

FEATURES AND REGULARITIES OF ELECTRON-ION-PLASMA MODIFICATION OF HIGH-CHROMIUM STEEL*

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The purpose of this work is to establish patterns of formation of the structure and properties of 20Cr23Ni18 steel (similar to the USA – 310 steel), subjected to high-speed heat treatment. Steel 310 is used for the manufacture of parts and mechanisms operating at temperatures up to (1000-1050)°C (parts of the combustion chambers, guide vanes of gas turbines, etc.) [1]. Heat treatment of steel was carried out on the SOLO electron-beam setup with an electron source based on a low-pressure pulsed arc discharge with grid stabilization of cathode plasma boundary and open anode plasma boundary [2]. Irradiation was carried out with the following parameters: the energy of accelerated electrons $eV = 18$ keV; electron beam energy density E_S (J/cm^2) = 20, 30, and 40; beam pulse duration τ (μs) = 50 and 150; the number of pulses $N = 3$; pulse repetition rate $f = 0.3$ s^{-1} ; residual gas pressure (argon) in the working chamber ~ 0.02 Pa. The study of structure and phase composition of the material was carried out by the methods of scanning and transmission diffraction electron microscopy.

It has been established that steel 310 in the initial state is a polycrystalline material. Globular particles of the second phase of submicron sizes are located in the volume and along the grain boundaries. The particles are concentrators of stress fields and will lead to the nucleation of fatigue cracks with subsequent destruction of the material. It has been shown that electron beam treatment of steel leads to the dissolution of globular particles and the formation of the structure of cellular crystallization (Fig. 1). Nanoscale (≈ 25 nm) particles of the carbide phase are located along the cell boundaries and stabilize the defective substructure of the material. Performed tests have shown that prior irradiating steel 310 surface with a pulsed electron beam leads to a more than twofold increase in the fatigue life of the material.

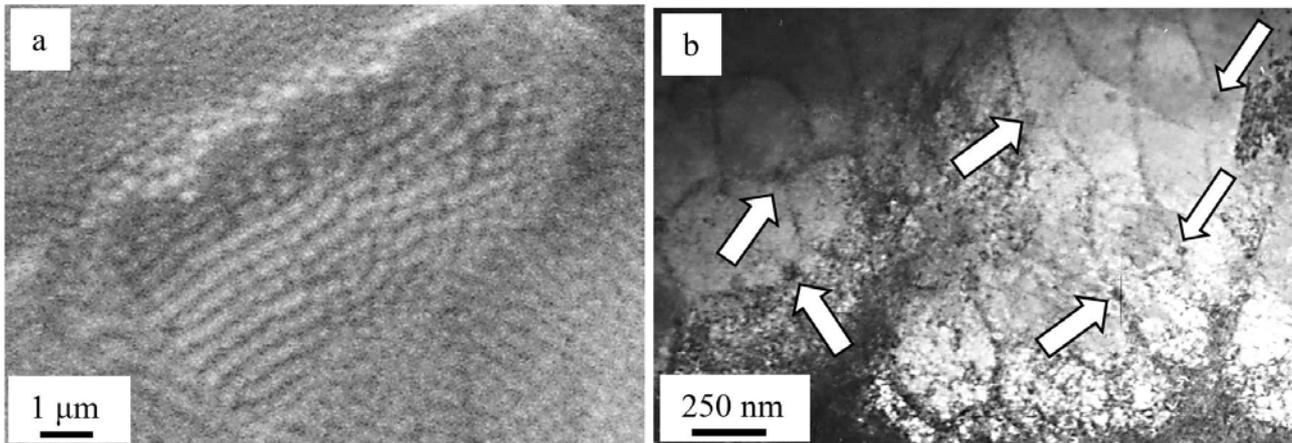


Fig. 1. Electron-microscopic image of surface layer structure of steel 310, irradiated with an electron beam. The arrows indicate particles of the carbide phase, located along the boundaries of the cells of high-speed crystallization. Irradiation mode: 30 J/cm^2 ; 50 μs ; 3 pls.

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DEPOSITION TISICN-COATINGS BY RF MAGNETRON SPUTTERING OF TITANIUM IN AR/N₂/((CH₃)₃Si)₂NH^{*}

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TISICN coatings, as is known, are capable to provide high protective properties at the expense of high hardness till 40-50 GPa and at their self-lubricating. High hardness is achieved by forming nanocomposite nc-TiCN and nc-SiC in amorphous matrix a-C and Si₃N₄ [1]. An analysis of the literary data showed the greatest microhardness is usually achieved at an chemical elements ratio Ti:Si:C:N ~ (45-40):(2-13):(25-35):(20-25) at.%. The present work was aimed to receive such four-component coatings at more lower temperatures by sputtering of required metal in activated gas environment of technological, accessible, cheap and ecological pollution-free Si-containing materials and afterwards to investigate this films. Titanium was deposited (~0.8 μm/h, ~1 h) by RF magnetron (400W) at total pressure ~0.5·Pa with simultaneous decomposition of gas mixture (hexamethyldisilazane 0.4-11 sccm, nitrogen 10-100 sccm and argon up to 80 sccm) by plasma of low energy electron beam of discharge with hollow self-heating cathode and distantly placed anode (40 cm) generated in DC (20 A) or pulse-periodic (50 kHz) mode <8-12 A>. Temperatures of using samples (quartz glass and stainless steel) were ~150 and ~500°C, maintained by additional heating of holder. The thickness of TISICN films was 0.3-10 μm.

The application of the discharge with self-heating cathode does not influence on current-voltage characteristic of magnetron discharge that does not allow to lower gas demand. The plasma composition was tested by an optical spectroscopy: the intensity of hydrogen H*(652.6 nm) lines in plasmas of magnetron discharge 400W or one with heating cathode (12A) was about equal, i.e. initially ((CH₃)₃Si)₂NH decomposition by both discharges was approximately identical. Also there was measured the thickness and microhardness of coatings, infra-red and X-ray diffraction spectroscopy were carried out.

The microhardness of coatings rose as increase the substrate temperature and at applying to the holder high-frequency (50 kHz) bias voltage $U_b=100$ V. Increasing of U_b up to 200 V led to reducing of that.

Optical microscopy shows films obtained at high nitrogen partial pressure are the strongly non-uniform and multiphase. The coatings on glass substrates have more homogeneous and smooth structure and possess by the greater microhardness.

An analysis of infra-red spectra allows to suppose, that both factors: increase of temperature and application of assisting discharge, as a rule, result in reduction a background signal of an absorption spectrum, included peaks from meta-stable atomic bonds, and minimizing of bonds being typical for an initial precursor molecule. Besides the absorption peaks of bonds SiC (670-706 cm⁻¹) and SiN (1100 cm⁻¹) amplified and ones of CN, SiH (2200-2300 cm⁻¹) and NH (3350-3380 cm⁻¹) diminished. The observed peaks close to 3800, 1700, 2600 cm⁻¹ may be connected with bounds of hydroxyl groups OH. The growth of temperature led to forming reflecting films at the equal fixed small precursor flows. Such spectrum modification testifies about preferential bonding of carbon and nitrogen in TiCN.

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INTENSIFICATION OF PROCESS OF DISSOLUTION OF SOLID SODIUM SILICATE ELECTRICAL DISCHARGES

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Currently, there are two main methods for producing liquid glass – autoclave and autoclave. The most productive way to obtain liquid glass is autoclave method. The main device in this technology is a rotating autoclave, which is a complex and cumbersome engineering structure.

Traditional methods of producing liquid glass have significant disadvantages. For example, the autoclave method involves the use of complex equipment equipped with a rotation drive, and the process itself requires high operating parameters – pressure and temperature. A sharp steam generator is required in the process production chain. In cases where the resulting product does not meet the required density, evaporators are used. They have low efficiency due to a small evaporation surface, and the evaporation process is energy-consuming [1].

Autoclave-free dissolution also has a significant disadvantage - namely, the starting material must be small, which requires grinding equipment. In addition, the autoclave-free dissolution of silicate blocks produces a significant insoluble residue.

The proposed technology for producing liquid glass – electric discharge [2], has a number of advantages over traditional. Electric discharge is characterized as a high-intensity process with a high concentration of energy. This is achieved due to the small time of electric discharge processes in the electric discharge channel and its small spatial dimensions.

Production time of liquid glass is an important technological and economic parameter. In the conditions of industrial production of liquid glass the main controlled parameter is its density. The process of producing liquid glass ends when the desired density is reached. Fig.1 the dependence of the density of liquid glass on the time of dissolution of silicate blocks for two methods of its production – electric discharge (ERS) and autoclave (Autoclave) is presented.

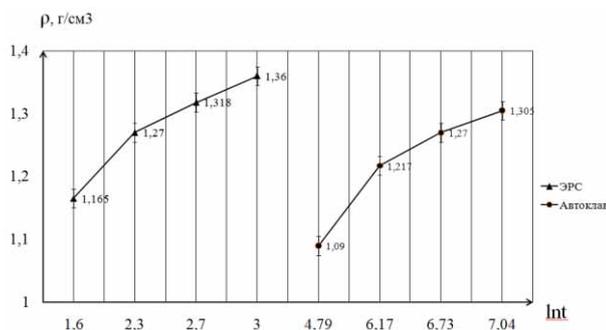


Fig.1 Dependence of the density of liquid glass on time

It follows from the presented dependences that the time of dissolution of a silicate block in a laboratory electric discharge reactor is 56 times less than the time of its dissolution in a stationary laboratory autoclave. The comparison was made for the obtained liquid glass with a density of 1.27 g/cm³.

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FORMATION OF BULK WC_{1-x}-BASED COATINGS ON METAL SUBSTRATES AT HIGH-SPEED SPUTTERING OF ELECTRIC DISCHARGE PLASMA JET*

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Since R.B. Levy and M. Boudart [1] has theoretically proved that tungsten carbide has catalytic properties similar to those of the platinum group metals for some chemical reactions, scientists have actively begun attempting to use it as a catalyst for the hydrogen production. It is known that the widely used hexagonal phases WC and W₂C, as well as the metastable cubic high-temperature modification WC_{1-x} (0 < x < 0.42), can be formed in the W-C system [2]. The main obstacle in the way of studying this cubic phase is the difficulty of its obtaining compared to the hexagonal W₂C and WC phases (narrow synthesis temperature range from ~ 2790 K to ~ 3060 K; transition to a hexagonal structure at slow cooling). However, there are some reports about the possibility of existing WC_{1-x} at room temperature [3], as well as it is believed that it can be obtained from the melt [2] with a crystallization rate of at least 10⁸ K/s [4]. In addition to an ambiguity in the issues of obtaining the cubic tungsten carbide phase (WC_{1-x}) in the form of powdered materials, there is a difficulty in obtaining a bulk sample, consisting in the phase transition from a cubic to a hexagonal lattice at a temperature above 700-800 °C [5]. This is one of the main obstacles and the reason why the bulk material has still not been synthesized and, accordingly, its structural, physico-mechanical, thermal and electro-physical characteristics have not been investigated.

Overcoming the above-mentioned obstacles of obtaining and studying WC_{1-x} powdered material is possible by implementing a synthesis process in a high-speed plasma flow containing carbon and tungsten atoms and generated by using a high-current (up to 10⁵ A) pulsed (500 μs) coaxial magnetoplasma accelerator of an electric erosion type. The distinctive features of the considered system are a high crystallization rate (10⁷-10⁹ K/s) and the versatility that allows synthesizing various materials (carbides, nitrides, oxides) in ultrafine form. The preliminary exploratory studies have already shown the possibility to synthesize the nanoscale cubic phase of tungsten carbide by this method [6].

This work presents the studies on the implementation of plasma dynamic method to create bulk coatings, predominantly containing cubic tungsten carbide, on the surfaces of metal substrates. It is found that using the plasma dynamic method it is possible to reach the yield of the WC_{1-x} phase in the coating structure of 85 wt. %. Such sufficient concentration of the WC_{1-x} phase allows estimating its physico-mechanical properties and completing the information about materials in the “W-C” system.

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MICRO-PLASMA ELECTROLYTIC TREATMENT OF THE METAL SURFACE: PROPERTIES OF COATINGS, THEIR APPLICATION

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Microplasma treatment of metals in electrolyte solutions by pulsed currents is one of the most promising, intensively developed methods for obtaining functional oxide coatings on aluminum alloys. Oxide layers can be formed by oxidizing the base material and by thermochemical transformations of electrolyte components and their subsequent melting on the surface of the part. The obtained MAO coatings of MANEL company are not inferior in their properties to ceramics and surpass the oxide coatings obtained by anodizing. The paper offers an overview of the results of tests and applications of MAO coatings of MANEL company.

THE POSSIBILITY OF APPLYING RUNAWAY ELECTRON PREIONIZED DIFFUSE DISCHARGE FOR SYNTHESIS OF DIAMOND, DIAMOND-LIKE COMPOUNDS AND GRAPHENE*

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Interest in the study of electric discharge due to the possibility of its use in various fields of human activity has not weakened for many decades. Until now, one of the main factors influencing on the classification of the types of discharge was the pressure of the ionized gas. Fairly recently, because of the development of technologies a new factor has appeared which one gives a possibility to redefine the existing classification and allows research to be targeted at the looking for a new types of electric discharge. This factor is the duration of lifetime or evolution of the electric discharge. In particular, a discharge occurring as diffuse discharge, independent of the pressure and type of the surrounding gas, was predicted and then realized – a runaway electrons preionized diffuse discharge (REP DD) [1,2].

For more than fifteen years our laboratory has been researching this type of discharge. Today the laboratory has implemented many experimental setups for the study of various characteristics of this discharge [3-5]. Also one of the tasks is to find opportunities for its practical applicability. It is already known that treatment by this type of discharge has a weakly penetrating effect into the surface of materials. So it is possible to use REP DD for fine surface cleaning of damage critical materials [5].

It is known that electric discharges are used for the synthesis of hydrocarbon compounds, diamond-like coatings and diamonds [6]. Theoretical calculations have shown that the energy characteristics of a runaway electrons preionized diffuse discharge make it possible to use this one in such synthesis process, but the efficiency will be very low. However, due to the relatively low energy deposition, it is feasible to produce materials with a high degree of purity and uniformity.

In a mixture of hydrogen and methane it is possible to synthesize diamond-like/diamond films using a REP DD, because it forms atomic hydrogen in a low-excited state required for etching non-diamond forms of carbon and CH-radicals which are necessary for the deposition of crystalline carbon with sp³-hybridization of carbon bonds. Heating of the substrate caused an increase in the homogeneity of the discharge. This paper presents the first results of the possibility of synthesis of carbon compounds under the action of REP DD depending on the gas pressure and concentration of its components.

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MOLYBDENUM CARBIDE EMBEDDED INTO CARBON MATRIX SYNTHESIZED BY DC ARC PLASMA *

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Molybdenum carbide is one of the most important material among metal carbides because of its unique physical and chemical properties such as good resistance to corrosion and oxidation, high abrasion resistance, relatively high electrical conductivity, high melting point, high hardness [1]. Molybdenum carbide due to its catalytic activity can be used for non-platinum electrocatalyst in order to develop cost-effective hydrogen-evolutions technologies [2]. Traditionally molybdenum carbide is synthesized by carbon and molybdenum (or molybdenum oxide) powder mix annealing at ~1400 °C - 1500 °C. Also molybdenum carbide crystalline phases can be obtained by DC arc plasma generation [3-4]. One of the useful material for catalysis is considered a composite based on molybdenum carbide nanoparticles embedded in to carbon matrix [5] because of possible particles surface oxidation exposed by air and aggregation of pure molybdenum carbide materials.

In this paper the material based on molybdenum carbide Mo₂C nanoparticles embedded into carbon matrix is presented. This material has been prepared by the DC arc discharge procedure. The typical high resolution TEM-image and selected area electron diffraction pattern are presented in the Fig. 1. The particle averaged size is about ~3-5 nm; according to the selected area electron diffraction data these particles are characterized by the structure close to the Mo₂C.

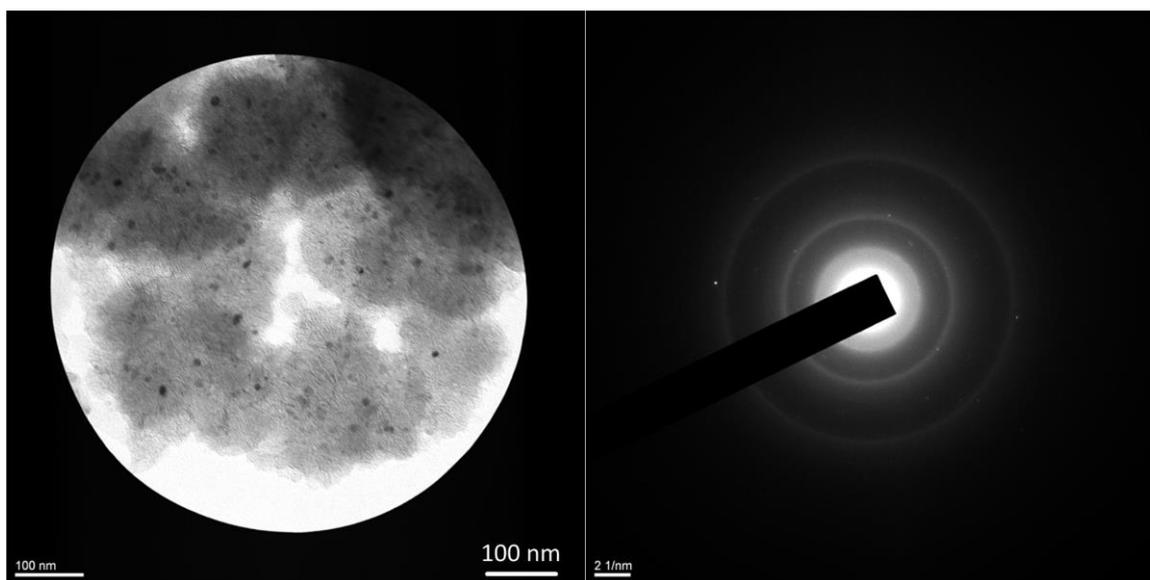


Fig. 1. Typical HRTEM-image and SAED

The arc discharge method is possible to use for molybdenum carbide embedded into the carbon matrix material synthesis. Such material according to the literature data can be useful as a catalyst for the hydrogen evaluation processes.

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MULTI-CYCLIC ELECTRON-ION-PLASMA ALLOYING OF SILUMIN: STRUCTURE, PROPERTIES*

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The aim of the research is to develop a method of high-cycle alloying of silumin samples surface layer by electron-ion-plasma combined method. As a material for the study, AK12 grade silumin (alloy Al-12% Si) was used in the cast state. The alloying of the surface layer of silumin with titanium was carried out by melting the «film (Ti)/substrate (AK12)» system with an intense pulsed electron beam. The thickness of the titanium film in each «deposition/irradiation» cycle was 0.5 μm ; number of cycles was 1; 5; 10. Multi-cycle alloying was carried out in a single technological vacuum at the «COMPLEX» setup [1]. The study of the elemental and phase composition, the state of the defective substructure was carried out by the methods of optical, scanning and transmission diffraction electron microscopy. The phase composition and the state of the crystal lattice of the phases were studied by X-ray phase analysis. Mechanical and tribological properties were investigated by determining microhardness, wear resistance and friction coefficient.

It was found that AK12 silumin in the cast state is a multiphase material and contains inclusions of the second phases of various sizes and shapes (Fig. 1, a). Irradiation of the «film/substrate» system leads to the modification of the surface layer up to 160 μm thick with the formation of a structure of high-speed cellular crystallization (Fig. 1, b, c). By methods of X-ray phase analysis it was revealed the formation of intermetallic compounds of TiAl_3 and TiAl composition in the surface layer of modified silumin.

It is shown that the microhardness of the modified surface of silumin increases by ≈ 1.4 times and reaches maximum values after 5 cycles of «deposition/irradiation». Wear resistance reaches maximum values also after 5 cycles of «deposition/irradiation» and exceeds the wear resistance of the initial material by 14.2 times.

It is obvious that high values of the wear resistance of the modified material are due to the release of intermetallic particles in the surface layer.

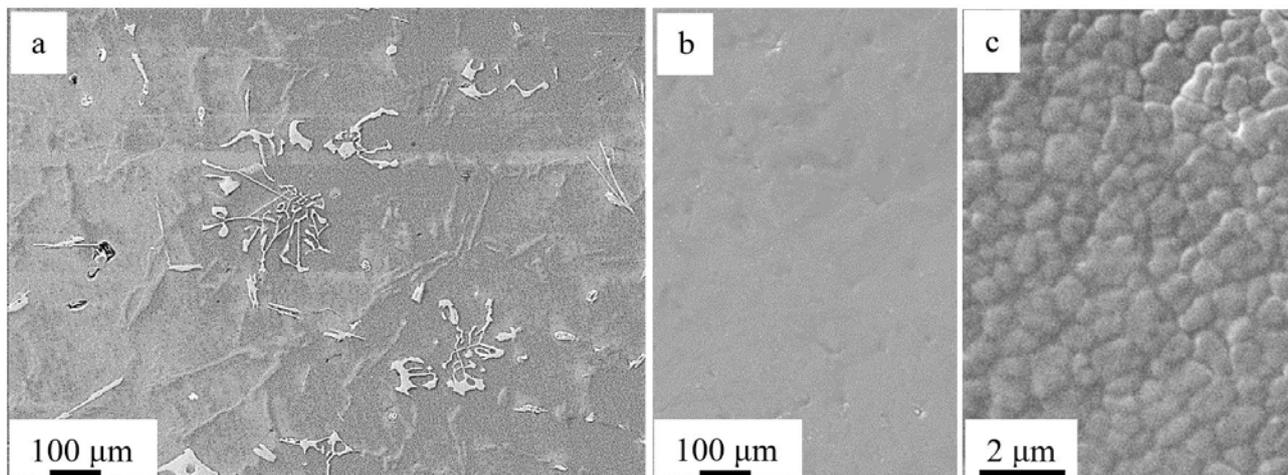


Fig. 1. Electron-microscopic image of AK12 cast silumin structure in the initial state (a) and after modification for five «deposition/irradiation» cycles (b, c).

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

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

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

The hypoeutectic silumin AK10M2N was used as a test material. At the first stage the electroexplosion ion plasma alloying of samples by the yttrium oxide powder was carried out using the following regime: the aluminium foil mass – 58.9 mg; Y₂O₃ powder mass – 58.9 mg; the discharge voltage – 2.8 kV. At the second stage the alloyed surface of the samples was irradiated by the intense pulsed electron beam at the plant SOLO. The following parameters of electron beam were used: the energy of the accelerated electron – 17 keV, the energy density of electron beam – 35 J/cm², the pulse duration – 150 μs, the number of pulses 3, the pulse repetition rate – 0.3 s⁻¹, the pressure of the residual gas (argon) in the working chamber of the plant – 2·10⁻² Pa.

