received by crushing of a plastic water bottle was used. The size of particles in the form of plates was about 2-3 mm at a standard thickness for plastic bottles. Initial polymer was put in a zone of formation of plasma structure between graphite electrodes. As a source of power supply the welding transformer of a direct current was used.

Under series of experiments transformation of polymeric material in a carbon ultradispersed product was realized. The XRD pattern reveals that the product is close to graphite structure, contains a considerable part of X-ray amorphous fraction. SEM image shows that product consists of particles with sizes less than 100 nanometers (Fig. 1).

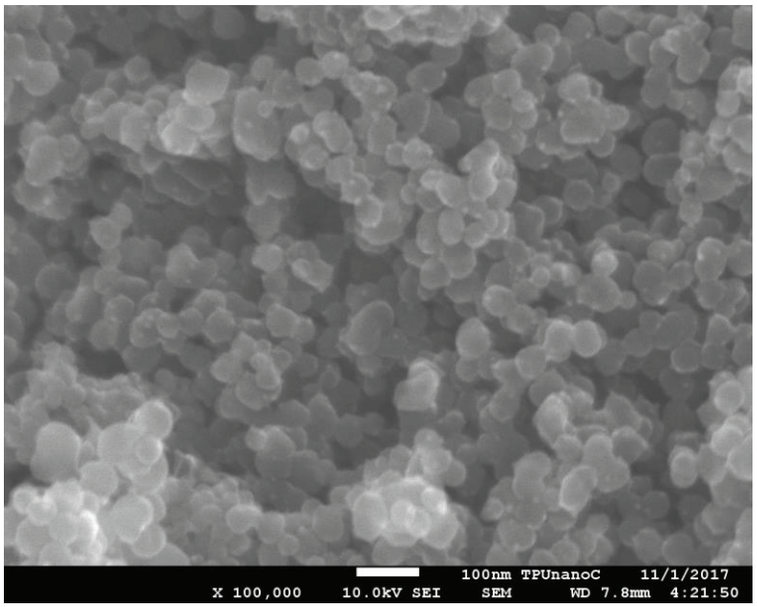

Fig. 1. SEM-image of obtained product (nanosized carbon)

Thus, processing of polymeric material in a carbon nanosized product in arc atmospheric plasma is experimentally realized.

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RECRYSTALLIZATION AND STUDIES OF THIN BISMUTH FILMS BY MEANS OF ELECTRON MICROSCOPY[1]

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Thin bismuth films are actively investigated in connection with their unusual thermoelectric, magnetic and quantum properties [1]. The Bi nanothin film for electron beam (e-beam) modifications and following transmission electron microscope (TEM) studies was prepared by vacuum evaporation on a carbon sublayer over mica substrate. Thin films separated from the substrate were placed on standard TEM grids. Irradiation of free-standing Bi film was carried out by an electron beam of LVEM-5 electron microscope in the scanning reflection mode using 5kV accelerating voltage. The irradiation time was from several seconds to about 10 seconds and more. Melting of a film with partial evaporation and a further recrystallization of Bi was the outcome. At exposure during ~ 10 seconds the carbon sublayer in the scanned area, Fig. 1a, is bared; during ~ 5 seconds– zones of a recrystallization and melting with partial evaporation, Fig. 1b, are distinguishable; ~ 4 and less – traces of a recrystallization were not found. After irradiation different film areas were investigated in TEM JEM-2100 (200kV).

In areas of partial evaporation Bi gathered in large drops of rounded shape not transparent for the e-beam. A small part of Bi crystallized in the form of the separate facetted crystals lying on a carbon sublayer Fig. 1c. On borders of a zone of local e-beam annealing Bi crystals with spherical edges, Fig. 1d, e, were created large, to several microns. Thickness of these crystals is estimated within 30 nanometers whereas thickness of an initial polycrystalline film lies within 10-20 nanometers. Details of their growth are of interest for further studies. In areas near regions of melting zones, near large single crystals, in some cases single crystalline areas with the strong internal bending of crystal lattice (around axis lying in the film plane) estimated from the analysis of bend contour patterns were formed. This unusual transrotational [2] nanostructure is quite often arising in thin films during crystallization from an amorphous state. The lattice bending in some areas is rather strong, about 60 degrees/micron, Fig. 1 f, g (right area at the bottom), as well as in most initial film fine crystal grains of hexagonal Bi phase, Fig. 1 d (left area).

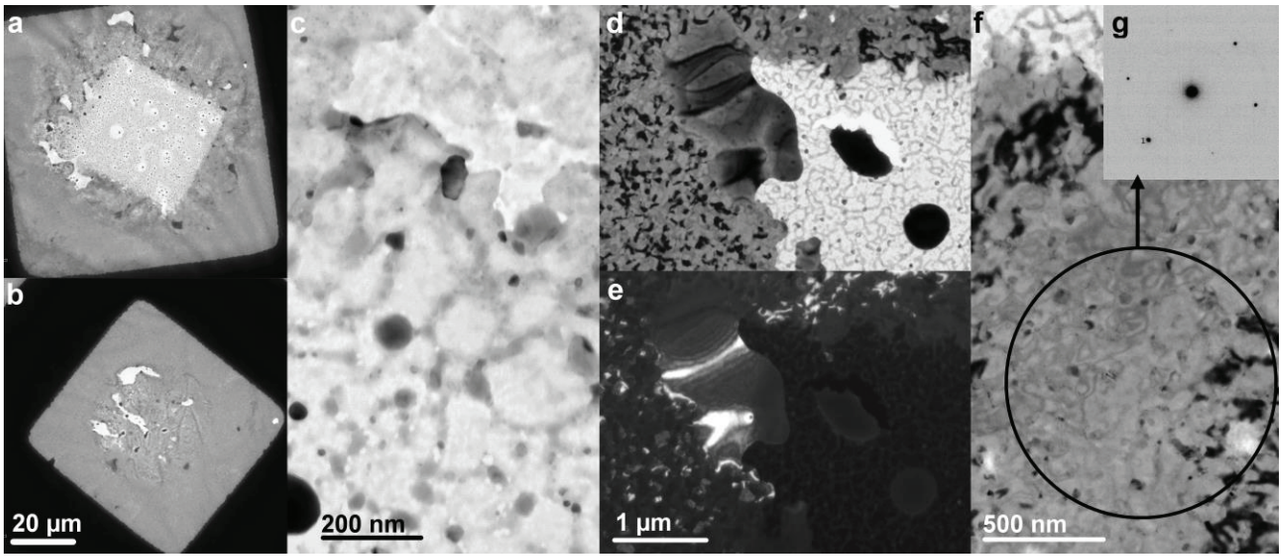

Fig. 1: TEM micrographs: film after e-beam irradiation for ~ 10 sec. (a), ~5 sec. (b); spherical particles and the facetted crystals in the field of partial evaporation of Bi (c); "large" single crystal near the meling zone border of initial fine-grained film (d); fine structure of bend contours in dark field used for thickness estimates (e); single crystalline areas near a zone of partial evaporation with strong internal lattice bending at the right bottom (f); selected area electron diffraction (g).

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