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

The cardinal transformation of the structure of the material's surface layer ≈ 70 μm thick consisting in the dissolution of silicon inclusions and intermetallides of micron and submicron dimensions characteristic of the cast silumin and the formation of the gradient multielemental submicro- nanodimensional structure has been revealed. It has been found that the modified layer has the structure of the high-velocity cellular crystallization and contains the inclusions of the faceted shape whose relative content decreases when moving away from the surface of modification. It has been shown by the methods of micro-X-ray spectral analysis that the surface layer of silumin is a multi-elemental one and along with the atoms of the initial material (aluminium, silicon, copper, nickel, chromium, iron) it is additionally enriched by the atoms of titanium, yttrium and oxygen. It has been established that the cells of high velocity crystallization are enriched by aluminium atoms and the interlayers separating the cells are enriched by silicon atoms. The inclusions of the faceted shape are enriched by the atoms of titanium, aluminium and copper and the interlayers along the boundaries of the inclusions contain, mainly, the yttrium atoms. The performed electron – microscopic microdiffraction analysis shows that the inclusions of the faceted shape are formed by the phase Al₅CuTi₂. Along the boundaries of these inclusions the interlayers having the phase composition of AlCuY are found. It has been revealed that the interlayers of silicon located along the boundaries and in the junctions of the boundaries of the crystallization cells formed by the solid solution based on aluminium have a nanocrystalline structure with the crystallite dimensions varying within 10-20 nm.

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EFFECT OF PULSE DURATION AND GAS PRESSURE ON DRY REFORMING OF METHANE IN NANOSECOND SPARK DISCHARGE

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Carbon dioxide conversion of methane (or “dry reforming” $\text{CH}_4 + \text{CO}_2 = 2\text{CO} + 2\text{H}_2$) is one of the main reactions of plasma-chemical processing of natural gas. Additional interest in this reaction is associated with the possibility of utilizing carbon dioxide in order to reduce the greenhouse effect[1].

For the excitation of the medium in the study of carbon dioxide conversion, various types of discharges are used: such as corona, diffuse, DBD, and spark. In [2] it was shown that when a medium is excited with a short pulse with a duration of 1 ns, the spark discharge has the highest efficiency. Also in [3] it was established that for a diffuse discharge, the degree of methane conversion strongly depends on the pressure of the medium and the proportion of reacting gases. In this paper, dry reforming of methane was investigated when a medium was excited by a spark discharge at various pressures of the gas mixture and discharge pulse durations.

In our experiments, high-voltage pulse generators with output voltage from 50 to 200 kV, pulse duration from 1 to 30 ns, pulse energy from 0.05 to 3 J were used. Methane conversion was studied at pressures from 0.5 to 5 bar for various ratios of methane and carbon dioxide. The main parameters defined were the degree of methane conversion and the specific energy consumption for the conversion of the methane molecule.

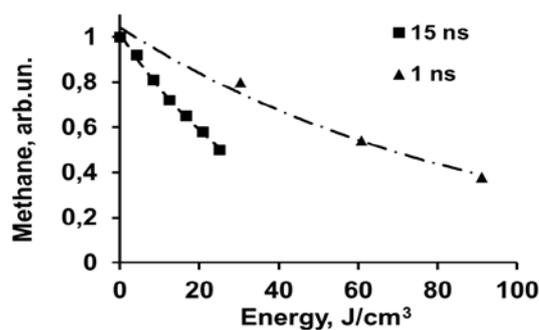


Fig.1. Dependence of methane concentration on the energy input to the gas for discharges with a pulse duration of 1 ns and 15 ns. Pressure is 1 bar, $\text{CH}_4 / \text{CO}_2$ mix 1: 1

It has been shown that for a spark discharge, an increase in the duration of the excitation pulse significantly increases the methane conversion efficiency. Figure 1 shows the dependence of methane concentration on the energy input to the gas for discharges with a pulse duration of 1 ns and 15 ns. In addition, as in the case of diffuse and corona discharges, for a spark discharge, an increase in the pressure of the gas mixture leads to a decrease in the degree of methane conversion. The minimum specific energy consumption for methane molecule conversion was about 20 eV / molecule.

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IMPROVEMENT OF EFFICIENCY OF THE USE OF PULSED CORONA DISCHARGE ENERGY DURING THE CONVERSION OF VOLATILE ORGANIC COMPOUNDS*

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The addition of chlorine- and fluorine-containing molecules to gas mixtures, which have the highest electron affinity energy, leads to the most noticeable changes in the properties of discharges and plasma. It is known that conversion of most volatile organic compounds (VOCs) is initiated by electrons with energies sufficient to break certain chemical bonds (most often double) in an organic molecule. When electronegative molecules are added to a mixture containing VOCs, it can be expected that concentration of electrons will decrease as a result of electron attachment processes, and it will lead to a decrease in the discharge current and at the same time to a proportional decrease in the degree of conversion of VOCs. However, experiments on the conversion of VOCs of various types in pulsed corona discharge plasma demonstrated that when CCl_4 or SF_6 is added to the mixture, a decrease in the discharge current and energy input into the gas mixture is observed, but the degree of VOC conversion either remains almost unchanged or even increases [1]. To explain the result obtained, it is necessary to analyze the processes occurring in different regions of pulsed corona discharge. During the formation of pulsed corona discharge in the “wire – cylinder” configuration, three regions can be distinguished, differing in the electric field strength and electron temperature (Fig. 1).

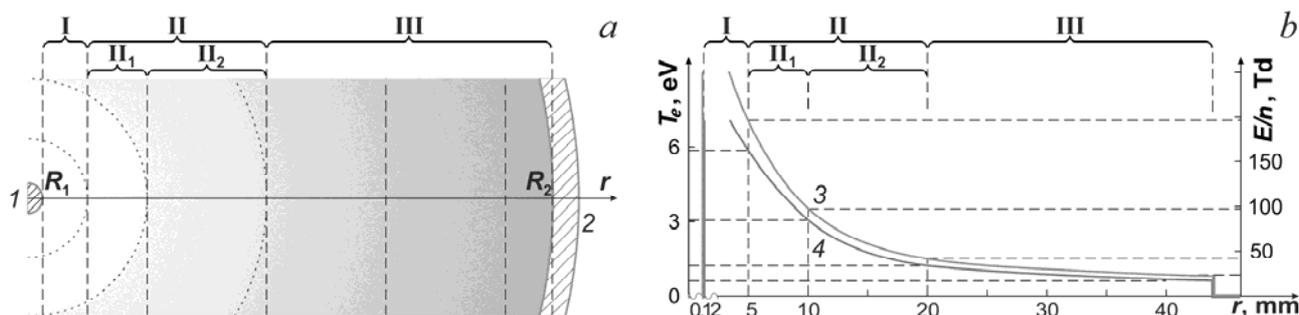


Fig. 1. (a) Regions of pulsed corona discharge formed between wire cathode (1) and cylindrical anode (2). (b) Dependencies of reduced electric field strength E/n (3) and electron temperature T_e (4) on the distance r from the cylinder axis. I – strong field region; II (II_1 and II_2) – moderate field region; III – weak field region

In region I ionization by direct electron impact, dissociation, and electron excitation of molecules occur, in region II_1 – electron and vibrational excitation of molecules, in region II_2 – vibrational excitation, and in region III – elastic processes and electron drift to the anode. Concentration of electrons in region I reaches $5.6 \cdot 10^{11} \text{ cm}^{-3}$. When CCl_4 is added, the processes in region I remain unchanged; however, dissociative attachment additionally occurs in region II_1 , and three-body attachment occurs in regions II_2 and III.

The VOC conversion is initiated only in region I, in which attachment processes do not occur. The attachment processes in regions II and III significantly reduce the discharge current and the energy input due to a decrease in the concentration of electrons not involved in the conversion of VOCs, whereas the conversion processes in the strong field region are not inhibited. Comparison of concentration of plasma electrons and changes in concentrations of gas mixture components in our experiments suggests that there are about 20 disappeared tetrachloroethylene molecules C_2Cl_4 , about 60 disappeared trichloroethylene molecules C_2HCl_3 , and about 10 appeared ozone molecules O_3 per one plasma electron.

Thus, addition of electronegative impurities allows to improve the efficiency of the use of pulsed corona discharge energy during VOCs conversion in atmospheric-pressure air.

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FEATURES OF ELECTRON-BEAM PROCESSING OF METAL-CERAMIC POWDERS IN THE FOREVACUUM

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Currently, the technology of creating materials with varying properties in at least one direction are based either on the synthesis of the entire volume of the material in one cycle – the traditional thermal sintering, microwave sintering, plasma-spark sintering method, etc., or on the creation of the material due to its gradual formation of thin layers - by lamination (LOM), selective laser Sintering (SLS) or melting (SLM) [1]. Each of these methods is applicable to a limited range of materials. The creation of functionally graded materials in one technological cycle by energy impact on the workpiece containing components with a given distribution of the composition in one direction is limited by the difficulty of controlling the impact, which leads to uncontrolled change in properties. In addition, the properties of the synthesized materials in this case directly depend on the properties of its constituent components, the control of which is carried out only at the initial stage of the formation of the product. Thus, the use of electron irradiation with a focused beam in the conditions of active gas medium of the forevacuum range will allow to create functionally graded materials of not only simple, but also complex volume form [2]. The success of the application of the electron beam in the forevacuum for the sintering of ceramics is presented in [3]. For layer-by-layer synthesis of bulk products with changing properties and control of parameters and properties of materials, a narrow-focused electron beam with an energy of 10 keV and a current of 50 mA and a diameter of less than 1 mm was used. As a result of the work, a thin layer of powder material was irradiated, containing aluminum oxide ceramics and titanium in a different mass ratio. Determine the modes of electron beam irradiation allows sintering of metal-ceramic powders in the forevacuum. These results indicate the principal possibility of obtaining by the method of layer-by-layer electron-beam sintering of bulk products from metal-ceramic powder.

The work is supported by the Ministry of science and higher education of the Russian Federation - grant of the President for doctors of Sciences MD-2649.2019.8.

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PARAMETERS OF ION STREAM FROM AN ELECTRON-BEAM-PLASMA GENERATED BY A RIBBON ELECTRON BEAM*

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Low-temperature plasma generated by a low-energy electron beam (1-10 Kev) passing through the gas atmosphere of the vacuum chamber [1] is used in various materials processing technologies [2, 3]. The parameters of such beam plasma can be controlled in a fairly wide range due to changes in the current and energy of the electron beam, as well as the composition of the gas atmosphere. The most effective plasma generation occurs at pressures of 5-100 PA – i.e. in the so-called forevacuum range. This paper presents the features of the generation and application of gas ion fluxes from the plasma formation of a large area formed during gas ionization by a ribbon electron beam in the forevacuum pressure range. The influence of electron energy and electron beam current on the concentration of the beam plasma and the current density of the ions extracted from it is determined. The results of materials processing by ion flux from beam plasma are presented.

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HIGH-INTENSITY PULSED ION BEAM GENERATION IN PLASMA EROSION MODE

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The results of a study of the generation of high intensity pulsed ion beam in a diode with a passive anode when operating in a two-pulse mode are presented. When the polarity of the accelerating voltage changes, the plasma erosion mode [1] is realized in the A–K gap, ions are accelerated from the gas plasma, which can ensure the formation of a pulsed beam of gas ions, see Fig. 1.

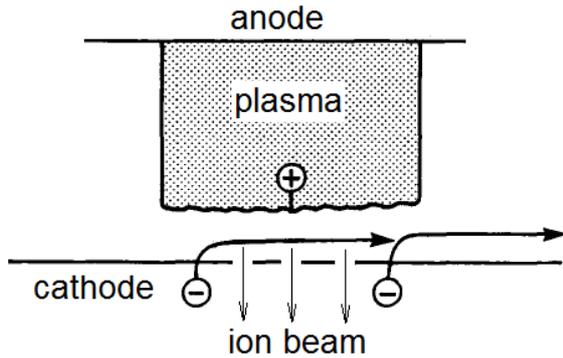


Fig. 1. HIPIB generation scheme in plasma erosion mode

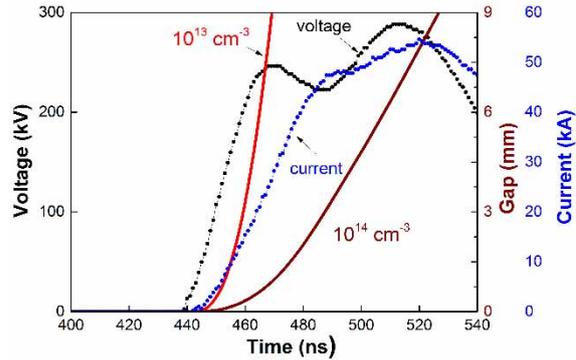


Fig. 2. Waveforms of accelerating voltage (second pulse), diode current and increasing of width of the vacuum gap λ near the cathode at different plasma density (line)

If the width of the vacuum sheath near the cathode in plasma erosion mode increases by an amount of $\Delta\lambda$ in a time of Δt , then the total ion and electron charge removed from the gap is:

$$\Delta Q = 2e \cdot n_0 \cdot S \cdot \Delta\lambda$$

where S - cathode area, n_0 - concentration of gas plasma.

Dividing both sides by Δt , we find the current in the diode:

$$I(t) = 2e \cdot n_0 \cdot S \cdot \frac{d\lambda}{dt}$$

Vacuum sheath near the cathode is:

$$\lambda(t) = \frac{1}{2e \cdot n_0 \cdot S} \int_{t_0}^{\infty} I(t) dt$$

Fig. 2 shows the calculation of the vacuum sheath near cathode (λ) during the plasma erosion mode.

The analysis performed showed that in our experimental conditions [2], the concentration of gas plasma in the A–K gap does not exceed 10^{13} cm^{-3} and the pulse duration of the ion current generated in the plasma erosion mode will be less than 20 ns. However, the duration of the ions beam formed by an ion diode is ~ 200 ns [2]. In addition, the time-of-flight diagnostics of the ion beam shows a good agreement between the experimental and calculated pulse shape of the ion current density [2]. We simulated the signal profile from the collimated Faraday cup provided that the accelerating voltage is equal to the total voltage, applied to the diode. This corresponds to the condition of the acceleration of ions from a thin plasma layer on the surface of the anode, and not from the gas plasma in the A–K gap, see Fig. 1. Therefore, the generation of ions in a diode with a passive anode when operating in a plasma erosion mode is unlikely.

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INFLUENCE OF ENERGY DEPOSITION MODE ON THE EFFECTIVENESS OF THE PLASMA TREATMENT OF WATER IN BUBBLE CHAMBER*

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It is that the non-thermal plasma treatment of liquid (depending on the conditions of the discharge) is based on the simultaneous influence of several physical and chemical factors on the initial liquid: ultraviolet radiation, shock waves, neutral, charged and chemically active particles [1]. The resulting liquid is generally referred to as «plasma-activated». To obtain such liquids, various devices are used, in particular, based on the fact that gas bubbles form in the liquid and a pulsed discharge is ignited. The corresponding class of technical means is called bubble discharge reactors.

In the present work, the influence of coordination of water bubbling process and energy deposition is studied. The setup is including a pulse voltage source with an amplitude of 8-10 kV, frequency from 2 to 50 kHz, pulse front from 20 to 900 ns. This source was connected to a wire anode with a diameter of 0.7-1 mm, located in a dielectric tube and having an internal diameter of 8-10 mm and a narrowing to 1.2-1.5 mm. Air was pumped through the dielectric tube (flow rate up to 5 l/min). The anode, dielectric tube and cathode were placed in water in a dielectric vessel. For matching the deposited energy, the capacity was installed between the cathode and the voltage source.

Voltage pulses from the source were fed to the anode, and at the same time there was an air supply into the dielectric tube, and the liquid was bubbled. As a result, a discharge is ignited between the anode and the interface between the liquid and gas, intensive ionization occurs. Then the chemical species forms and saturate the water as the bubbles move to its surface.

We tested this experimentally using the described setup in case of distilled water treatment. The water was bubbled with voltage pulse repetition rate was up to 50 kHz. Processing was carried out for 2 minutes. The bubbles formation frequency f_b , which was set by the gas flow rate, was determined using an ultra-linear condenser microphone PMC-2 with a three-contact output and a frequency response of up to 20 kHz, close to linear, or using videorecording.

After discharge treatment, the water changed its chemical composition, turning into a solution with new optical absorption spectra. They consist of characteristic NO_3^- absorption band in the wavelength range of 200-250 nm. Registration of absorption spectra was carried out by StellarNet EPP2000-C25 spectrometer. The acidity and conductivity of the solution were also monitored.

In the described experiment, water solutions containing NO_3^- ions were obtained. It is known that only this kind of ions provide nitrogen absorption in plants. Then the obtained solutions in tenfold dilution were used for irrigation of flax seeds ("TOST1" cultivar) and wheat ("Irgina" cultivar). Observation of the development of the root system of seeds showed that compared with irrigation with ordinary water, wetting with treated water leads to a 2.5-5 fold increase in the length of the roots of plants and a twofold increase in their dry weight. This is a strong evidence in favor of the industrial applicability of the obtained solutions to stimulate plant growth in agriculture.

It should be noted that for additional matching of energy deposition into the bubble with its life time, it is also necessary to coordinate the electrophysical properties of the liquid with the parameters of the voltage pulses. We assume that the value of the voltage pulse front ρ must be consistent with the conductivity of the liquid ρ as τ [ns] $\sim 1 / (1-10 \cdot \rho)$ [$\mu\text{Sm/cm}$] for ρ values from 1 to 1000 $\mu\text{Sm/cm}$. For large values ρ , the discharge treatment process will be inferior to electrolytic methods. This is a physical limitation on the efficiency of the energy input mode in the plasma treatment of water in the bubble chamber.

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X-RAY MICROANALYSIS OF SILUMIN IRRADIATED BY AN INTENSE PULSED ELECTRON BEAM*

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Al-Si alloys (silumin), due to their low specific weight, relatively high specific strength, good fluidity, belong to cast alloys that are widely used in automotive industry, aircraft-shipbuilding [1]. The presence of coarse inclusions of the second phases, leading to a high brittleness of the material is a clear disadvantage of silumin. The purpose of this work is to establish the patterns of redistribution of alloying elements during irradiation of silumin by an intense pulsed electron beam.

Silumin AK10M2H was used as the study material. Silumin was irradiated at the SOLO setup [2] with the following parameters: the energy of accelerated electrons is 17 keV; electron beam energy density of 35 J / cm²; irradiation pulse duration 150 μs; number of pulses 3; pulse repetition rate 0.3 s⁻¹. Irradiation was carried out in argon plasma at a pressure of 0.02 Pa. The studies of the elemental composition and the state of the defective substructure of the samples of cast silumin and after electron beam irradiation were carried out using transmission diffraction electron microscopy (JEM 2100F).

Using micro X-ray spectral analysis of thin foils, it was established that in cast silumin the alloying elements form inclusions of the second phase of complex chemical composition (Fig. 1, a – c). The dimensions of the inclusions from units to tens micrometers. The irradiation of silumin in the mode of melting of the surface layer is accompanied by the formation of a submicro-sized cellular structure (Fig. 1, d-f). The inclusions of the second phases with sizes less than 100 nm are quasi-uniformly distributed in the volume of the modified layer and are located along the boundaries and in the volume of the cellular substructure. In the aggregate, this change in structure is accompanied by a multiple increase in the microhardness and wear resistance of the material.

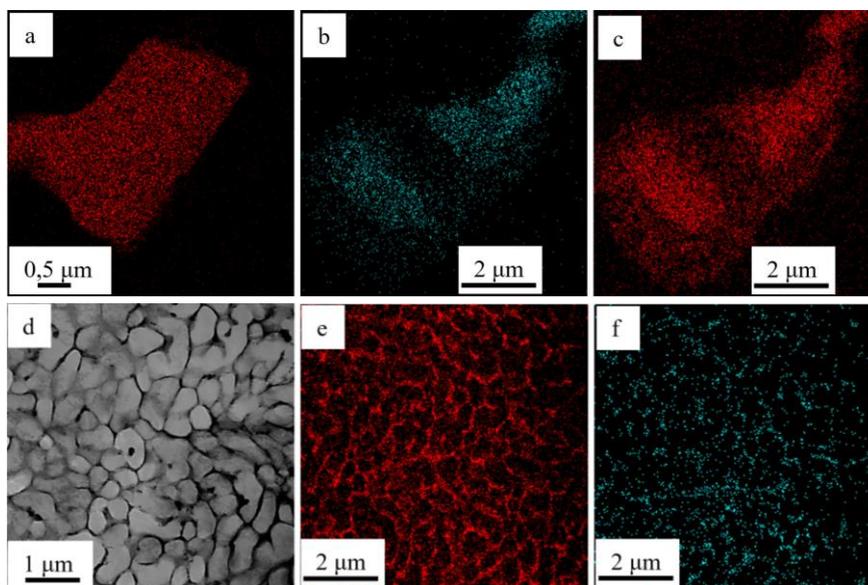


Fig. 1. The structure of silumin in the cast state (a-c) and after irradiation with an electron beam (d-f); images (a, f) were obtained in the characteristic X-rays of silicon atoms; b, f - nickel; c - copper.

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THERMODYNAMICAL ANALYSIS CLINKER FORMATION PROCESSES UNDER THE CONDITION OF LOW TEMPERATURE PLASMA

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Plasma-chemical synthesis of cement clinker is one of the promising methods for cement production [1,2] and is characterized by the intensity, single-stage and complete physical and chemical processes occurring in the liquid phase, which determine the relevance of research in this direction. At the same time, the study and analysis of a set of complex phenomena occurring during chemical interactions and phase formations of clinker minerals are necessary: they provide an opportunity to get valuable information about the methods of targeted reactions, ways of rational management of technological processes and finding new ways to improve the technology of cement production.

Analysis of the obtained results allowed us to establish the thermodynamic probability and the sequence of formation of calcium silicates: the primary compound (about 600 K) in the system is CS (1). This is indicated by the first intersection of the graph with the x-axis at 582 K (309 °C). Subsequently, the formation reactions of C₃A (6), C₂S (3), C₄AF (7) and C₃S₂ (2) are carried out, in the temperature range of 650–750 K (377–477 °C). As the temperature rises, the probability of formation of clinker minerals changes: from 708 K (435 °C), the predominant compound formed during heat treatment is C₂S (3), the synthesis of which is possible on the basis of CS (1). This advantage persists to a temperature of 2180 K (1907 °C), after which the formation of a C₃S compound (4) is most likely. This is indicated by the intersection of the graphs of these reactions and the subsequent increase in the absolute values of the Gibbs energy, which at 3000 K (2727 °C) reach 197.6 and 186.7 kJ/mol, respectively, for reactions C₃S (4) and C₂S (3). So in the area 685–2180 K (412–1907 °C) the probability of the formation of a phase of dicalcium silicate is high. According to the schedule, with an increase in temperature (more than 2180 K), only the phase of tricalcium silicate (4) can exist in the melt, which can be formed on the basis of such minerals as C₄AF (7), CA (5), C₃A (6), CS (1), C₃S₂ (2) and CaCO₃, CaO. Along with this, it was found that the synthesis of C₃A (6) from CA (5) and CaO or CaCO₃ is impossible, since the graphs of ΔG change in the formation of these compounds do not overlap. This suggests: the formation of tricalcium aluminate is carried out from the melt, which is consistent with the studies of V.I. Grandma's.

As a result of thermodynamic analysis of the reactions of the synthesis of silicates, aluminates, calcium alumina ferrites, it was found that the use of low-temperature plasma (LTP) for the synthesis of cement clinker is appropriate for the studied mixtures and their C:S ratios. Analysis of the obtained calculations showed that in the traditional temperature range (1000 ÷ 1800 K) the probability of mineral formation and the sequence of reactions with the ratio of oxides in the raw mixtures C: S = 3.32: 1 is preserved and can be located in the following sequence: C₂S, C₃S, C₃S₂, CS. With an increase in the temperature range under study to 3000 K, the thermodynamic probability of the formation of silicates changes: C₃S, C₂S, C₃S₂, CS. Beginning with 2180 K, C₃S is the most stable compound formed under NTP conditions, which is impossible with the traditional technology (1673–1723 K). Thus, the use of highly concentrated heat fluxes in cement production creates unique conditions for modifying the sequence of chemical reactions during the formation of clinker minerals, and has a positive effect on the quality and properties of the sample being synthesized.

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CREATING A CERAMIC COATING ON METAL*

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The aim of the work was to obtain electrical insulating ceramic coatings on the surface of metals.

The evaporation of ceramics was carried out by a continuous electron beam in a vacuum chamber at a pressure of 5-6 Pa in a residual air atmosphere. The continuous electron beam was generated by a fore-vacuum plasma electron source, the principle of which is based on the emission of electrons from the plasma of a hollow cathode through a single emission channel. Constructive and functional features of this source, as well as its parameters are described in detail in [1, 2]. In the present work, the diameter and length of the emission channel were 1.5 and 2 mm, respectively, at which, during the evaporation process at an accelerating voltage of 18–20 kV, the emission current was 16–30 mA. The diameter of the electron beam on the target was controlled with a single magnetic lens and maintained so that the melting bath forming the vapor from which evaporation takes place fills the entire upper part of the ceramic target. The evaporation process was preceded by a preliminary sintering of the ceramic target, which was carried out by gradually increasing the parameters of the electron beam for 20–25 minutes. The evaporation time at the exposure of the above parameters was 30-60 minutes. Tablets of alumina powder with modifying additives were used as ceramic targets. The deposition rate of the ceramic coating without the formation of a droplet fraction was 50-100 nm / min. Coating thickness 1.5-2.5 microns. The composition of the coating mainly contains aluminum oxide. The contribution of modifying additives is no more than 1-2%.

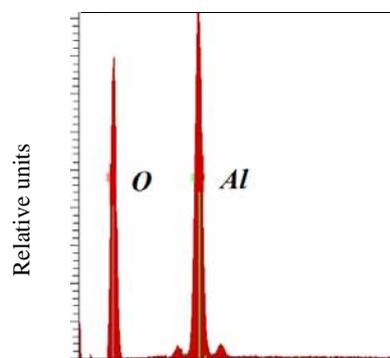


Fig. 1. The composition of the coating

As a result, there is a continuous film containing micro-cracks on the metal surface [3]. In our opinion, this is due to the difference in the thermal expansion coefficients of the metal substrate and the ceramic film during its deposition.

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ELECTROEXPLOSIVE ELECTRICAL EROSION RESISTANT COATINGS OF THE AG-W SYSTEM USED FOR ELECTRICAL CONTACTS OF POWER MINE EQUIPMENT*

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When analyzing the structure on the metallographic cross-sectional microscope of all three samples, the formation of a multilayer structure is revealed, which consists of a low-porosity coating, slightly varying thickness, a liquid-phase alloying layer, and a heat-affected layer. The coating thickness equals to $49.04 \pm 0.7 \mu\text{m}$ for mode 1, $68.5 \pm 0.9 \mu\text{m}$ for mode 2 and $61.26 \pm 0.6 \mu\text{m}$ for mode 3. Coating thickness is measured using the vertical secant method. Coatings in modes 2 and 3 were more uniform in width than in mode 1, which can be explained by the higher temperature of the jet and, therefore, the rate of coating diffusion to cooling was higher. It can be seen from figure 1 that in the first treatment mode, pores of 3 to 30 μm in size are present in the coating. In treatment mode 2, the pore size decreases compared to mode 1. The average pore size in the second mode is 16 μm . In the third treatment mode, the average pore size is 8 μm . Thus, as the absorbing power density increases, the average pore size in the coating of the Ag-W system decreases.

The average value of the layer thickness with the changed state between the coating and the substrate is 14 μm for mode 1, 18.5 μm for mode 2 and 20 μm for mode 3. The width of the layer of the changed state between the substrate and the coating increases as the values of the absorbing power density on the coaxial electrodes increases as well. The analysis of the transition layer between the coating and the substrate showed that the boundary is not even. A zone of mutual mixing of the coating with the substrate is formed. Analyzing the data of the comparative histogram, it can be concluded that the processing mode 2 has the maximum average microhardness of the coating layer, in comparison with other investigated modes. It is 457.5 ± 55.2 . In the substrate the microhardness values are smaller than in the coating layer and are $119.4 \pm 2.5 \text{ HV}$ and $122.0 \pm 3.3 \text{ HV}$ at a distance of 5 and 40 μm from the coating, respectively. It is also important to note that the average microhardness at a distance of 5 μm from the coating for all treatment modes is less than the average microhardness at 40 μm from the coating. It is possible to make an assumption that the reason for this is the heat effect, which is realized when the coating of the Ag-W system is applied by the electroexplosive deposition method. The final stage of the complex study of the regularities in the formation of electroexplosive electroerosion-resistant coatings of the Ag-W system was the atomic force microscopy of the coating obtained in the optimal exposure mode.

The atomic-force image made it possible to establish that the application of coatings of the Ag-W system on the copper contact by the method of electroexplosive deposition in the optimal treatment mode leads to the formation of a structure consisting of a coating, a layer of changed state and a substrate material. The average thickness value of the layer with the changed state located between the coating and the substrate is 16 μm , which correlates with the data obtained in the metallographic analysis.

In the present research devoted to the investigation of electroexplosive coatings of the Ag-W system formed on copper contacts KPV-604, the modes of deposition and the sample weights of Ag-W powder were chosen, coatings were applied by electroexplosive deposition on copper contacts in various modes, metallographic studies for microhardness were carried out and atomic force microscopy of the coatings obtained. The results of microindentation made it possible to reveal the optimum deposition mode ($U_2 = 2.5 \text{ kV}$), in which the coating layer has the greatest average microhardness value in comparison with other investigated modes. This value is $457.5 \pm 55.2 \text{ HV}$, which is 3.8 times higher than the average value of microhardness in the copper substrate. The analysis of the thin section roughness in Mode 2 showed that the average roughness value of the coating is greater than the average roughness values of the substrate and the layer with the changed state by 50.015 nm and 22.849 nm, respectively. Thus, coating of the Ag-W system on copper contacts improves their mechanical and physical properties. The treatment modes studied can significantly increase the microhardness of the surface contact layer, as well as increase their electroerosion resistance due to the presence of tungsten particles in the coating, and also maintain the necessary electrical conductivity due to the presence of silver particles.

* The present work was performed within Grant of the President of the Russian Federation for state support of young Russian scientists – doctores of sciences MD-89.2019.2

EFFECT OF NITROGEN PRESSURE AND PULSED POWER SUPPLY PARAMETERS ON THE PROCESS OF ION NITRIDING IN GLOW DISCHARGE PLASMA.

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Plasma nitriding has been used in industry for over 30 years and is an alternative to gas nitriding [1]. Important advantages of plasma nitriding are the absence of pollution, high energy efficiency and process controllability [2]. The report presents the results of experiments on hardening the surface of details (steel AISI 5140) using nitriding in a glow discharge plasma. To generate a gas discharge in the fore-vacuum pressure range, a power source was used, which is capable of operating in direct and pulsed current mode. Fig. 1 shows the current-voltage characteristics of a glow discharge that forms at different pressures of nitrogen. As shown in the graph, high discharge power (20 kW) and, accordingly, details temperature (550–580°C) can be maintained over a wide range of operating pressure by varying the discharge voltage and current. In this work, three main regimes were investigated: at low (50–100 Pa), medium (250–300 Pa), and high (500–550 Pa) pressures. The work investigated the influence of the working pressure and electrical parameters of the discharge on the physico-mechanical characteristics of the nitride and diffusion layers. It is shown that the regulation of gas pressure makes it possible to control the length, structure and hardness of diffusion saturation regions in steel. The use of a pulsed power supply allows reducing the arcing at the stage of training and heating of parts, which makes it possible to reduce the total duration of the process.

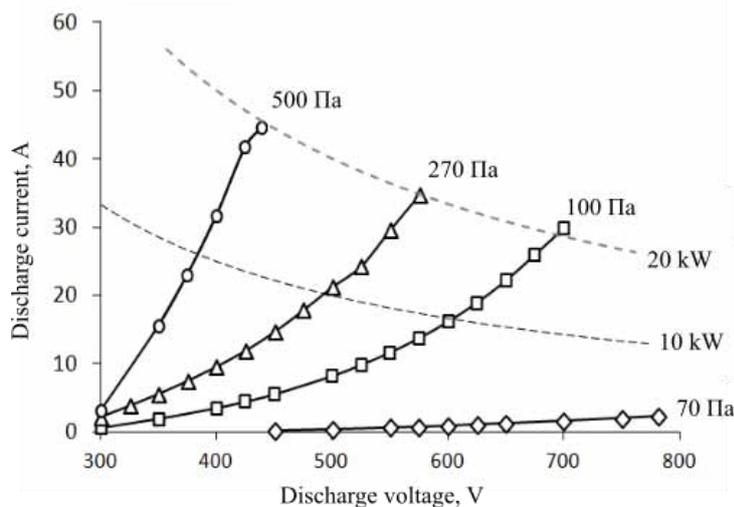


Fig. 1. V-A characteristics of a glow discharge formed in a plasma nitriding system at various pressures of nitrogen in the chamber.

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LOW-TEMPERATURE CEMENTATION OF STAINLESS STEEL IN ELECTRON BEAM GENERATED PLASMA*

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The method of low-temperature (400-500 °C) cementation of 12X18H10T stainless steel by decomposition of acetylene in a wide (100 cm²) low-energy (200-300 eV) electron beam generated plasma in an Ar+C₂H₂ gas mixture was investigated. The composition of a beam Ar+C₂H₂-plasma is investigated and it is shown that the degree of decomposition of acetylene varies with the current and energy of the electron beam. It is shown that the magnitude of the flow of acetylene significantly affects the formation rate and hardness of the hardened layer (Fig. 1). From the obtained results it can be seen that at fixed values of argon pressure (~0.8 mTorr), beam current (3.5 A), bias voltage (-120 V), sample temperature (500 °C) and exposure time (3 h) increase in Q_{C₂H₂} from 1 to 4-5 cm³/min leads to an increase in the thickness and microhardness of the hardened layer. With a further increase in Q_{C₂H₂}, an abrupt decrease in the rate of formation of the solid layer occurs. One explanation for the nature of the dependence of the properties of the hardened layer on the acetylene flow may be that for small values of Q_{C₂H₂} an increase in the growth rate of the layer is achieved due to an increase in the concentration gradient of active particles on the sample surface; particles by ion etching the surface and diffusing carbon atoms into the sample volume. When more active carbon particles enter the surface than are carried away by ion etching and diffusion into the reinforced volume, then a carbon film is formed on the surface separating the active saturating medium from the material being hardened and no layer is formed.

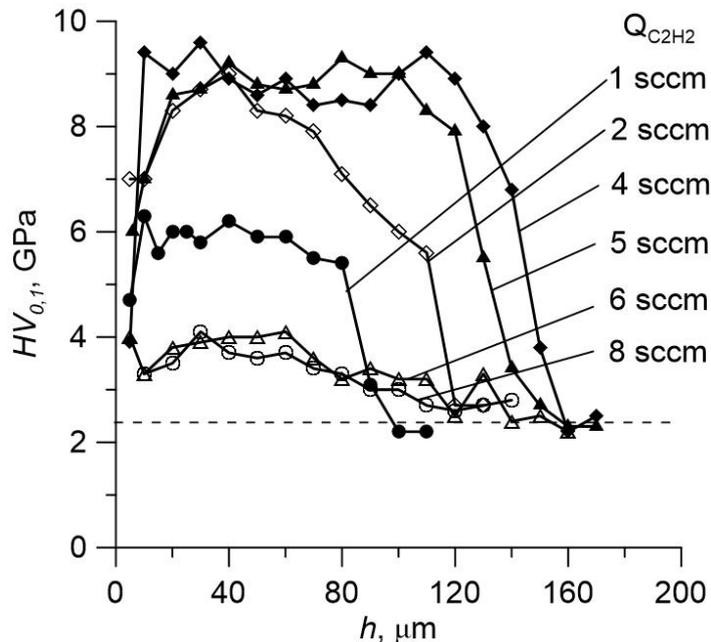


Fig. 1. Microhardness profiles of near-surface layers of hardened samples. The beam current is 3.5 A, the temperature is 500 °C, and the treatment time is 3 hours. The flow of acetylene is 1-8 sccm.

* This work was supported in part by RFBR, grant No. 18-38-00561_mol_a.

INVESTIGATION OF THE CONDITIONS FOR THE FORMATION OF SiCN-BASED COATINGS IN ELECTRON BEAM GENERATED PLASMA*

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Method of deposition of SiCN-based coatings in a large volume chamber (0.3 m³) by decomposition of organosilicon precursor (hexamethyldisilazane, HMDS) in nitrogen-argon electron beam generated plasma at pressure of ~1 mTorr at temperatures 200-600°C was investigated. The analysis of plasma composition by optical emission spectroscopy are carried out. The influence of electron energy (50-500 eV), beam current (10-40 A) and the flow of HMDS (1-10 sccm) on plasma composition and HMDS decomposition degree was investigated. It is shown that the degree of decomposition of precursor molecules in electron beam plasma is higher, than in discharge with self-heated hollow cathode [1]. On the surface of samples made of glass and stainless steel, silicon carbonitride (SiCN) coatings up to 4 μm thick with hardness up to 28 GPa were obtained during 2 hours at 600°C. 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).

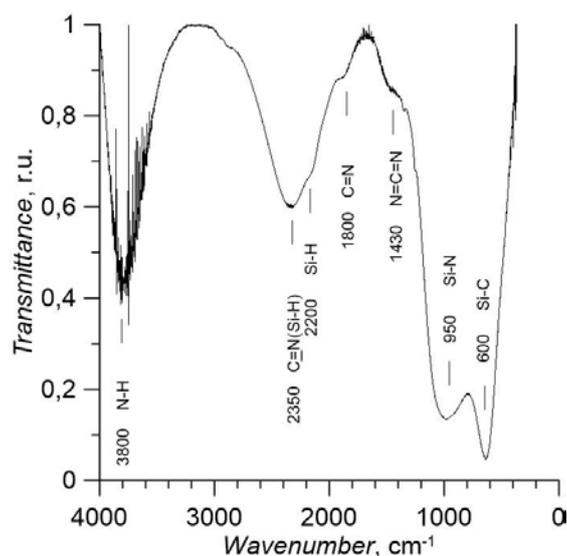


Fig. 1. IR-spectrum of the coating on steel 12X18H10T. Temperature 500°C, beam current 1,5 A, bias voltage -100V, pressure Ar+N₂+HMDS 1 mTorr (Q_{Ar} : Q_{N₂} : Q_{HMDS} =5:1:2).

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CHARACTERIZATION OF NANOSILICA PRODUCED BY ARC PLASMA METHOD

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Obtaining functional nanomaterials is a crucial task of modern science. Silicon dioxide nanopowder (nanosilica) is in demand in various industries and it is advisable to develop new methods for its production.

The research shows possibility of production and characterization of the structural and morphological properties of silicon dioxide nanoparticles obtained by arc plasma method [1-3]. This method allows to use available and environmental friendly natural high-silica materials such as diatomite, quartzite and quartz sand. The arc plasma method is based on physical processes of melting and evaporation of raw material under the influence of thermal plasma of electric arc discharge with subsequent condensation of nanoparticles.

Developed atmospheric pressure DC arc plasma installation was used to obtain the nanosilica. The main structural and morphological characteristics of the obtained nanoparticles were determined. The method of transmission electron microscopy (TEM) was used to study the morphology and size distribution. Brunauer–Emmett–Teller (BET) method was used to study the surface. Energy-dispersive (EDX) and X-ray photoelectron spectroscopies (XPS) were used to determine an elemental composition. The nature of chemical bonding of obtained nanopowder was characterized using a Fourier transform infrared (FTIR) absorption spectroscopy. To study the processes of phase transitions in raw materials after plasma influence, the method of X-ray diffraction (XRD) was used.

The particles of nanosilica obtained by arc plasma method have spherical shape, the size distribution 10-300 nm, the specific surface area 37-71 m²/g.

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FORMATION OF CATALYTIC LAYERS BY ION BEAM ASSISTED DEPOSITION OF METALS FROM VACUUM ARC DISCHARGE PLASMA*

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Currently, processes of surface modification with the use of ion-plasma technologies are active being developed, which is due to the possibility of a substantial increase in the performance of structural and functional materials without changing the structure and volume properties. The ion-beam modification of functional materials, the properties of which are determined primarily by the surface composition, in particular, of heterogeneous catalysts of chemical reactions, is of great interest [1, 2].

The goal of our research is the preparation of catalytic layers with use of ion beam assisted deposition of active metals from vacuum arc discharge plasma, which is generated in metal vapor.

Catalytic surface layers were formed by ion beam assisted deposition (IBAD) of platinum as basic active metal and one of metals (Ir, Sn, Ce, Gd, Dy, Ho, Yb) as an activating additive. We carried out IBAD mode in which the deposition of the metal and the mixing of deposited layer with the substrate are carried out by accelerated ($U = 10$ or 5 kV) ions of the same metal [3–10]. Deposition of the metal and mixing of the deposited layer with substrate by accelerated ions of the same metal were performed in an experimental unit, respectively, from a neutral fraction of metal vapor and the vacuum arc discharge plasma of a pulse arc ion source, respectively. A vacuum of $\sim 10^{-2}$ Pa was maintained in the working chamber. Active layers of the electrocatalysts were formed by ion beam assisted deposition of metals onto substrates from valve metals (Al, Ti, and Ta) and carbon materials (glassy carbon, and Toray Carbon Fiber Paper TGP-H-060 T and AVCarb® Carbon Fiber Paper P50 carbon fiber catalyst carriers which are used as material of diffusion layers of membrane electrode assemblies of electrolyzers and fuel cells with polymer membrane electrolyte).

Investigation of the microstructure and composition of layers was carried out by SEM, EBSD, EDX, WD XRF, XPS, and RBS methods. It has been established [3–10] that the obtained catalytic layers are characterized by amorphous atomic structure and contain atoms of the deposited metals, substrate material, as well as impurities of oxygen and carbon; their thickness reaches ~ 30 – 50 nm. The content of each deposited metal is several percent by weight. Inclusions of the deposited metals of about several micrometers occur on the surface which is conditioned by metal drops deposition from the arc source; they cover less than 1% of the surface area.

Electrocatalysts with the obtained layers exhibit activity in processes of hydrogen evolution and oxidation of alcohols – methanol and ethanol [3–5, 7, 8]. These processes underlie the principle of operation of alternative hydrogen energy devices: electrolyzers for producing hydrogen and direct methanol and ethanol fuel cells (DMFC, DEFC). Electrocatalysts containing one of rare earth metals (Ce, Gd, Dy, Ho, Yb) as an activating additive exhibit higher activity in oxidation reactions such as ethanol and methanol. A distinctive feature of the resulting electrocatalysts is their higher activity during the oxidation of more complex ethanol molecules compared with methanol, where it is required to ensure the breaking of the C–C chemical bond. Formation of an active surface during the ion beam assisted deposition of the two metals is carried out under vacuum conditions in two steps, which compares favorably with traditional multistage methods for the preparation of supported catalysts based on impregnation of the support with solutions of the compounds of each of the metals, their reduction to the metallic state, drying and etc.

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FORMATION OF THE SILICON COATING ON THE NITI SUBSTRATE BY PLASMA IMMERSION ION TREATMENT¹

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Thin films and coatings made from silicon on various substrates are widely used in diverse fields of industry, science and engineering. The high biocompatibility of silicon provided use of silicon in medicine as biological sensors and coatings for implants. One of the problems is the deposition of silicon coatings on hard-shaped products such as intravascular stents. The prospect decision for this problem is considered as the using of plasma immersion ion implantation and deposition (PIII&D) method. Because of it, the study of formation mechanism of silicon coatings and their physical-mechanical characteristics by using this method is the topical issue.

In this work, the research of structure and properties of silicon coatings on the nitinol substrate was performed by using PIII&D method depending on technological parameters. The choice of nitinol with nickel content equaled 50,9 at.% is explained that it is used for production of self-expanding intravascular stents. The silicon target with purity equaled 99,999 was used for creation silicon coating. The formation of silicon coating was conducted on device called «SPRUT». This device has plasmatron with thermoemission cathode and magnetron sputtering system with non-balanced magnetron [1]. There is a circle platform for planetary rotation, where the samples of nitinol can be situated in special holders on periphery. The minimum and maximum distances between the samples and the magnetron were 200 and 600 mm respectively. Argon under pressure of 1 Pa was used as an inert gas.

Depending on the mode, the coatings with different thickness were obtained (Fig. 1). It is identified that the main influence on the coating thickness is rendered by the time of treatment and the distance between the magnetron and the substrate. The value of the voltage affects the coating thickness to a much lesser extent: the thickness of coating decreases virtually evenly. The microhardness of coating was from 10 to 15 GPa.

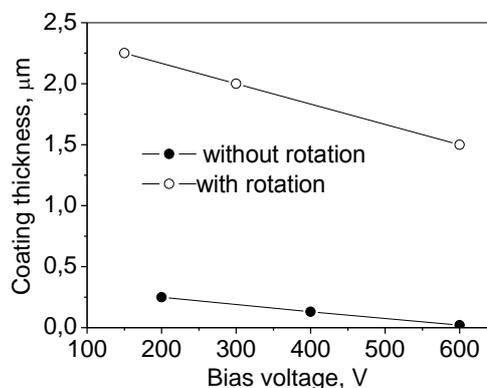


Fig. 1. The dependence of the coating thickness on the value of bias voltage

The value of adhesion toughness of coating and substrate depends on the temperature of prerequisite heating of cover. There was the delamination of coating from substrate during the process of cover formation when the temperature of heating less than 180 °C. The prerequisite heating of substrate up to 300 °C produced good adhesion.

Thus, the using of PIII&D method allows to obtain the silicon coatings on the nitinol substrate in the range of thicknesses from 0.1 to 2.2 µm with good adhesion.

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ABOUT INFLUENCE OF A CHANGE RATE OF A SUBMILLISECOND ELECTRON BEAM ENERGY DURING ITS PULSE ON MODIFICATION OF A STEEL SURFACE*

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The purpose of these studies is to analyze the structure and surface properties of SUS321(12Kh18N10T) stainless steel exposed to a high-current low-energy submillisecond electron beam depending on the change rate of the electron energy of the beam during its pulse. The samples were irradiated with the following general parameters of the electron beam: $U_0 = 20$ kV, $\tau = 200$ μ s, $W = 30$ J/cm², and the different change rate of the electron beam energy during its pulse was achieved by using a different capacitance high-voltage capacitor bank (3 and 12 μ F) while maintaining the energy density W of the beam due to a proportional change in the amplitude of the discharge current (the smaller the bank capacitance, the greater the discharge current).

Studies performed by scanning electron microscopy have shown that, regardless of the mode of irradiation, a polycrystalline structure is formed in the surface layer of steel, a characteristic image of which is shown in Fig. 1, a. The structure of high-speed cellular crystallization is revealed in the volume of grains (Fig. 1, b). It was established that the average sizes of grains and cells depend on the mode of irradiation and vary within (3.5 - 10) μ m and (230-940) nm, respectively.

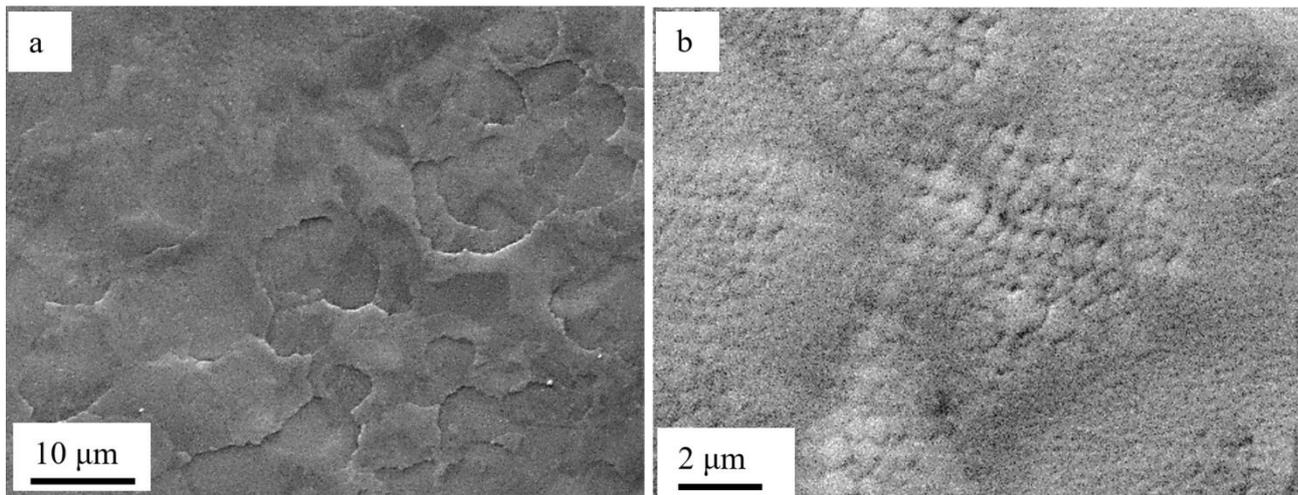


Fig. 1. Electron-microscopic image of the surface structure of samples of steel SUS321 irradiated with a pulsed electron beam.

The results of mechanical and tribological tests showed that the microhardness of the surface layer of steel reaches maximum values when irradiation is occurs by the pulsed electron beam using the battery capacity of $C = 3$ μ F and exceeds the microhardness of steel in the initial state by ≈ 1.1 times, however, the wear resistance of the material in this case increases by ≈ 2.2 times.

* The work was partially supported by the Ministry of Education and Science of the Russian Federation and by the Siberian Branch of the Russian Academy of Sciences (project SB RAS No. 10).

LOW ENERGY IMPLANTATION OF NITROGEN IONS BY EXTENDED BEAM WITH A BALLISTIC FOCUSING IN A STAINLESS STEEL*

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The results of experiments on the low-energy implantation of nitrogen ions in stainless steel 12X18H10T (analogue of AISI 321) are presented. The processing with a pulsed extended beam of nitrogen ions, obtained using a ballistic focusing system was carried out. The source of ions was the nitrogen plasma of a non-self-sustained gas arc discharge with a thermionic cathode [1]. The formation of a pickling hole on the sample surface as a result of ion etching is shown. The profile of this hole is depends on the parameters of ion beam action. An increasing of surface hardness up to 4 times when processing stainless steel in such system is shown. The hardness increasing is due to the formation of a modified layer in the surface occurs. The layer contains nitrides of iron and alloying elements and its thickness reaches 50 microns. The formation of a modified layer across the processed samples surface, including outside the ion beam focusing region, is shown.

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TRIGGERED GAS SWITCHES WITH A SHARPLY NON-UNIFORM ELECTRIC FIELD AT THE ELECTRODE WITH NEGATIVE POTENTIAL *

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The paper presents the design and results of studies for two triggered high-pressure gas switches intended for pulse-periodic capacitive storages with the stored energy level of about 100–1000 J. The operating voltage of the first switch is over 200 kV, for the second switch is up to 50 kV. A feature for both switches is a configuration with a sharply non-uniform field at the electrode with negative potential. In the charging process of a capacitive energy storage, a corona-streamer discharge occurs in the switch, the current of which increases with increasing voltage. Cathode discharge plasma shields a sharp cathode edge and local areas of inhomogeneity with increased emission and also gives a uniform flow of initiating electrons into the gap of the switch. As a result, conditions in the discharge gap vary slightly from pulse to pulse and stabilization of the breakdown voltage is achieved. The triggered switches do not impose strict requirements on the working pressure and has a steady trigger when the air pressure changes by more than 1 atm.

* This work was supported by Russian Foundation for Basic Research (Project No. 19-08-00115-a)

PROTECTIVE AND ANTI-REFLECTION SILICON-CARBON FILMS FOR IR OPTIC

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1. Introduction

When creating equipment operating in the IR wavelength region, films based on carbon (a-C, a-C:H) are of great interest. They can provide not only high transparency of IR optics, but also protective properties [1, 2]. However, high residual stresses in these films lead to their poor adhesion and the impossibility of forming thick films ($>1 \mu\text{m}$) [3]. a-C:H films doped with silicon and oxygen (a-C:H:SiO_x) have low internal stresses, and possess good physico-mechanical and tribological properties [4]. In this paper, we have investigated structural, mechanical and optical properties of a-C:H:SiO_x films deposited on Si by plasma assisted chemical vapor deposition (PACVD) method and their thermal stability.

2. Results

The a-C:H:SiO_x films were deposited by the PACVD method using a bipolar substrate bias in a mixture of Ar and polyphenylmethylsiloxane vapors. The substrates were single crystal silicon plates with a transparency of about 50% in the IR region ($\lambda = 2\text{-}14 \mu\text{m}$). The transparency of the samples in the IR region was measured using a Nicolet 5700 FTIR spectrometer. To determine the hardness of films, a Nanotest 600 nanoindenter was used. Raman spectroscopy was used to analyze changes in the structure of the films, in particular, the content of sp³ and sp²-hybridized carbon atoms. The morphology of the films surface was investigated by atomic force microscopy. Properties and the structural behavior of a-C:H:SiO_x films were examined after annealing at various temperatures up to 500 °C.

It is shown that deposition of films on both sides of Si substrate provides an increase in the integral transparency of the sample from 50 to 87% in the in the 3-5 μm spectral region. Wherein, the hardness of the film is about 20 GPa, the modulus of elasticity is 152 GPa, the plasticity index H/E is 0.13, and the resistance to plastic deformation H^3/E^2 is 352 MPa. It is shown that high-temperature annealing up to a temperature of 500 °C does not reduce the integral transparency of the films in the 3-5 μm spectral range, and the film hardness does not change significantly. Although, according to Raman spectroscopy, significant changes occur in the structure of the film after annealing: the location, intensity, width of the D and G peaks, and the I_D/I_G ratio change.

Acknowledgments

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MEASUREMENT OF THE SURFACE TEMPERATURE OF TiCuN COATING / A7 SUBSTRATE SYSTEM AT PULSED ELECTRON-BEAM TREATMENT*

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Surface modification of metal materials and products using pulsed electron-beam processing is widely used in various industries, aircraft and automotive, aerospace and medical technologies, etc. [1-3]. Recently, technologies for surface engineering of materials, combining the application of thin (hundreds of nanometers – few microns) coating films and subsequent liquid-phase mixing with the substrate material using a pulsed electron beam have been developed [3-4]. Thus, it is possible to create surface layers with desired physical and mechanical properties. To fully understand the processes taking place in the surface layer under pulsed thermal effects, it is necessary to know the values and rates of temperature change of the surface being modified. However, direct measurement of the surface temperature by the contact method at pulsed (tens-hundreds of microseconds) heating is not possible. Previously, only numerical methods for calculating thermal fields, which cannot fully take into account all the effects arising in the material, were used to describe such processes. At present, high-speed infrared pyrometers have appeared for contactless determination of the surface temperature of materials with a wide range of temperature measurements (from hundreds to several thousand degrees).

In this work A7 aluminum alloy (substrate)/TiCuN (coating) system was used as samples of 15x15x5 mm size with the coating thickness varying from 1 to 6 μm . The surface of the samples was subjected to a pulsed electron-beam treatment on the “SOLO” vacuum setup [5] (included in complex of unique installations “UNIKUUM”), which is based on a pulsed electron source with a plasma cathode. During irradiation, the following parameters were varied: the energy density per pulse was 5–10 J/cm^2 , the pulse duration was 100–200 μs . Such a range of parameters was chosen on the basis of preliminary experiments in order to not destroy the coating (TiCuN begins to deteriorate due to the loss of nitrogen at temperatures above 800 $^{\circ}\text{C}$). The maximum temperature in the experiments did not exceed 1000 $^{\circ}\text{C}$, which is significantly higher than the melting point of the aluminum substrate (650 $^{\circ}\text{C}$). To measure the surface temperature of the samples during irradiation, a high-speed infrared pyrometer “Kleiber KGA 740-LO” with a measurement range of 300-2300 $^{\circ}\text{C}$ was used. The signal from the pyrometer was recorded as an oscillogram of the samples surface temperature dependence on time.

An analysis of the results showed that with increasing coating thickness, *ceteris paribus*, the maximum temperature reached on the surface of the samples is increases. This is explained by the thermal conductivity of the TiCuN coating and the corresponding decrease in the effect of the aluminum substrate on the heat removal from the surface of the samples. With a decrease in the power density in a pulse (with an increase in the pulse duration at the same energy density in the pulse), the effect of the coating thickness becomes less significant, since the heating rate becomes comparable with the heat removal rate due to the thermal conductivity of the coating, while the maximum temperature remains almost unchanged during the pulse (thermodynamic equilibrium is reached on the surface).

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ADDITIVE MANUFACTURED VT6 TITANIUM ALLOY SURFACE MODIFICATION BY ELECTRON-ION-PLASMA METHODS*

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Purposeful design of the surface of materials and products using modern electron-ion-plasma methods is an important task, since its solution allows to create functional layers and coatings that significantly increase the physicomechanical and performance characteristics of machine parts and mechanisms, as well as a variety of tools, increasing their service life in extreme conditions of operation and thus leading to energy and resource saving [1-4]. A new step in the development of integrated electron-ion-plasma technology, which determines its undoubted scientific novelty and practical significance, is the combination of its constituent processes in a single vacuum cycle: (1) the formation of a gradient multiphase surface layer by introducing elements (nitrogen, carbon, oxygen, etc.), (2) synthesis of thin metal films or superhard nanostructured coatings based on nitrides (carbides, borides, etc.) of refractory metals (TiCuN, ZrMoN, TiSiN, etc.) by ion-plasma methods and (3) formation of surface alloys when mixing a film/substrate system with predicted functional properties or fusing these coatings into the substrate with a high-intensity pulsed electron beam to increase adhesion forces of the coating/substrate system [5].

Works on formation on a surface of the samples of VT6 titanium alloy made by method of additive manufacturing, the film/substrate system with the subsequent mixing by means of a pulse electron beam are carried out. Zirconium film of 2 μm thick was deposited by method of plasma assisted arc in the presence of gas plasma of arc discharge generated by «PINK» plasma generator [6].

Superficial alloying of a substrate with material of the deposited film and finishing processing of a surface was carried out as a result of pulse melting of the film/substrate system by a high-intensity pulsed electron beam.

Mechanical properties of a surface of the modified samples (roughness, microhardness, structure, wear resistance) made by means of additive technologies and irradiated in selected optimum modes in comparison with initial samples are defined. Mechanical tests of samples on stretching are carried out («Instron», model 3369). Regularities of change of structure and mechanical properties of a surface depending on the mode of processing of samples are revealed.

It is shown that alloying of the samples of VT6 titanium alloy made by method of additive manufacturing in a single vacuum cycle on «COMPLEX» installation [5] (included in complex of unique installations «UNIKUUM») by deposition of a thin film of zirconium and the subsequent liquid-phase mixing by means of pulse electron beam treatment allows considerably to reduce roughness and porosity of a surface layer and to increase its mechanical properties. In the optimum modes of processing the increase in microhardness at $\approx 40\%$ in comparison with initial samples has been received. Values of roughness, tensile strength and wear resistance correspond to initial material.

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NANOSTRUCTURING OF THE T31507 STEEL SURFACE BY VANADIUM BORIDES UNDER THE INFLUENCE ELECTRON BEAMS IN A VACUUM

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The work discusses the features of surface hardening of T31507 steel under the influence of powerful electron beams, on account of quenching and the formation of layers based on vanadium borides. The most important characteristic of borides, determining their practical use, is their high hardness associated with the directional nature and energy strength of interatomic bonds. The experiments used a vacuum electron beam installation "SOLO", created in IHCE SB RAS, which is based on an electron source with a plasma cathode based on a pulsed low-pressure arc discharge with grid stabilization of the cathode plasma boundary.

Thermodynamic calculations showed that boride V_3B_4 using the stoichiometric mixture V_2O_3 -B-C cannot be obtained due to the formation of iron borides Fe_2B , FeB (interaction with the metal base) and borides of alloying elements (CrB_2 , WB , MnB_2). The introduction of excess amounts of boron and carbon made it possible to choose the optimal compositions for the preparation of composite layers with the maximum yield of borides. To do this, vary the concentration of B in the mixture of 50-50 (steel – reaction mixture). Will find the maximum flow rate for maximum output V_3B_4 23 wt%.

Analysis of the thermodynamic calculations made it possible to determine the optimal conditions for the interaction of the V_2O_3 :B:C reaction mixture with the surface of carbon T31507 steel for forming the composite coating to a depth of 5-150 μm .

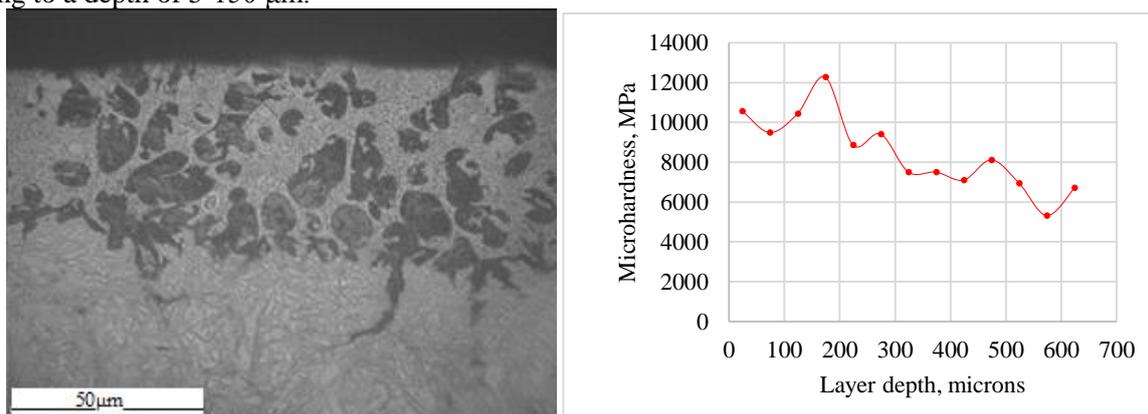


Fig. Structure and microhardness of V_3B_4 layers on T31507 steel ($P = 10^{-3}$ Pa)

After electron beam treatment of the T31507 steel, with boron-containing coatings applied on them, non-uniform layers are formed on the surface, 50-100 microns thick. The resulting layers have a specified thickness of almost the entire length. In fig. it can be seen that various regions are present in the layer, which allows one to conclude that these are particles of borides of alloying elements (tungsten, chromium, manganese, and vanadium). The layer is firmly held on a metal base.

In figure shows the measurement of the microhardness of the vanadium boride layers with a step of 30-50 microns. An uneven distribution of microhardness in thickness was found. However, in all the studied samples, a regular distribution of microhardness was observed depending on the layer thickness. Some very rare inclusions have $HV \approx 13000$ MPa and are located in the surface zones of the layer. Layers are characterized by the most complex disordered structure. The chaotic distribution of microhardness in thickness in boride V_3B_4 is explained by the fact that this boride is formed at a lower temperature, and, accordingly, there are very many impurities in the layer.

THERMOPHYSICAL MODEL OF ELECTRON BEAM BORIDING OF CARBON STEEL ST3.

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The work is aimed at developing a thermophysical model of electron-beam boriding of carbon steels.

One of the main tasks for creating a thermodynamic model is to determine the optimal synthesis conditions, in particular the formation temperature at different pressures in the working chamber. It is known that the temperature of formation of iron borides decreases to 800 K with a decrease in the total pressure to 10^{-3} Pa (figure 1). For the experiments, used electron-beam installation with axial gun for thermal cathodes. Reaction daubs of various stoichiometric compositions of $Fe_2O_3: 3B: 3C$, $Fe_2O_3: 2B: 3C$ were deposited on the surface of the sample. Power density of the electron beam $W = 5.7 \times 10^2$ W/mm², processing time 1-3 min. The experiments were carried out at a pressure of 10^{-3} Pa. Previously, a sequence of chemical transformations occurring in the synthesis of borides was established, namely, "oxides \rightarrow carbides \rightarrow lower borides \rightarrow higher borides". 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 [1].

To simulate the process of electron-beam processing, selected samples have the form shown in figure 2. The electron beam is first focused on the surface of the sample, and then converted into a raster with the help of an electron beam control unit and scanned across its diameter. The calculation of thermal fields was performed using the Maple 18 program. The physical model of the electron beam treatment process is determined by a number of parameters: the power of impact of the electron beam, the speed of processing the details, the time of exposure. Data on the size of the part, the depth of penetration of electrons into the sample are taken into account $S = 2,1 \times 10^{-12} \frac{U^2}{\rho}$, the depth of maximum energy deposition $h = 0,75 \times S$, as well as the thermophysical characteristics of a particular material.

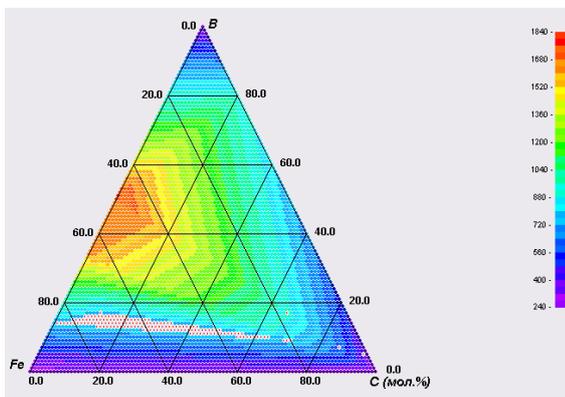


Fig.1. Isotherms in the Fe-B-C system at a pressure of 10^{-3} Pa.

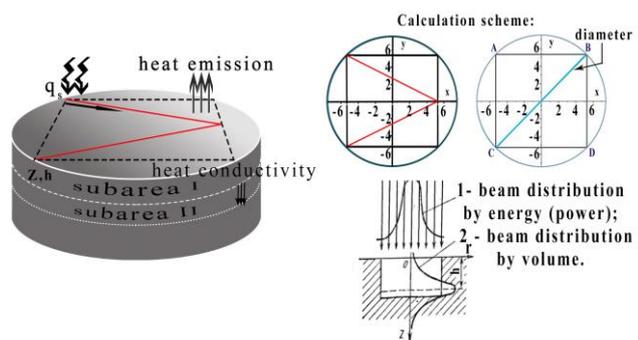


Fig.2 Scheme of energy input to the sample surface.

This paper presents a model for the formation of iron borides, analyzes the thermal processes and considers the phase equilibria of these structures. The strength characteristics of the obtained iron borides layer were investigated.

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PLASMACHEMICAL SYNTHESIS OF FULLERENES C60 AND C70 AT ATMOSPHERIC PRESSURE AND THE EFFECT OF FULLERENES ON THE HYDRATION OF PORTLAND CEMENT

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The article describes the results of the study of plasma chemical synthesis of fullerenes C60 and C70 at atmospheric pressure in high-frequency arc discharge and results of modification cement stone by fullerenes. Composition of carbon soot can be controlled by changing helium pressure. The yield of higher fullerenes increases with increasing helium pressure, while the yield of carbon nanotubes decreases. Elevated pressure reduces the concentration of carbon in the carbon vapor, which leads to increase in the yield of fullerene C60 and higher fullerenes C70.[1,5]

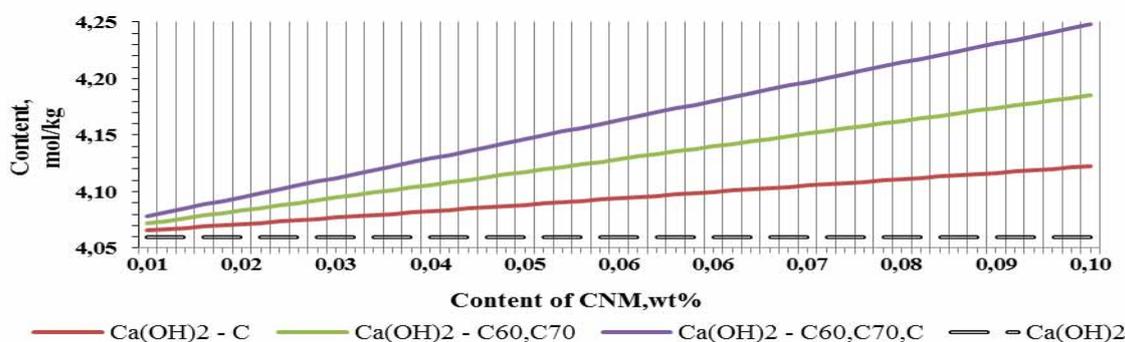


Fig. 1. . Calculated yield of phase Ca(OH)2 from content of CNM in system «cement – H2O-CNM»

Obtained carbon soot was used for modification cement stone for improved physical and mechanical properties. Change in phase composition, structure and properties of modifying cement stone were investigated. The effects of the CNM on the early hydration process of cement were studied using X-ray diffraction analysis and thermodynamic modeling. The CNM were found to accelerate the hydration reaction of the C3S in the cement. In particular, the CNM appeared to act as nucleating sites for the hydration products, with the CNM becoming rapidly coated with C-S-H. The addition of CNM improves the strength characteristics of cement stone because it reduces the porosity of cement stone and building materials based on it. Thermodynamic calculations (Fig.1) were performed in the Terra program [6]. The study of cement stone hydration and structure was performed using X-ray phase analysis and electron microscopic analysis [5]. Thermo-kinetic analysis [4] and thermodynamic modeling revealed a temperature increase of hydration with the introduction of CNM into the cement paste that shows the complete progress of the reactions. The dynamics of reduction of calcium silicate peaks characterizes the activation of the processes of hydration and binding of portlandite, which explains the increase in strength of the modified cement stone. The structure of the cement stone has a high density and uniformity in the introduction of CNM.

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STRUCTURAL FEATURES OF THE MAGNETRON SPUTTERED CUO/GDC FILMS FOR SOLID OXIDE FUEL CELL APPLICATION*

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Solid oxide fuel cells (SOFCs) are promising devices for efficient and environmentally friendly electricity generation. For operation on hydrogen, Ni-containing cermets are usually used as a SOFC anode, since they have a high catalytic activity. But if synthesis gas is used as fuel, Ni shows a high rate of degradation due to poisoning by impurities of sulfur or carbon [1]. Carbonization blocks catalytically active centers, changes the microstructure of the anode and, as a result, reduces the cell performance. In recent studies [2], Cu has been considered as a new material with good electronic conductivity instead of Ni in SOFC anodes. The aim of the present study is to investigate the microstructural characteristics of magnetron sputtered CuO/GDC layers and their changes after reduction in hydrogen atmosphere and heat treatment.

CuO/GDC thin films were formed by the method of reactive magnetron co-sputtering of Cu (99.995% purity) and Ce_{0.9}Gd_{0.1} targets (75 mm diameter). Sputtering was carried out at working pressure of 0.2 Pa (Ar and O₂ flow rates were 26 and 33 sccm, respectively). The discharge power of a Ce-Gd magnetron was 1 kW in all experiments. The discharge power of the Cu magnetron ranged from 100 to 700 W to control the volume content of CuO in the film.

The composition and microstructure of the CuO/Ce_{0.9}Gd_{0.1}O_{2-δ} (CuO/GDC) thin films were characterized after deposition, annealing in air and reducing atmosphere using energy dispersive X-Ray spectroscopy, scanning electron microscopy and X-ray diffractometry. The as-deposited film consists of cubic fluorite structures of GDC and Cu₂O and has a dense, homogeneous structure (Fig. 1,a). It was shown that, Cu segregation is observed in CuO/GDC films, after reduction in hydrogen at a temperature of 600°C, with the formation of Cu agglomerates on the surface (Fig. 1,b).

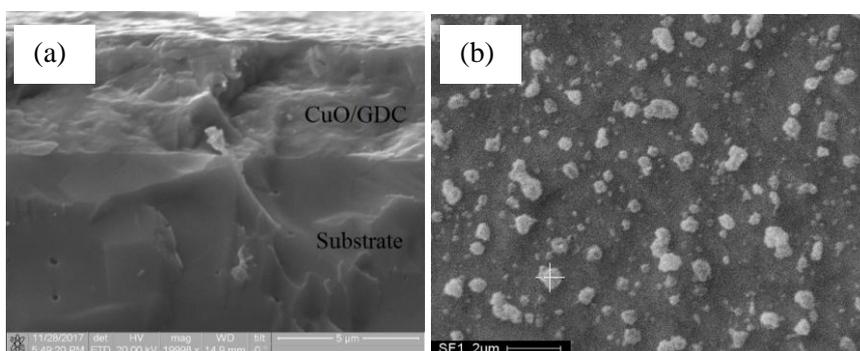


Fig. 1. The cross-section SEM image (a) of as-deposited CuO/GDC film on 10Sc1CeSZ substrate. Surface of Cu/GDC film after reduction in hydrogen at 600°C (b).

In order to prevent this process, the influence of pre-annealing of the as-deposited films in air atmosphere in the temperature range 1000–1200°C was studied. X-ray diffraction showed that after annealing at 1000°C, the film is saturated with oxygen and Cu₂O phase is transformed into CuO. It is shown that pre-annealing at a temperature of 1000°C does not prevent the agglomeration of Cu in a reducing atmosphere. As we have shown, the pre-annealing at 1200°C, which is successfully used for nickel-containing films, also cannot be used for magnetron sputtered copper-based films due to Cu evaporation. Thus, the problem of Cu agglomeration after heating in a hydrogen atmosphere still remained unresolved.

The catalytic activity of the magnetron sputtered Cu/GDC films will be determined at the next stage of research.

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HYBRID DC+HIPIMS MAGNETRON SPUTTERING DEPOSITION OF CU AND CUO FILMS*

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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) plasma density could be as high as 10^{12} cm^{-3} . However, a significant disadvantage of HIPIMS is the reduction in the films deposition rate compared to DC sputtering. Different ways have been offered to improve deposition rate, for example, pulse waveform modulation [1]. Another perspective approach is hybrid technology, known as hybrid DC + HIPIMS co-sputtering process [2].

The properties of Cu and CuO films deposited on glass and Si substrates in DC + HIPIMS mode of magnetron sputtering have been investigated. Hybrid mode (DC + HIPIMS) of the magnetron sputtering was realized at an average power of 0,5 kW, applied to a planar magnetron with a Cu target. The main parameters that changed during the experiments were: amplitude of pulsed discharge current, pulse frequency, average power of DC-discharge in hybrid mode DC + HIPIMS. In the latter case the total average discharge power and the duration of high-current pulses are remained unchanged - 500 W and 100 μs , respectively. The amplitude of the current pulses varied from 20 to 300 A, and the pulse repetition rate varied from 100 Hz to 500 Hz. Figure 1 shows the current-voltage characteristics of a magnetron discharge in various modes at an argon pressure of 0.3 Pa.

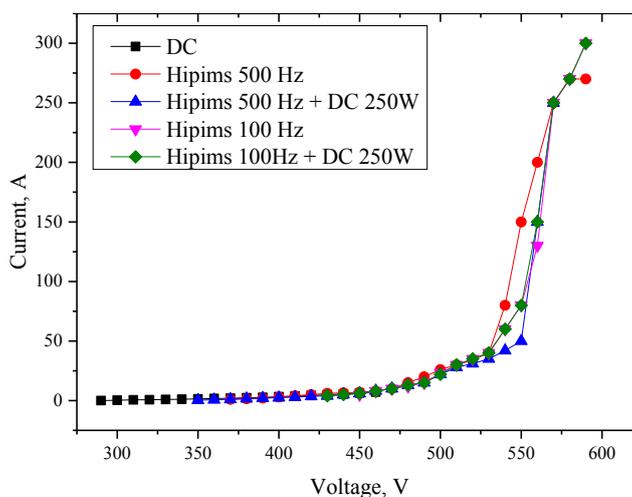


Fig. 1 Volt-ampere characteristics of a magnetron discharge in various modes at an argon pressure of 0.3 Pa.

Thin Cu films were obtained and their properties (resistivity, surface morphology, hardness) and the deposition rate were measured depending on the deposition regimes. It was established that the deposition rate in the hybrid mode (DC + HIPIMS almost coincides with the deposition rate in the DC mode (5.8 and 6 $\mu\text{m}/\text{hour}$, respectively) and more than twice the deposition rate in the HIPIMS mode (2.6 $\mu\text{m}/\text{hour}$) at the same average power (500W).

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INFLUENCE OF VACUUM-ARC AND MAGNETRON DISCHARGES COMBUSTION MODES ON STRUCTURE AND PHASE COMPOSITION OF THE FORMED COMPOSITE TiN-Cu COATINGS*

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The paper studies the formation conditions, structure and phase composition of the TiN-Cu composite layers which received at different values of the arc current and the magnetron discharge current. The composite layers deposition was carried out in a modernized installation equipped a vacuum-arc evaporator and a planar magnetron [1]. Main plasma parameters generated at low-pressure vacuum-arc and magnetron discharges were studied with the help of a single Langmuir probe. It is shown how much the plasma concentration and the ion current density on the substrate changes depending on the change in the currents a magnetron discharge and vacuum arc evaporator. For example, at a constant pressure $P = 8 \cdot 10^{-3}$ Torr with a discharge current of magnetron increase from 0.2 A to 0.6 A concentration of plasma grows from $2.6 \cdot 10^{12} \text{cm}^{-3}$ to $1.1 \cdot 10^{13} \text{cm}^{-3}$.

Some results on the composite coatings TiN-Cu synthesis at different values of arc current and magnetron discharge current have been received. The layers' thickness was from 2-3 μm to 5-7 μm depending on the duration of the depositing time.

X-ray phase analysis is performed and according to this analysis the samples contained Ti_2N , TiN phases with different crystal cell and volume fraction. Besides the reflexes of copper with the intensity of about 1-2% there are fixed (Fig.1). With the help of a METAM PB-22 microscope the structure of a surface TiN and TiN-Cu coatings was investigated.

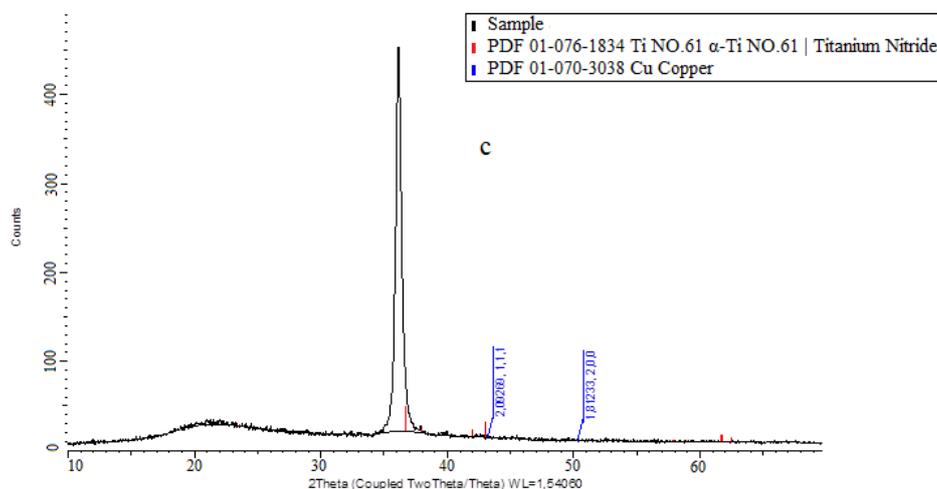


Fig. 1. XRD patterns for coating TiN-Cu (80A, 1,1A, $8 \cdot 10^{-3}$ Torr)

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THE FORMATION OF A PLASMA ANODE IN A HIGH-CURRENT ELECTRON GUN WITH THE USE OF A HYBRID DISCHARGE

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The plasma anode is one of the key elements of high-current electron guns used to generate low-energy (up to 40 keV) beams, which are widely used for surface modification of materials [1, 2]. To improve the beam homogeneity, it is preferable to create a plasma anode with an increased ion concentration in the peripheral region. For this purpose, in [3], it was proposed to use a hybrid discharge that combines a high-current reflective (Penning) discharge (HCRD) with vacuum arcs initiated by a dielectric surface flashover.

In this work, we investigated the spatial structure of the glow and the temporal dynamics of such a discharge with an increased (relatively to [3]) anode voltage, and also carried out thermal imaging measurements of the energy density distribution over the beam section.

It was found that in the range of 0.1–1 mTorr, the time delay of the HCRD ignition does not exceed 15 μs and weakly depends on the working gas (argon) pressure and depends somewhat more on the amplitude of the voltage pulse applied to the anode (Fig. 1). In the range of anode voltages of 6–10 kV, the voltage and current discharge waveforms are very stable, as well as the ignition of vacuum arcs. Vacuum arcs provide a luminescence structure like a ring (Fig. 2), which makes it possible to improve the homogeneity of the energy density distribution over the beam cross section.

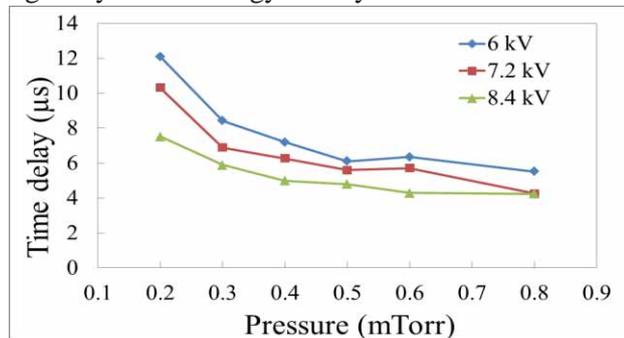


Fig. 1. Dependence of the time delay of the ignition HCRD on the argon pressure at different values of the anode voltage amplitude.

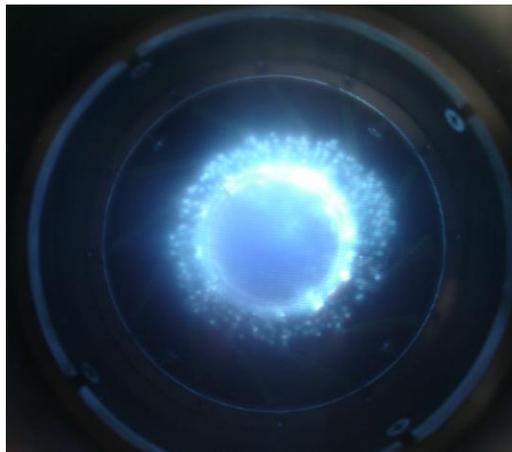


Fig. 2. The time-integrated glow of the discharge plasma. Anode voltage – 7.2 kV, argon pressure – 0.5 mTorr.

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MODELING OF THE SYNTHESIS OF ‘CORE-SHELL’ COMPOSITE PARTICLES BASED ON SEGREGATED OXIDATION OF TITANIUM AND SILICON TETRACHLORIDES IN FLOW-TYPE PLASMACHEMICAL REACTOR*

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Synthesis of nanocomposite powders of oxide ceramics is one of the most promising trends in up-to-date technologies. These powders belong to new class materials with controlled physico-chemical properties ranged depending on their applications. In particular, nanosized titania (TiO₂) powders are being widely used in industry. In many applications it is required to extremely inhibit photocatalytic activity of TiO₂ nanoparticles. For instance, it is the case when TiO₂ pigments are used to modify physico-chemical properties of paint films, plastic materials, in paper manufacturing as well as that of sun blocking agents. In order to achieve this it is necessary to decrease an area of photo-active free surface of titania pigment as much as possible, with titania optical properties being unchanged. This requirement might be met by the synthesis of nanocomposite ‘TiO₂ core - SiO₂ shell’ particles. In these powders the greater thickness of silica amorphous layer and the lesser its microporosity, the lower photoactivity of the composite powder [1]. In numerical study [2] first time the simulation of one-step synthesis of composite TiO₂/SiO₂ nanoparticles in the working zone of plasmachemical reactor has been performed providing segregated oxidation of titanium and silicon tetrachlorides. The range of varying the flow rate of mixture of air and silicon tetrachloride allowed a decrease of reacting flow temperatures below 1200 K at which a formation of chlorosiloxanes (SiO_xCl_y)_n is possible. In addition, in the model a minimal shell thickness is not restricted.

In this work the results of simulation of aforementioned one-step synthesis of composite TiO₂/SiO₂ nanoparticles have been presented taking into consideration above mentioned aspects.

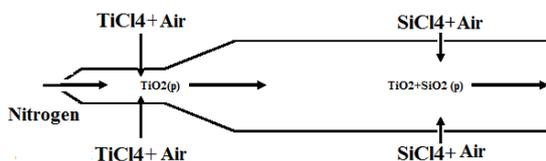


Fig. 1. Diagram of the working zone of the flow-type reactor. N₂ mass flow rate – 1 g/s. Mass average temperature of nitrogen plasma jet – 4500 K. Air+TiCl₄ mixture flow rate – 2.5 g/s, air+SiCl₄ mixture flow rate – 1.0 g/s. The model accounts for a synthesis of TiO₂ vapors, followed by formation and growth of titania particles as result of coagulation process.

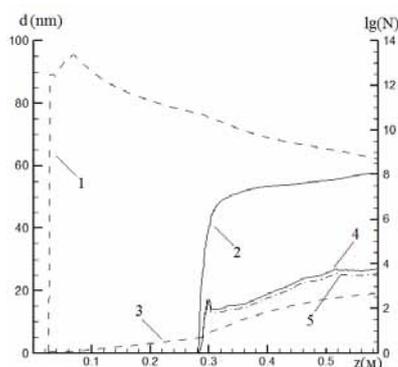


Fig. 2. Mass average particle diameter (nm) and logarithm of the particle total number distributions along the reactor: 1 – logarithm of the total number of TiO₂ particles; 2 – logarithm of the total number of composite particles; 3 – diameter of TiO₂ particles; 4 – diameter of composite particles; 5 – diameter of TiO₂ core of composite particles.

The results of the numerical investigation show that 50% of particles at the reactor exit are TiO₂/SiO₂ composite ones. The shell thickness is approximately equal to 1.5 nm.

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STUDY OF OZONE PRODUCTION IN A DIELECTRIC BARRIER DISCHARGE IN OXYGEN-CONTAINING MIXTURES FOR PLASMA ASSISTED COMBUSTION

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Active studies of plasma assisted ignition and combustion are currently under way [1, 2], because low-temperature non-equilibrium plasma is an effective tool for accelerating chemical processes associated with combustion. A dielectric barrier discharge (DBD) is of particular interest for combustion applications due to relative simplicity of its technical implementation and the ability to be easily integrated into various gas ow configurations [3].

The paper presents the results of experiments on the generation of ozone by a barrier discharge in oxygen and in a mixture of oxygen and methane, as well as the results of numerical simulation. For various experimental configurations, the dependences of $[O_3]$ on the electric power deposited in the discharge are obtained. In addition, the work presents the results of experiments on the combustion of fuel-air mixtures of different degrees of enrichment.

The simulation of the barrier discharge is performed by simulating a sequence of discharge pulses with a given repetition rate. Analysis of the simulation results showed that the main mechanism leading to a decrease in the ozone concentration when methane is added to oxygen is the dissociation of methane in the discharge, which leads to the formation of hydrogen atoms, triggering the chain mechanism of ozone destruction.

The simulation results are in qualitative agreement with experimental data.

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THE STUDY ON PULSED ELECTRON BEAM ENERGY DISSIPATION IN GAS COMPOSITIONS OF INCREASED PRESSURE IN THE PRESENCE OF A CONDENSED PHASE (WATER, ASH)*

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The urgency of the project is justified by serious environmental problems of the environment (cleaning of flue gases) both in Russia and abroad. Electronic continuous accelerators (Indianapolis, USA and Karlsruhe, Germany) are currently used to purify flue gases. The initiation of plasma-chemical processes by a pulsed electron beam is one of the actively developing methods of activating chemical processes. The use of pulsed electron beams ensures the formation of plasma with a high degree of nonequilibrium in the ion and electron temperatures, which causes a number of advantageous features of this process when used in various industrial processes. The transfer of industrial processes to a new level of energy and resource efficiency is a modern trend with scientific and economic validity. Reduction of unproductive energy losses for heating nodes, aggregates, binders by combining reaction and plasma volumes will lead to higher productivity and economic efficiency of production. The use of nonequilibrium, fast-flowing processes in plasma will significantly increase the speed of chemical processes, and, therefore, reduce costs.

However, one of the important factors restraining the development of flue gas cleaning with the use of pulsed electron accelerators is the lack of an adequate physical model based on experimental data of the processes occurring during the interaction of pulsed electron beams, not only with model objects in the condensed and gas phase, but also with objects with a complex chemical composition, which are basic in technological processes. The complexity of the development of physical models is complicated by the nonlinear nature of the processes of energy absorption carried by beams, the formation of charged and excited particles, chemical reactions in the interaction zone, secondary radiation, and a number of other phenomena accompanying the interaction of pulsed energy flows with matter in the condensed and gas phases. A significant role in solving this problem is assigned to experimental research, experimental data both from the point of view of providing results for the formation of a model and its testing, and from the point of view of a quantitative description of the processes accompanying the development of specific technological processes.

In this work, the process of energy dissipation of a pulsed electron beam in gas compositions in the presence of a condensed phase will be investigated. The main components of flue gases (N_2 , CO_2 , O_2) were chosen as gas compositions. Water, ash were chosen as a condensed phase. These substances are either components of flue gases, or initial reagents or products of plasma-chemical reactions of the purification process using pulsed electron beams.

The TEA-500 accelerator and a drift volume were used in studies of the process of propagation of pulsed electron beams in gas compositions of increased pressure in the presence of a condensed phase (water, ash). The help of reverse current shunts, the reverse current and the current of the electron beam reaching the end flange of the drift tube determined.

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ELECTROSPARK METHOD OF OBTAINING NANOPOWDERS*

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The development of energy-saving, environmentally-friendly methods for obtaining nanopowders of various substances is an urgent issue of modern science. This is due both to the practical need for the creation of nanomaterials, which are widely used nowadays due to their uniqueness, and the fundamental need to understand the processes that occur in the preparation and application of nanoparticles by various methods [1-2].

In work the electrospark method is used to synthesize metallic nanocomposite. The following components were included: an electrode system; movement system; system for measuring processing parameters (oscillograph and current sensor, high voltage voltage divider, manovacuum meter); source of current pulses; vacuum system (vacuum pump, gas cylinders with working gas, gas routes, gas cranes). The peculiarities of the installation are the application of the power supply circuit from two generators, working on one interelectrode gap. The generator consists of three main units: low-voltage part, high-voltage part, control system. The high-voltage part of the generator is designed to form the initial spark channel. The low-voltage part of the generator serves to transfer the energy of the capacitor to the spark channel. The generator control system is designed to synchronize the high-voltage and low-voltage parts of the generator. The main parameters of the generator: a) high-voltage part: pulse duration 1 μs , pulse amplitude 18 kV, pulse energy 0.01 J; b) low-voltage part: duration of low-voltage pulse 5..100 μs , stored energy 0.1..0.6 J, frequency 0.1..5 Hz.

The essence of the electric spark method of obtaining nanoparticles is as follows: a metal plate (copper, zinc, iron), which is an electrode, will be located in the chamber. After the preliminary pumping, the gas cuvette is filled with gas (argon, nitrogen, air), depending on the type of compounds synthesized. At the end of this, the pulse generator is started, simultaneously with it the system of movement of the metal plate automatically turns on. The process of nanoparticle production begins. At the end of this process, the generator switches off automatically, and the movement system returns to the starting point. The method for obtaining nanoparticles is based on the use of the energy of an electric spark discharge, formed between the electrode and the target. When a voltage pulse is applied between the electrode and the target surface, a plasma channel of a spark breakdown is formed with an initial diameter $RK \sim 0.1$ mm. The current flowing through the channel heats it, the pressure in the channel rises, the channel expands. The plasma temperature reaches values of $3.8 \cdot 10^4$ °K, the energy flux density in this case is 10^6 - 10^9 J/m². As a result of the action of a concentrated energy flux on the target, a rapid local overheating of the surface leads to sublimation of the material. Under the influence of gas dynamic forces, the target material is removed from the discharge region where the nanopowder is condensed and formed, and due to the special design of the electrode system, the formed nanopowder is deposited in a special trap. As a result, the resulting nanopowder will obtain a composition identical to that of the target, or an oxide corresponding to the material of the electrode. Sedlating materials will be used as targets for the research: steel (st3), copper, zinc. The use of an electrode tool with an erosion coefficient smaller than that of the target will allow synthesizing a nanomaterial that has the chemical composition of both electrodes used. The energy in the plasma channel is sufficient for the plasmochemical process to proceed, as a result of which it is possible to form composite nanomaterials with a solid solution. An additional regulator of the properties of synthesized nanoparticles can be the composition of the gas phase used in the production process. The properties of the resulting nanomaterials evaluated by IR analysis, transmission electron microscopy, X-ray dif-fraction analysis, BET surface area analysis, TG/DSC/DTA thermoanalysis.

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TEMPORAL CHANGES OF THE IR SPECTRA OF HEAVY WATER AFTER ITS TREATMENT BY DIFFUSE DISCHARGE AND AFTER IRRADIATION BY A NANOSECOND DURATION STREAM OF ELECTRONS*

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Investigations of the absorption spectra of heavy water treated by a diffuse discharge preionized by fast electrons with a short rise-time of voltage pulse and after irradiation by a nanosecond duration stream of electrons are presented in this report. With repeated irradiation of water, a change in the absorption spectrum of the substance was recorded. Analysis of the absorption spectra of water in the IR range showed the difference between the absorption spectra of irradiated and non-irradiated water and the change in the spectrum after irradiation over time.

* The work is performed in the framework of the State task for HCEI SB RAS, project #9.5.2.

ATMOSPHERIC PRESSURE DIFFUSE DISCHARGE TREATMENT OF AQUEOUS SOLUTION OF METHYLENUM COERULEUM*

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Recently, various methods of degradation [1] of different type of organic pollutants in waste gas and water have been studied. One of the possible way for water treatment is direct UV-photolysis, as well as in combination with a variety of chemically active species (O, H, OH, H₂O₂) [2]. In [3] was shown, that methanol concentration in water-methanol solution decreased 23 times under the action of UV-radiation with wavelength of 222 nm and presence of nitric acid. However, in a coloured wastewater, UV-light cannot penetrate deep into the water. Thus the decolorization is the most significant problem for the methods based on UV-photolysis. Since high voltage discharges generated in ambient air above the solution surface produce various oxidative species, they may be utilized to degrade organic dye molecules. In this study, we used runaway electrons preionized diffuse discharge (REP DD) where the tip high voltage electrode was placed above the surface of aqueous solution of *methylene coeruleum* poured into a quartz cuvette. The cuvette was set up on the grounded plane electrode, the distance between the solution surface and the cathode was 8 mm. REP DD was formed by NPG-18 3500N generator produced negative voltage pulses with amplitude up to 18 kV in an incident wave and pulse repetition rate of 100 Hz.

Figure 1 shows the transmittance spectra of aqueous solution of *methylene coeruleum* before and after REP DD plasma treatment during 10 and 20 minutes. As can be seen, the transmittance changed a lot in the visible spectral range after 20 minutes of exposure, but still remained significant in the UV range. It reveals that products of chemical reaction between oxidized and dye-molecules are smaller fragments of dye-molecules, and it takes longer time to completely destroy them into inorganics compounds.

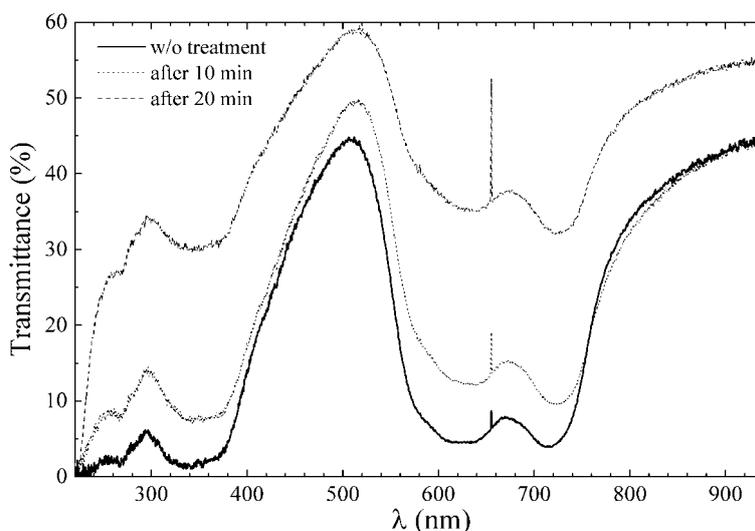


Fig. 1. UV-transmission spectra of aqueous solution of *methylene coeruleum* before and after diffuse discharge treatment during 10 and 20 minutes.

Thus, experiments have shown that REP DD plasma treatment in ambient air may be used for decolorization of wastewater. Utilizing this method in combination with photo-dissociation process may be a promising wastewater treating technology in the textile industry.

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* This work is performed in the framework of the State task for HCEI SB RAS, project #13.1.4.

LASER-PLASMA SYNTHESIS OF DIAMOND FILMS*

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The operation of a setup based on a previously created microwave module [1, 2] and the newly developed quasi-cylindrical cavity in the TM_{012} -mode is considered (Fig. 1). Unlike the traditionally used scheme of cylindrical cavity for microwave plasmatrons, when the cavity is divided by a quartz partition into a working chamber where plasma is formed and a microwave input chamber to prevent breakdown with higher gas pressure, our solution in the form of a quasi-cylinder allows laser beam input. A laser beam injected through an evanescent waveguide passes through a not very dense microwave plasma, forming a spot of near-surface laser plasma at the plasmatron output. The microwave module allows one to initiate microwave plasma synchronously with laser radiation pulses (1-5 μ s, 30-150 kHz) with an adjustable phase (delay) and provides a basic background, reducing the ignition threshold and increasing the spot area of the laser plasma. Preliminary experiments have shown the operation of the microwave cavity and a possibility of synthesizing a diamond-like film on a molybdenum substrate (Fig. 2).

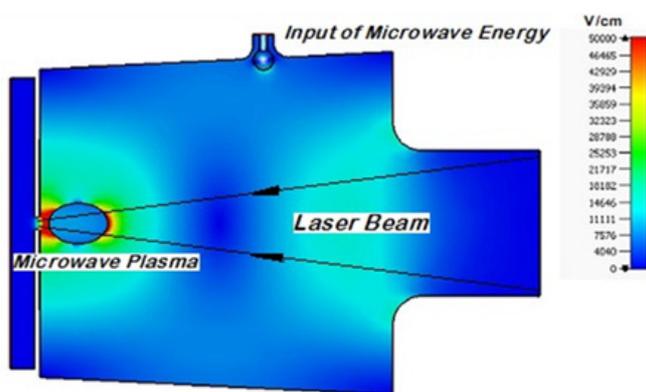


Fig. 1. Calculation of electric field configuration in a quasi-cylindrical cavity, TM_{012} -mode, copper, $f = 2.47$ GHz, $P = 5$ kW. Plasma simulation - graphite, $\sigma = 10^{-4}$ Cm/M, $\epsilon = 12$, $E_{max} = 75$ kV/cm.

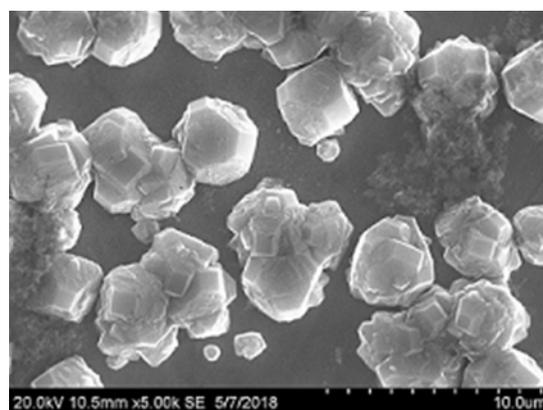


Fig. 2. Results of synthesis of a diamond-like film on a molybdenum substrate in an atmosphere of gases: CH_4 , H_2 , Ar.

The results of experiments on the interaction of microwave plasma with a CO_2 laser beam will be presented. Experimental estimates of the effect of microwave plasma on the ignition thresholds, the surface spot configuration, and the bulk torch of laser plasma will be given.

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EFFECTS AFFECTING THE MORPHOLOGY OF MICROSPHERES OBTAINED IN THERMAL PLASMA FLOW*

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A technique of the introduction of spherical particles, solid and hollow, in the matrix of constructional materials and coatings is being rapidly developed both in applications and science [1]. Nano- and micro-scale hollow particles, in which gas distributes in the volume or concentrates in separate inclusions, can be synthesized by treating both precursors [2] and different types of powders having the developed bulk structure [3]. Heating due to melting of the condensed phase up to the formation of hollow particles is observed in both cases.

In our theoretical and experimental studies we used silicon dioxide (SiO_2) obtained from sifted silica sand at Tuganskoe deposit due to its high SiO_2 concentration ($98 \div 99.2$ wt.%). Sifted silica sand also contained such impurities as refractory oxides Al_2O_3 , Fe_2O_3 , CaO , MgO and TiO_2 in the amount of much less than 1%. The experimental plasma generator was used to treat the powder agglomeration based on sifted silica sand [4].

Figure 1 presents a photograph of the plasma treatment of the powder agglomeration at stationary operation conditions. Plasma treatment parameters included 250 A current, 110 V voltage, 0.4 g/s and 1.1 g/s flow rates respectively for carrier gas and plasma gas, and 2.2 kg/h powder discharge.

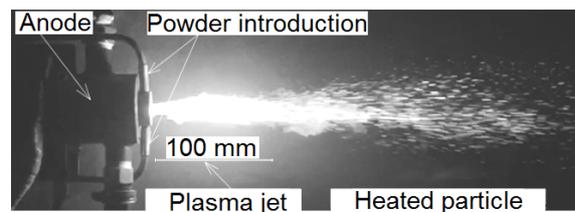


Fig. 1. Photograph of powder agglomeration plasma treatment.

Figure 2 presents scanning electron microscopy (SEM) observations of the individual powder particle and obtained microspheres. According to this figure, the agglomerated particle represents a coalescence of heterodisperse particles. The shape of the agglomerated particle is considered to be oval.

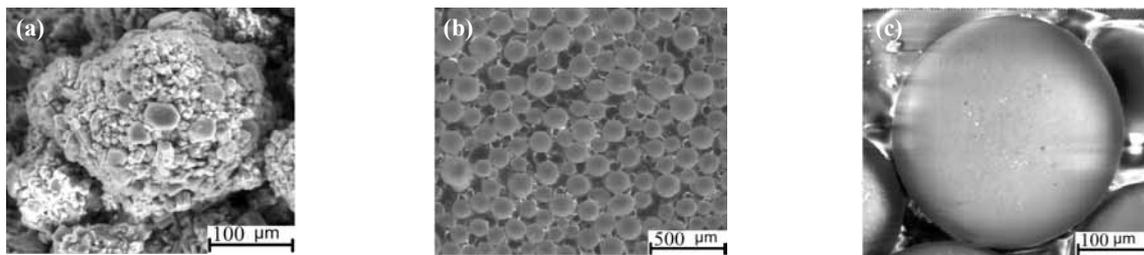


Fig. 2. SEM images of agglomerated particles: *a* – powder; *b*, *c* – microspheres.

The first important point is that the outer diameter of initial particles approaches to that of obtained hollow microspheres. This fact is supported by SEM images presented in Figure 2. As can be seen, the size of particles before and after the plasma treatment is almost similar. Micron-sized additional particles form on the surface of the hollow particles due to the disintegration of agglomerates during their travel in the dusty plasma flow.

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PHYSICAL AND TECHNICAL PROCESSES OF OBTAINING SILICATE MELTS AND MATERIALS BASED ON THEM IN LOW-TEMPERATURE PLASMA*

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Currently, it is not possible to obtain a silicate melt homogeneous in temperature and composition from raw materials with a melting point of more than 1500 °C using traditional technologies. The use of energy of low-temperature plasma makes it possible to increase the heating rate of silicate mixtures hundreds of times and to achieve consistently high temperatures of 3000–3500 °C upon receipt of silicate melts from raw materials with a melting point of 1500 °C and more. The development of methods for producing silicate melts using low-temperature plasma energy is of current interest.

Based on the results of theoretical calculations and the established modes of heat transfer when melting silicate raw materials using low-temperature plasma energy, the individual advantages of electroplasma installations were previously established in comparison with the silicate materials traditionally used in the processing of silicate materials. Based on the analysis of melting furnace designs, traditionally used in the preparation of silicate melts, years of experience in optimizing the parameters and design of plasma-chemical reactors, plasma variants for the production of chemically homogeneous silicate melts are proposed.

It was established experimentally that the operating conditions of an electroplasma installation make it possible to achieve specific heat fluxes of $1.0\text{--}2.6 \cdot 10^6 \text{ W/m}^2$, sufficient to produce a melt with the required temperature and viscosity.

As a result of the analysis of the work of the created plasma installations, taking into account their features and shortcomings, an experimental plasma-chemical reactor (RF Patent 2503628) was developed to produce silicate melts (Figure 1).

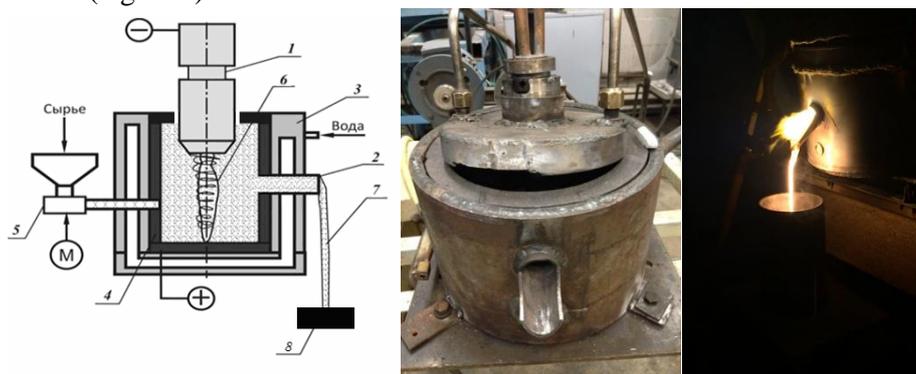


Fig. 1. Diagram of an experimental plasma setup for producing high-temperature silicate melts: 1 - plasma torch; 2 - drainage chute; 3 - water cooled melting furnace; 4 - graphite crucible; 5 - screw feeder; 6 - plasma arc; 7 - silicate melt; 8 - device for collecting the melt

Production of silicate melts in low-temperature plasma conditions proceeds in two stages: simultaneous melting of all phases of the mixture with the formation of a heterogeneous melt and homogenization of the melt under conditions of low viscosity due to overheating of the material above the melting temperature, in contrast to the melt production according to the traditional technology consisting of four stages: the formation of eutectic melts, the dissolution of the refractory components in eutectic melts, the production of a heterogeneous melt and its homogenization.

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STUDY OF SOME OPTICAL PROPERTIES OF TiO THIN FILMS PREPARED BY ION SPUTTERING

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The knowledge of optical properties of materials in IR range is important for different optoelectronic devices such as bolometers, transition edge sensors, etc. The optical and thermal properties of thin films are different from bulk materials [1]. Metal and metal oxides films are under consideration for the optoelectronic devices sensitive elements [2].

Titanium oxide (TiO) thin films (with a thickness of 500...600 nm) were prepared with ion (current $I = 100...150$ mA, voltage $U = 400...500$ V) sputtering in a mixed argon and oxygen medium (pressure $P = 2...3$ mPa). Layered silicate plates were used as substrates. Samples were characterized by FTIR (FSM 2201 spectrometer), XRR (with the procedure [3]) and laser profilometry. The films were identified as cubic titanium monoxide. The spectral (within the wavelength range of 1.8...8.0 μm) transmittance T_λ and the optical density D_λ (defined as $D_\lambda = -\ln(T_\lambda)$) of the films and the substrate are present in Fig. 1. The deposited films reduced T_λ values in 3...9 times.

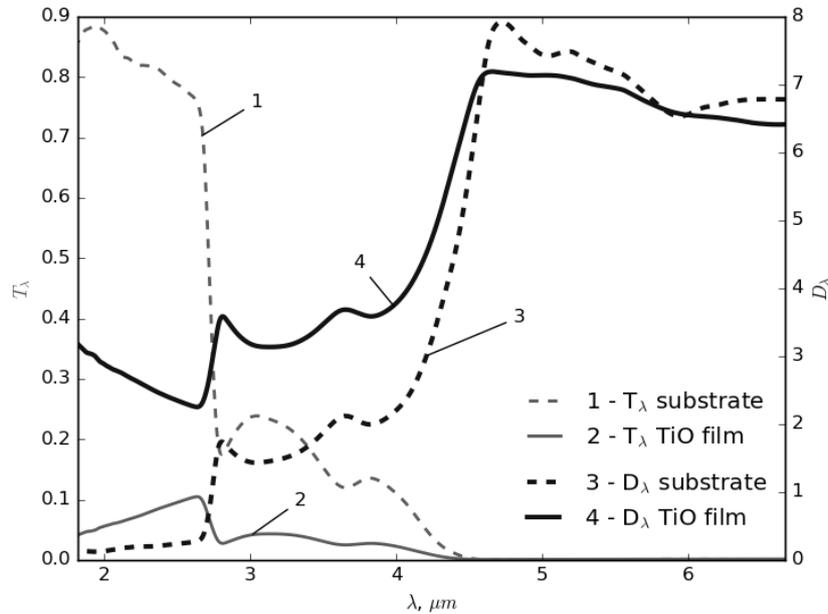


Fig. 1. Spectral transmittance T_λ and optical density D_λ .

Cubic TiO films on silica are under consideration for manufacturing of systems with a low level of IR transmission. These samples are prospective for IR absorbers and detectors.

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EFFECTS OF DIELECTRIC BARRIER DISCHARGE GENERATED PLASMA ON THE PHYSICO-CHEMICAL AND TECHNOLOGICAL PROPERTIES OF GEOMATERIALS*

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The feature of the mining and metallurgy development in Russia is the increase in volume of rebellious natural raw materials (geomaterials), with low content of valuable components and finely disseminated aggregates composed of minerals with similar process properties [1]. To date in Russia and other world countries the nontraditional (nonmechanical) processes for physicochemical, electrochemical, and pulsed energy effects have been intensively advanced with intent to improve disintegration and opening of finely dispersed mineral aggregates, to enhance the contrast of structural-chemical and flotation properties of rebellious mineral materials [1-3]. In recent years, dielectric barrier discharge (DBD), as a common approach to generate non-thermal plasma at atmospheric pressure, has been proved to improve the physical and chemical properties (namely, hydrophobicity and hydrophilicity surface) of polymers and other materials.

This work presents new experimental data on changes in the structural and chemical surface properties, water contact angle, microhardness and floatability of semiconducting sulfide minerals and quartz as a result of low-temperature plasmas action produced by dielectric barrier discharge at atmospheric pressure under standard air conditions. The gas (air) temperature in the discharge region of the DBD cell (reactor) remained near room temperature for 30-60 s and generally increased with increasing applied voltage amplitude in the range of 2–20 kV and driving frequency in the range of 2–20 kHz. We examined the variations of DBD properties with changing applied voltage and frequency. The rational condition we defined here as the operational conditions of voltage and repetition driving frequency under which the highest changes of minerals properties are achieved (20 kV, 16 kHz, 8 μ s voltage pulses, 100 ns pulse leading edge).

We performed our studies using samples of dry-crushed, pulverized and sieved to obtain a particle size in the range –100+38 μ m sulfide minerals (pyrite, arsenopyrite, sphalerite) and polished specimens 1×1×0.45 cm in size. Microhardness of minerals in the initial state and after DBD-plasma treatment of polished specimens estimated by Vickers' method (HV, MPa) at microhardness-meter PMT-3M; indenter load 50–100 g for sulfide minerals, and 200 g for quartz, loading time 10-15 s. The change in the hydrophilic-hydrophobic state of the quartz surface was evaluated by the change in the water contact angle (θ°) of the polished specimens surface by the sessile drop method using a microscope with the digital camera Moticam 2300 and the software suite Motic Image Plus 2.0 ML for image input and processing. The change in the flotation activity of minerals was measured by the recovery of sulfide mineral particles (pyrite, arsenopyrite, sphalerite) into the flotation froth. We examined the morphology of the minerals surface by analytical scanning electron microscopy (SEM–EDX) and confocal laser scanning microscopy (Keyence VK-9700).

Microstructural changes in the surface layer of minerals caused by DBD-plasma treatment resulted in an effective softening of their surface: the maximum relative change (decrease) in microhardness of quartz was observed after a $t_{\text{treat}} \sim 150$ s treatment and was 7% (from 1420 to 1321 MPa). Short plasma treatment of quartz ($t_{\text{treat}} = 10–30$ s) caused an increase in the hydrophobicity of the mineral surface: the contact angle of the quartz surface increased from 43.7° to 53°. With an increase in the treatment time ($t_{\text{treat}} = 30–150$ s) of mineral, there was a slight change in the contact angle from 44–47° to 48.4° in quartz. In selective flotation of pyrite, arsenopyrite, and sphalerite, the optimal parameters were found of the preliminary DBD-plasma treatment ($t_{\text{treat}} = 30–50$ s) and the reagent regime has been optimized for sphalerite recovery, resulting in a 6% (from 45 to 51%) higher recovery of sphalerite, 4% lower recovery of arsenopyrite (from 11 to 7%), and 22% lower recovery of pyrite (from 37 to 15%).

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MATCHING OF MASS SPECTRA OF IONS AND TERAHERTZ RADIATION OF LOW-INDUCTIVE VACUUM SPARK WITH LASER INITIATION

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One possible source of broadband THz radiation (TR) can be a vacuum spark with laser initiation. It is possible to propose several mechanisms for generating THz radiation from a vacuum spark differing in their origin: thermal (Planck) radiation, radiation of electrons in a magnetic field of a discharge current (cyclotron mechanism), and radiation caused by rapidly developing instabilities arising in the micropinch structure [1].

The goal of this work was to study the mass spectra of ions emitted from a plasma of a laser-initiated vacuum spark, and to compare the charge distribution of ions with the intensity of TR generated by the plasma.

The magnitude of the energy of the initiating discharge laser pulse $E_L \leq 20$ mJ with duration $\tau \leq 20$ ns. The laser radiation was focused on the anode of the discharge system. The energy released in the discharge is $E_D \approx 17$ J at voltage on the discharge capacitance $U_{AK} = 12.5$ kV. Electronic plasma temperature $T_e \leq 1$ keV.

In this work a time-of-flight mass spectrometer with a magnetic analyzer was used [2]. In the experiment, the mass spectra of plasma ions of a vacuum spark and the intensity of a terahertz signal were simultaneously recorded.

Figure 1 shows for comparison of two types mass spectra obtained for the mode with stable TR detection (amplitude at the detector $P_T = 500$ mV) and signal at the noise level (amplitude $P = 50$ mV). When results were processing, the concentration of electrons was calculated by the formula

$$N_e = \sum Z \cdot N_i(Z), \quad (1)$$

where $N_i(Z)$ – number of ions with charge Z .

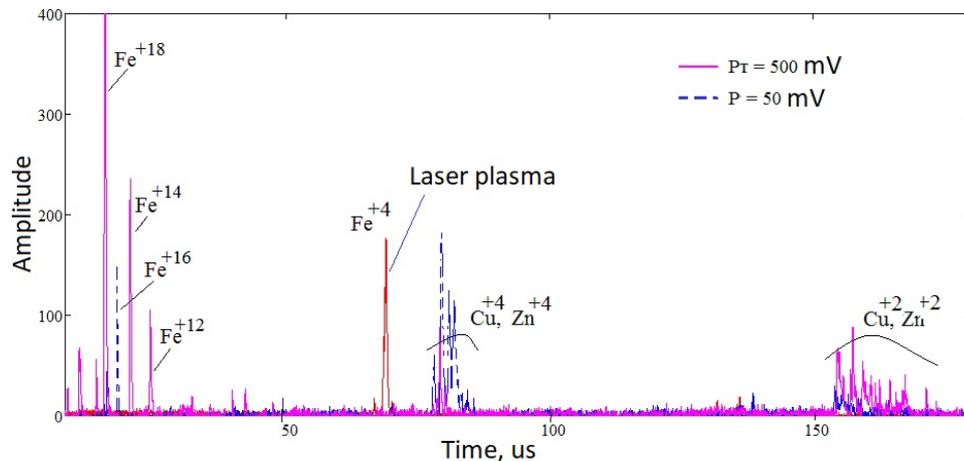


Fig.1. The averaged mass spectra at a fixed current of the magnetic analyzer: 1) continuous lines - the amplitude of the terahertz signal $P_T = 500$ mV; 2) the dotted line - the amplitude of the signal at the noise level is $P = 50$ mV.

The analysis of mass spectra showed that, generation of TR has threshold nature, non-linearly dependent on the concentration of Ne. From estimation of the electron concentration for modes with $P_T \approx 500$ mV and $P \approx 50$ mV, we can assume a power-law dependence of the intensity of terahertz radiation $P_T \sim (Ne)^x$, where $x \geq 2$.

Apparently, the condition for "effective" generating TR in the plasma vacuum spark is existing of high electron density in plasma in the presence of certain configured manner sufficiently strong magnetic fields.

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INTERACTION OF LOW TEMPERATURE PLASMA WITH FUSION MATERIALS

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The paper presents research results of candidate and structural materials of a fusion reactor. In order to support Tokamak KTM as well as to create a base for interaction between plasma of fusion reactor and materials, established a simulation tests-bench with plasma-beam installation [1].

The installation provides the following plasma current parameters: diameter of plasma flux to a target is up to 30 mm; maximum magnetic field strength generated at the chamber axis of plasma-beam charge is 0.1 Tl; intensity of current in plasma is up to 1A; plasma flux density in a beam is up to $10^{22} \text{ m}^{-2} \cdot \text{s}^{-1}$; ion concentration in plasma is up to 10^{18} m^{-3} ; electron temperature of plasma is up to 100 eV; ion energy is up to 2 keV.

The simulation test-bench with plasma-beam installation, which is line simulator, was involved into tests to study how low temperature plasma affect plasma, beryllium and tungsten. A plasma-beam charge was implemented on such working gases as helium, hydrogen and deuterium.

To research structures, determine samples' element composition and their physical and mechanical properties as well as to study fracture mode of irradiated layers of materials, such methods as X-ray diffraction phase analysis, transmission electron microscopy, scanning electron microscopy, and emission spectroscopy were used, in addition micro hardness of material surface was defined after plasma effect.

Based on result of research, initial data on interaction of plasma with material surface, which are actual for applying in fusion reactor, have been obtained. In the course of works data on parameters of plasma-beam charge in different gases was received, installation was systematically upgraded to make plasma specification of simulation test-bench more similar to plasma of fusion reactor.

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OPTIMIZATION OF THE NUMERICAL MODEL OF THE TRIGGERABLE LTD SPARK GAP SWITCH

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In LTD generators, one of the main parts of the primary discharge circuit is the triggerable spark gap switch. For the cavities with oil insulation, the OrCAD switch model was developed [1-3] by taken into account the effect of the trigger pulse on the triggering time of these switches and it's spread. According to the real design, the switch in this model is simulated as a block consisting of six independent spark gaps connected in series. However, because of such detailed description of the switch structure this initial model is quite complex causing significant complications in simulation of the full generator in case it includes numerous switches. To avoid that complexity, in the given paper we investigate the possibility to optimize the initial complex model, and present at the output much less complicated switch model with acceptable simulation error.

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STUDY OF ADHESION CHARACTERISTICS OF A NI-CU SURFACE ALLOY FORMED BY A LOW-ENERGY HIGH-CURRENT ELECTRON BEAM

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Ever since the treatment of material surfaces by deposition of functional metallic coatings and thin films has become a widely used, the adhesion of these coatings to substrates represents a scientifically interesting topic with a huge practical impact in industry. Adhesion is one of the most important characteristics of coating efficiency. As structural factors (such as the state of the substrate surface, substrate roughness, the thermal expansion coefficients, etc.), and factors associated with the deposition process (such as internal stresses, thickness and the presence of impurities and structural defects in the coating, etc.) affect the adhesion of the coating to the substrate. The influence of some factors can be avoided by using various methods of preparing the substrate surface, others using additional interlayers of materials that help reduce the internal stress of the coatings. However, in all these cases, the level of adhesion will be the result of the interaction of two surfaces at the interface - the surface of the coating and the surface of the substrate. The [1,2] report that the forming surface alloys method using by a low-energy high-current electron beam (LEHCEB) can drastically improve the adhesion of the coating to the substrate. The surface alloy is formed by alternating operations of deposited a film on a substrate, followed by LEHCEB liquid-phase mixing of the film with a substrate. This method of coating synthesizing leads to the formation of a transition layer between the coating and the substrate, in other words to the blurring of the interface between the coating and the substrate. Figure 1 shows an example of such a transition layer, which can be several microns thick.

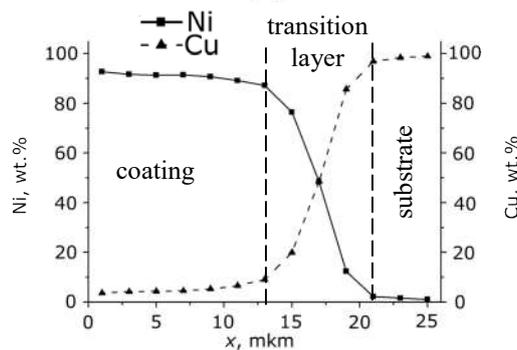


Fig. 1. In-depth elements distribution in Ni-Cu surface alloy.

However, there is practically no information in the literature on the effect of this transition layer on the adhesive characteristics of surface alloy. Therefore the aim of this work was to investigate the effect of thickness of the transition surface alloy layer on its adhesive characteristics.

The electron-beam machine “RITM-SP” with an explosive-emission cathode and a plasma-filled diode generating the LEHCEB was employed in the work [3]. This machine is equipped with a magnetron sputtering system enabling formation of surface alloys. The adhesive characteristics of the surface alloy were investigated for the Ni-film/Cu-substrate system. This system of materials is a good model system for studying the adhesive characteristics of the surface alloy, because copper and nickel form a continuous series of solid solutions that is important for the formation of a transition layer. In addition, nickel is a harder material than copper, which means that the system as a whole (both the coating and the substrate) will work under loads. Different transition layer thickness was obtained by varying the parameters during the formation of the surface alloy, such as different thickness of the deposited film and the parameters of the LEHCEB (energy density, number of pulses). Different techniques like SEM, XRD, EDS have been used for characterization of the surface morphology, phase and elemental composition of the surface alloys. Much attention in the work has been paid to investigation of in-depth elements distribution of surface alloy and adhesion characteristics by scratch-test.

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OBTAINING NANODISPERSED PRODUCT OF TITANIUM DIBORIDE IN AN ARC DISCHARGE OF MAGNETOPLASMA ACCELERATOR

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Titanium Diboride (TiB₂) is a promising material for using it in many industrial applications. TiB₂ has combination of thermal, mechanical and electrical properties such as high thermal conductivity, extreme melting point, high hardness, elastic modulus and fracture toughness, good electrical conductivity, chemical stability and excellent wear resistance [1-5]. These outstanding properties can be used in different engineering applications, such as impact-resistant armour, cathodes in Hall-Heroult cells, solar thermal absorbers and cutting tools [6-8].

Nowadays there are a lot of ways to synthesize TiB₂: in situ, sol-gel reduction, mechanical alloying, gas phase method, carbon/boron thermal reduction method, self-propagating high-temperature synthesis, chemical vapor deposition [1, 6, 9-11]. However, there are some problems in synthesize nano-dispersed powders TiB₂: obtaining unsatisfactory size and dispersal of the product, high time and energy costs.

In this paper, a new method of synthesis is proposed – plasma dynamic synthesis in a hypersonic plasma jet using titanium electrodes. The installation called a coaxial magnetoplasma accelerator (CMPA). The main part of it consists of a coaxial electrode, a central electrode, isolators and a plasma formation zone.

In order to initiate an arc discharge it is necessary to put an amorphous boron powder into a plasma formation zone and stretch between coaxial and central electrode conductors. Different ways of initiation an arc gas discharge can be suggested for obtaining TiB₂ powdered products using titanium conductors, carbon fibers or graphite aerosol (graphitization). Depending on the method of initiation of the arc discharge, a different output of titanium diboride is obtained. The higher the content of titanium diboride in the product, the better and higher will be the properties of the ceramics obtained on the basis of the powder. The content of TiB₂ in the experiment using Ti-conductors is 26,8 %, using carbon fibers is 62,1 % and with graphitization is 93,2 %. All experiments were implemented with the argon atmosphere, which filled the volume of the reactor chamber. The time of the process of initiation an arc discharge is various and depending on the using conductors: with Ti-conductors $t = 110$ ms, with carbon fibers $t = 150$ ms, graphitization $t = 160$ ms. The faster the process of exploding the conductors, the less boron powder warmed up and interacted further with titanium eroded from the surface of the electrode of the trunk. So in the experiment with graphitization the highest yield of TiB₂ is 93,2 %.

Based on the obtained powder, ceramics was synthesized by the method of spark plasma sintering. This method is one of the perspective method due to the speed of the process (5 min), high temperature (1800 °C) and pressure (60 MPa). Obtained ceramics showed high value of hardness. The values of the obtained hardness were: 1) Ti-conductors $P = 24,7$ GPa; 2) Carbon fibers $P = 28,3$ GPa; 3) Graphitization $P = 30,3$ GPa. The higher the content of the TiB₂ the harder the ceramic samples.

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OPTICAL CHARACTERISTICS OF LITHIUM FLUORIDE CRYSTALS IRRADIATED BY LITHIUM IONS AND MICROWAVE DISCHARGE PLASMA*

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The paper presents the results of studies of the optical characteristics of LiF crystals after irradiation with a beam of lithium ions, with a fluence of about 10^{17} ions / cm^2 and an energy of 100 keV. The absorption spectra of lithium fluoride crystals exposed are some overlapping bands with absorption maxima at 250 nm, 441 nm and 500 nm. The first and second bands are responsible for the absorption of F and F_2 (F_3^+) color centers, respectively. The third is the plasmon band resulting from the formation of lithium nanoparticles from implanted ions. The luminescence spectra of lithium fluoride crystals when excited by laser radiation with a wavelength of 470 nm showed that, along with the luminescence band with peaks at 550 and 680 nm, a luminescence band with a maximum at 650 nm was detected. The first and second bands are characteristic of the luminescence of the F_3^+ and F_2 color centers, respectively, which appear in the thin surface layer upon irradiation with ions. The third band resulted from lithium colloids. Measurements of the kinetic characteristics of the luminescence revealed four decay times, namely 15.3 and 7.6 ns, close to the characteristic decay times for the F_2 and F_3^+ centers of the luminescence in the LiF crystal. In addition, two fast components with a decay time of about 2.3 and 0.5 ns were found that were associated with the luminescence of lithium nanoparticles.

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FEATURES OF SELF-SUSTAINED MAGNETRON SPUTTERING OF EVAPORATING METAL TARGET

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Studies of the mechanisms and regularities of the discharge operation were performed when the magnetron sputtering system (MSS) was operating in the self-sputtering mode of an evaporating metal target under conditions of extremely low pressure in the vacuum chamber (less than 0.1 Pa) due to the termination of the inlet of the sputtering gas argon. It has been established that the magnetron is able to work in gasless mode if, due to evaporation of the target, the concentration of metal atoms near the surface of the target is necessary to maintain the discharge. Using the example of an MSS with a copper target, the minimum required power has been determined, starting from which the discharge can function without the inlet of the sputtering gas. The threshold power value depends on the crucible substance and the type of power source (mid-frequency, high-current). Thus, in the case of using a copper target in a molybdenum crucible and a mid-frequency power source, the minimum power density required for stable self-sputtering without supplying the sputtering gas is 19.4 W/cm^2 , and in the case of a high-current power source - 33 W/cm^2 . It has been revealed that thermo-electronic emission is not a necessary factor in maintaining the discharge of a magnetron operating on vapor of a target substance.

It has been found that the erosion yields of metal targets at evaporation reach several tens of atoms per ion, which is an order of magnitude higher than the sputtering yields. Due to this, the coatings deposition under self-sputtering conditions takes place without reducing a deposition rate as compared to the case with sputtering gas.

The evolution of the intensity of the spectral lines of the plasma optical radiation during the transition of a magnetron with an evaporating copper target into the self-sputtering mode and switching off the sputtering gas was studied. A correlation was found between the intensity of the spectral lines of copper atoms and ions with the evolution of evaporation. As the intensity of evaporation increases, argon atoms are displaced from the burning region of the discharge in front of the target.

The flux densities of deposited particles and energy under conditions of intense evaporation of a copper target were studied. It was found that in the considered MSS power range, due to evaporation, the flux density of the deposited particles increases by about an order of magnitude. The main source of energy entering the substrate is heat radiation from the target. The magnitude of the total energy flux is about the same as when the target is sputtered in an argon atmosphere, and in the self-sputtering mode.

Experiments were carried out on the deposition of copper coatings with argon at a pressure of 0.2 Pa and in the full self-sputtering mode at a pressure of 0.01 Pa. Different power sources were used (mid-frequency and high-current). The microstructure, crystal structure and roughness of coatings obtained at different evaporation rates were studied. It turned out that under conditions of intensive evaporation, there is no noticeable pronounced influence of the self-sputtering factor and the type of power source on the studied characteristics of copper coatings. At low evaporation rates, the structural characteristics of the coatings turn out to be better in the gasless mode.

OBTAINING OF HIGH-POWER ELECTRON BEAMS IN A PLASMA ANODE ELECTRON SOURCE POWERED BY MARX GENERATOR WITH MATCHED LOADS *

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The paper presents the results of experiments for obtaining high-power microsecond electron beams of circular and rectangular cross-section in an electron source with an explosive emission cathode and a plasma anode. The use of the plasma anode allows to increase the current and energy of the electron beam, increases the reliability of the electron source operation, provides the possibility to realize a controlled mode of the source operation with the beam current control without changing the accelerating voltage.

The source of high voltage was the Marx generator with stages in the form of artificial long lines with matched loads. The generator consists of 6 stages and has air isolation. The wave impedance of the generator is 25 Ω , the pulse duration at half-height equals to 5 μs and corresponds to the time of the wave travel along the line. The generator provides obtaining rectangular voltage pulses up to 200-250 kV without reflections at a constant arbitrary resistive load in a single-pulse mode. In the case of time-varying resistive load, the wave form of the generated pulses differs from the rectangular one, but the pulse duration remains the same [1, 2].

In the experiments, round and rectangular multipoint cathodes with round and rectangular regions of location of the points, respectively, were used. To form a plasma anode, plasma was injected into the interelectrode gap using plasma guns with a discharge along the dielectric surface. Beam formation was realized when the longitudinal magnetic field was applied to the interelectrode gap, plasma anode, and the transportation region.

The performed experiments show that the use of a generator with matched loads prevents the breakdown and ignition of the arc discharge in the interelectrode gap. It is possible to implement operation modes of the electron source with quasi-constant values of the accelerating voltage and beam current. At the accelerating voltage of 200 kV, electron beams of round or rectangular cross-section of $\sim 100\text{-}200\text{ cm}^2$ with a current of 1-1.5 kA and duration of 5 μs were received. The rotation of the beam around the axis was registered. The possibility to extract an electron beam through a foil window into the atmosphere was tested.

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PRODUCTION OF AL-O-N NANOPOWDERS IN A PLASMA REACTOR WITH A LIMITED JET FLOW*

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Experimental studies of aluminum oxynitride nanopowders synthesis in a reactor with a confined plasma jet by the interaction of disperse aluminum with ammonia and oxygen in the flow of nitrogen plasma generated in an electric arc plasma torch are carried out.

Optimal design of the reaction prechamber of the reactor selection were made.

Powders with an average particle size in the range of 20-200 nm, having a cubic structure and consisting of aluminum oxynitride phases, were obtained.

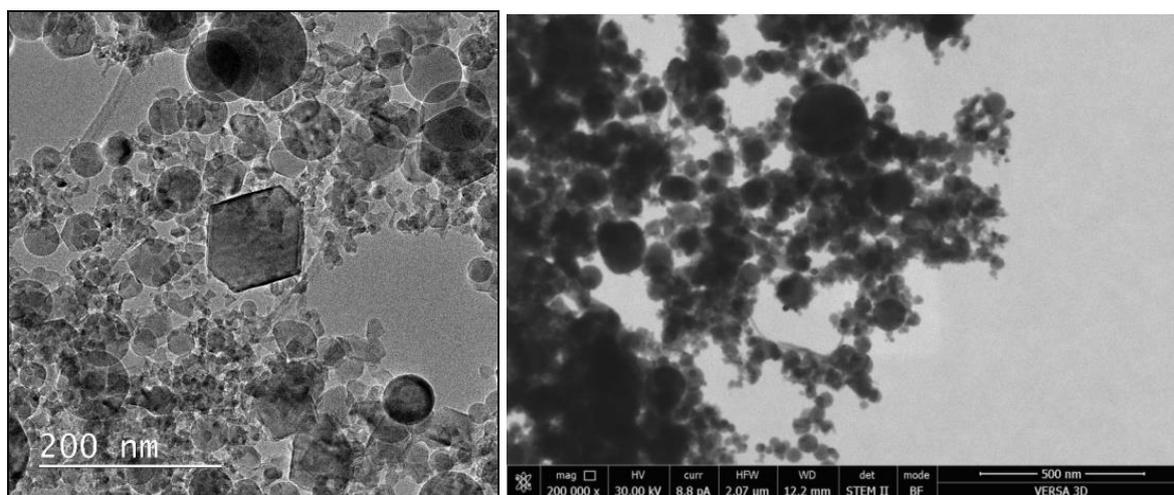


Fig. 1. SEM and TEM images of obtained nanopowders.

As a result of the research, the possibility of controlling the phase composition of the obtained nanopowders, as well as such properties as the specific surface area and the content of nitrogen and oxygen in the product, has been established

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OBTAINING THE ULTRADISPERSE MATERIAL OF THE Al-Mg-O SYSTEM BY PLASMA DYNAMIC METHOD

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Nanomaterials, in particular metal oxides, have a wide range of applications. Prospects for use of such materials are associated with their unique features in the ultrafine state. Aluminum oxide is a wide-gap dielectric with high wear resistance, mechanical and chemical resistance, which is used in medicine, optics and various technical fields [1, 2]. It is known that there is a possibility of improving the characteristics and improving the functional properties of the material due to the introduction of a small amount of the MgAl₂O₄ spinel phase into its composition [3].

To date, there are many methods for producing nanosized aluminum oxide, for example, gas-phase method, electric explosion method of conductors, or sol-gel method [4-6]. Of course, these methods have advantages and disadvantages. The disadvantages include unsatisfactory dispersion of the product and high duration and multi-stage nature of the material production process.

The method of plasma dynamic synthesis developed at the Tomsk Polytechnic University is devoid of the above-noted drawbacks and can be considered as an alternative method for producing nano-dispersed aluminum oxide. This method is based on the use of high-current high-voltage coaxial magnetoplasma accelerator of the erosion type with an aluminum accelerator channel. The main advantage of the method is its speed – the synthesis time takes less than 1 ms [7]. At the same time, the resulting products are distinguished by their high dispersion. The simplicity of the method lies in the fact that, using a simple aluminum alloy tube containing about 7% magnesium as a barrel, and when the gaseous precursor oxygen is pumped into the reactor chamber, it is possible to obtain unique aluminum oxide and spinel phases. Synthesis of aluminum oxide was carried out due to erosion of the aluminum barrel. When the arc flows through the acceleration channel, the base material, aluminum, is produced, after which it is carried into the chamber, where it enters into a plasma-chemical reaction with oxygen, forming the desired product. To obtain purer product of plasma dynamic synthesis, it was proposed to use a system with the separation of the synthesized product into a large and small fraction.

The paper shows experimentally the possibility of producing aluminum oxide and spinel in a system based on the use of a pulsed high-current coaxial magnetoplasma accelerator of the erosion type. The average particle size in the product varies from 50 nm to 250 nm. It should be noted that the installation allows you to change the ratio of Al₂O₃ to MgAl₂O₄. In the future we plan to use this material to obtain bulk ceramic samples.

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ANALYSIS OF ELECTRICAL CHARACTERISTICS OF CERAMICS ON THE BASIS OF ZnO-Bi₂O₃, OBTAINED BY SPARK PLASMA SINTERING*

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Ceramic zinc oxide varistors are the most promising fast-acting means of protecting electrical circuits from impulse overvoltages. They have high non-linearity of electrical characteristics. Due to the recent need to protect semiconductor control circuits of power equipment, the limiting voltage of which only slightly exceeds the operating one, it is important to increase the nonlinearity of varistors in the field of switching currents, limiting overvoltage to a safe level [1].

Obtaining ceramic samples from nanopowders is a challenge. Currently, effective methods of pressing powdered materials include the following methods: hot pressing, hot isostatic pressing and spark plasma sintering (SPS). The main advantage of the SPS is the speed of the process of consolidation of powders [2]. The paper considers the possibility of sintering powdered materials of the ZnO-Bi₂O₃ system, obtained by the method of plasma dynamic synthesis (PDS) [2, 3] and under commercial conditions. The PDS method does not require preliminary preparation of the main precursors - zinc and oxygen. Zinc enters the plasma due to the electroerosive wear of the zinc barrel and is carried into the reactor chamber, which is pre-filled with oxygen. In addition, the channel of formation of the plasma structure can be filled with additional precursors (for example, bismuth), which also enter the plasma structure when initiating the arc discharge. Metallic bismuth Bi (purity 99%) in the form of a powder with an average particle size of about 100 microns was inserted into the channel of formation of the plasma structure of a high-current discharge at the beginning of the accelerating channel (AC) of the zinc barrel of a coaxial magnetoplasma accelerator (CMPA). During the course of the arc according to the AC, the production of the base material, zinc, which enters the plasma structure, in which bismuth is already present, occurs. After that, the plasma structure is carried into the chamber, where it enters into a plasma-chemical reaction with oxygen. The collection of the highly dispersed fraction of the product was carried out after its complete precipitation from the suspension on the walls of the reactor chamber. The sample was sintered using the IPA method in vacuum in a graphite mold under a pressure of 60 MPa and at a sintering temperature $T = 1200$ °C.

The paper shows the possibility of obtaining ultrafine composite materials of the ZnO-Bi₂O₃ system with the core-shell structure using a high-current high-voltage coaxial magnetoplasma accelerator. In addition, studies have been conducted on the consolidation of materials by the method of spark plasma sintering. Sintered ceramics based on the PDS product is characterized by the fine-grained structure of zinc oxide ZnO (average grain size 1.3 μm) with a uniformly filled intergranular space bismuth oxide Bi₂O₃. Analysis of the current-voltage characteristics of ceramics of different composition showed a significant advantage (nonlinearity coefficient, breakdown voltage, leakage current) of using the PDS ZnO-Bi₂O₃ product with the core-shell structure compared to commercial materials.

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PRECISION CUTTING OF HIGH-TEMPERATURE DIELECTRICS BY THE FOREVACUUM PLASMA ELECTRON SOURCE*

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Electron-beam technologies find wide application for welding various kinds of materials [1], among which are cutting, welding, evaporation, surfacing. The interest in electron-beam processing of dielectrics has led to the development of forevacuum electron sources [2, 3], operating at pressures from a few to hundreds of pascals. The features of the operation of such sources, associated with the passage of electrons of the beam in a gaseous medium at elevated pressures, provide charge neutralization of the dielectric surface during processing. Due to this, forevacuum sources are capable of producing continuous electron-beam processing for a long time, and the beam power density achieved so far ensures effective precision cutting of such high-temperature dielectrics as ceramics and quartz.

This paper presents the results of usage the forevacuum source of a focused electron beam with currently record specific beam parameters (electron energy up to 30 keV, beam diameter up to 0.15 mm, beam power density in the crossover about 10^6 W/cm²) [4, 5] for cutting of such dielectrics having high melting point as ceramic and quartz glass. The effect on the size of the single hole created in the dielectric of the main parameters of the electron beam processing mode (energy of electrons, beam power density and time of electron-beam processing) is investigated. It is shown, that depth of the single hole created by the electron beam directly determined by the beam power density (fig 1), and for beam power density at the level of 10^6 W/cm² can reach several centimeters. Also the paper demonstrated the possibility of creating extended holes in ceramic and glass.

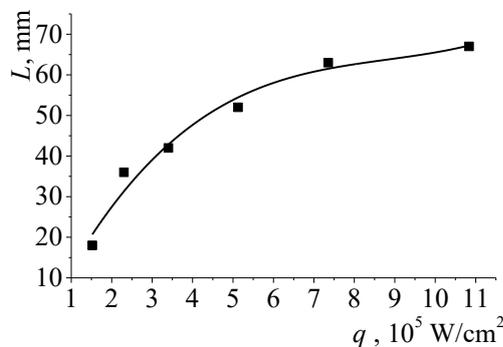


Fig. 1. The dependence of hole depth L on beam power density of the electron beam q

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DIRECT CURRENT ARC-PLASMA SYNTHESIS OF B-C POWDER PRODUCT*

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Boron carbide is widely spread, super-hard material, which is characterized by low density ($\sim 2.5 \text{ g/cm}^3$) high melting temperature ($\sim 2620\text{--}2740 \text{ }^\circ\text{C}$), high resistivity to some radiation and other unique properties [1]. Boron carbide can be synthesized by several methods, such as: carbo-thermal reduction of boron oxide, plasma spraying, melt crystallization, and CVD [2-5]. Last years a new method of direct current arc discharge has been developed for carbon nanostructures [6] and boron carbide [7] synthesis. The main feature of this method is the possibility to operate at generating the ambient air plasma. This procedure is becoming possible due to carbon monoxide generation during arcing, that results in gas, which can insulate the reaction zone and prevent oxidation from synthesis products. The procedure implementation has been discussed before in [6-7]. In this paper we study the crystalline phase composition and crystallinity of powder products in link with a synthesis process time. The arc discharge experiments were conducted by DC APAS-method (direct current arc plasma air synthesis) at a plasma chemical reactor that is introduced in [7].

According to X-ray diffraction data (Shimadzu XRD 7000s, $\lambda=1.54060 \text{ \AA}$, $\text{CuK}\alpha$) a typical product consists of three main crystalline phases: graphite (C (graphite)), boron oxide B_2O_3 , as initial raw materials, and synthesized boron carbide B_{13}C_2 . The phase composition depends on arc discharge time as mentioned below in Fig. 1.

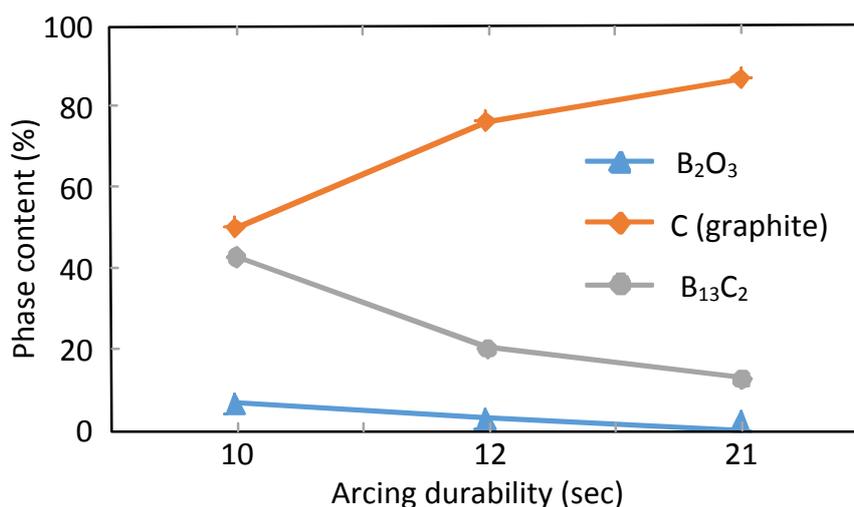


Fig. 1. Phase composition and arcing time dependence

The maximum time is 21 seconds that can lead to full mass of initial boron oxide consumption. At this time the content of graphite increases owing to the anode erosion effect [8]. According to these data we make a conclusion that arcing time influences the phase composition through energy input variation by time control. Besides, it is possible to obtain two phase composition of a product by full mass of the boron oxide consumption.

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PLASMACHEMICAL PROCESSING OF GERMANIUM-CONTAINING MINERAL AND TECHNOLOGICAL RAW MATERIALS *

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The aim of the work was to study the possibility of plasma-chemical enrichment of germanium-containing fly ash. The essence of the method consists in converting germanium in the form of monoxide into the gas phase and its subsequent separation from the solid phase.

Thermodynamic modeling of the Ge-O-C-N system showed (see fig. 1) That the minimum temperature for the existence of GeO is 900 ° C. Those. for a given composition of the mixture, monoxide below this temperature is unstable and, depending on the ratio of the components, passes into either Ge (at C > 15 g) or GeO₂ (at C < 15 g).

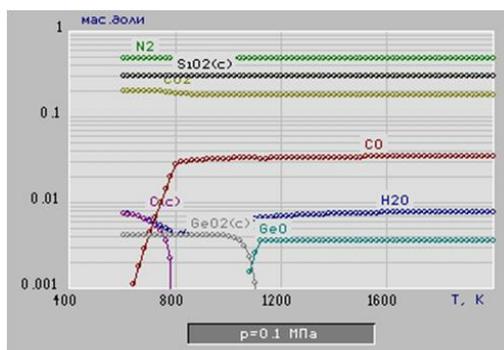


Fig 1. The equilibrium composition of the mixture: GeO₂- 1 g, SiO₂- 70 g, O₂-35 g, N₂-115 g, C = 15 g

In a direct-flow plasma-chemical reactor with hot walls, experiments were conducted to investigate the possibility of plasma-chemical extraction of germanium with the following parameters: air consumption – 2 g/s, consumption of germanium-containing ash from coal combustion - 1.5 g/s. The dependence of the degree of extraction of germanium in the gas phase on the composition of the plasma-forming gas was investigated. The composition of the gas phase in the reactor was changed by feeding propane.

As a result of processing raw materials, two types of solid products are obtained: molten slag and powdered ash. The residual germanium content in the slag was below the sensitivity limit (0.001%) of the method used. The powdery product of plasma chemical processing was characterized by a relatively low degree of extraction of ~ 70–80%, which is apparently explained by large particle sizes, which is why the necessary temperature was not reached. Considering the data obtained, as well as the fractional composition of the feedstock (particle size reached 5 mm), it was concluded that the processing of ash with the melting of its mineral part was expedient. This simplifies the processing scheme due to the simpler separation of gas from the melt of the mineral part of the ash.

Plasma-chemical processing of germanium-containing ash in a plasma-chemical reactor with a flowing melt film showed that the degree of germanium extraction from ash was 95-96%. The residual germanium content in the slag was 0.04% with the initial content of 0.65%. In the experiments, the supply of propane to the reactor was not used, and the residual germanium content in the slag approximately corresponded to the previous experiments in the direct-flow reactor without the supply of propane. Therefore, by optimizing the composition of the gas phase in the reactor, it is possible to further increase the degree of germanium extraction from the processed raw material.

Thus, the experiments carried out showed the possibility of efficient plasma-chemical separation of germanium from low-concentrated raw materials with the degree of enrichment in the resulting product up to 30-40 times.

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ENERGY DEPTH DISTRIBUTION OF PULSED ELECTRON BEAM OF WIDE ELECTRON KINETIC ENERGY SPECTRUM FOR AN ALUMINUM TARGET*

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An electron beam is a high-tech tool which can be used in various fields of radiation technology [1, 2]. Utilizing of pulsed electron accelerators with a wide range of electron kinetic energies significantly reduces the cost of their production. Besides, the electron beam ejected to the atmosphere can significantly expand its application scope. Transmission of the electron beam to the atmosphere through sealing membranes substantially changes its initial spectrum. Therefore, knowledge of pulsed electron beam characteristics is necessary for use it for scientific and practice applications.

Current work analyze the pulsed electron beam extracted from the vacuum diode through a titanium foil (60 microns) of the diode exit window. Electron beam energy depth distribution was measured for a target made of different number of aluminum foils. A pulsed electron beam with a wide range of kinetic energies was generated by the ASTRA-M accelerator (260 kV of accelerating voltage, up to 1kA of beam current, 150 ns of beam pulse duration at FWHM)[3]. Total absorption calorimeter was used to measure beam characteristics. Calorimeter included two collectors: first for measuring of a beam energy after aluminum foils, and a second one for measuring total beam energy. All measurements were performed at 10^{-5} Torr background pressure after the exit window foil. As a result, the electron kinetic energy spectrum of the beam out of diode has been reconstructed. The calculation of electron energy spectrum after titanium foil was made with help of database[4].

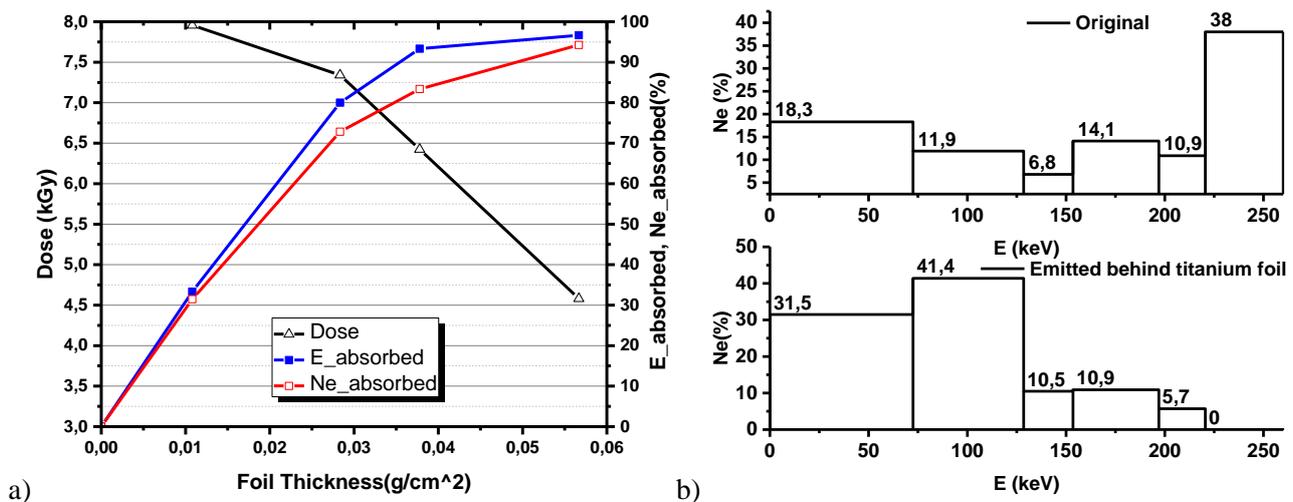


Fig. 1. a) Absorbed Dose, beam energy and electron numbers distribution from depth aluminum target, b) Electron beam energy spectrum before and after titanium foil.

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HARD ALLOY MODIFICATION BY GLOW DISCHARGE PLASMA

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The use of hard alloys makes it possible to process metals at ultrahigh cutting speeds since these alloys have a very high hardness and wear resistance. Such alloys are used in the processing of parts from high-strength, heat-resistant and stainless steels. Therefore, improving the performance characteristics of such alloys is an important task the solution of which will ensure an increase in labor productivity and economy of imported materials. One of the most promising is a method of improving the operational characteristics of cutting tools made from hard alloys by treatment with a glow discharge. A distinctive feature of this treatment is the versatility of the method with a high degree of productivity as well as the possibility to process much larger areas. Plasma treatment does not require the use of liquid solutions as a result it is environmentally friendly and energy-intensive.

The treatment of hard alloys was carried out by a dc glow discharge excited in a medium of residual atmospheric gases with a pressure of 1.33 ... 13.33 Pa at a voltage of 0.5 ... 5 kV, a current density of 0.05 ... 0.5 A/m², a pulse frequency of 35 KHz± 30 % and the ratio of the areas of the anode to the cathode 0.010 ... 0.015 [1]. Such processing ensures the formation of unique structural and phase states in their surface layers as well as a wide scale of structure modification [2, 3]. These changes lead to changes in the mechanical and operational properties of the surface layer.

The aim of this work is to study the effect of modifying treatment by a glow discharge on the structure and operational characteristics of hard alloys. Electron microscopic, X-ray diffraction methods were used for this study to analyze the phase composition, structure and properties of the surface layer. Along with the study of the structure, the hardness and wear resistance of the samples were studied. This study may determine how the structural changes in the surface layers affect the change in hardness and wear resistance of the samples. The measurement of the hardness of the samples was carried out according to the Vickers method. The study of the effect of treatment in a glow discharge on wear resistance was carried out on an equipment based on a machine for testing materials for friction and wear.

As a result of the research it was found that plasma treatment leads to an increase in the surface hardness of hard alloys by an average of 10 ... 15%. The processing of hard alloys with a glow discharge with certain technological characteristics leads to an increase in their wear resistance up to 3 times. Moreover, the modifying treatment leads to relatively uniform wear throughout the cutting until critical wear is achieved. The increase in hardness and wear resistance of the hard alloys is associated with the creation of a modified layer to a depth of 70 microns.

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ELECTRIC ARC SYNTHESIS OF MICRO DIAMONDS.*

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Currently, methods of chemical vapor deposition (CVD - chemical vapor deposition) or methods of self-organization at high pressures and temperatures are used to synthesize artificial diamonds. It was by these methods that large artificial diamonds and industrial-use diamonds were grown. In [1], it was reported about the effectiveness of the use of elements of the fourth group of the periodic table as catalysts in the process of diamond synthesis. The authors of [1] managed to grow diamond crystals with a size of 20–100 μm in 60 hours using the CVD method.

This paper presents the results of experiments on the synthesis of microdiamonds from graphite in an argon arc using germanium as a catalyst. To understand the synthesis of diamonds, one must have an idea of the temperature field in an arc discharge. The most reliable information about the temperature field was obtained by conducting a numerical experiment taking into account the greatest number of physical processes in [2]. The results of these studies allowed designing complex electrode assemblies both for the synthesis of nanotubes [3] and for the synthesis of diamonds in this work.

For this, a “hybrid” anode was made, consisting of a graphite frame with a diameter of 1.5 cm, into which a germanium rod 5 mm thick was inserted. Figure 1 on the left shows the traces left by diamond microparticles on the glass surface, and on the right - microdiamonds surrounded by germanium nanoparticles.

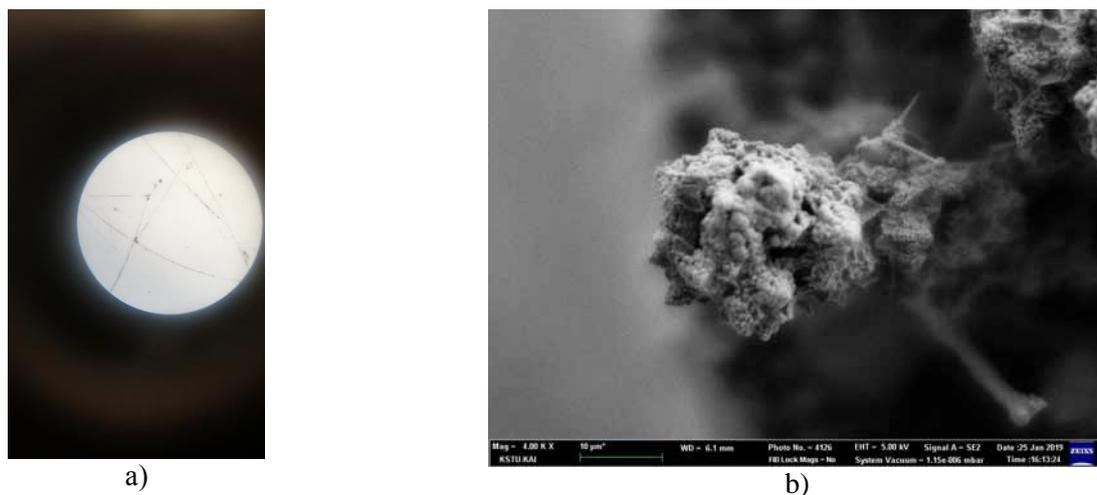


Fig. 1. a) traces of microdiamonds left by their friction between the panes. b) micro-diamonds surrounded by germanium nanostructures. Magnification 4000 times.

It has been revealed that the forms of the grown nanomaterials are significantly affected by both the surrounding gaseous medium and the electrical parameters of the arc discharge — the current and the distribution of the electric field intensity. Simple microdiamonds were obtained, as well as diamonds of a complex configuration, which, apparently, were formed in this form due to the absence of an initial embryo. The formation of microdiamonds is a few tens of seconds, which is much different from the traditional methods of their production.

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PLASMA CHEMICAL PROCESSING OF HYDROCARBONS.*

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In the present work, a method of plasma-chemical processing of hydrocarbon raw materials with the aim of obtaining valuable products, including nanostructures, is proposed. For this purpose, a method of organizing an electrical discharge of direct current in the thickness of the liquid raw material was used. In order to automate ignition processes and optimize the discharge burning process, an installation with a rotating electrode mechanism was developed and designed, which eliminated sticking of the electrodes and discharge attenuation. In fig. 1 a) presents photograph of the discharge in the thickness of the hydrocarbon raw materials. The following operating modes were considered. When the distance between the electrodes is 0.5 mm, a stable arc is observed at currents of 0.1 - 1 A and at voltages of 550 - 100 V. With these discharge parameters, the decomposition of hydrocarbons into light fractions occurs. In the process of decomposition, the formation of a vapor-gas mixture is continuous, and the formation of carbon samples occurs in small quantities.

The products obtained as a result of the interaction of a gas-discharge plasma, initiated in the bulk of the hydrocarbon feedstock with the latter, were subjected to thorough analysis. In fig. 1 b) and c) images of carbon deposits formed at the cathode, taken with an electron microscope.

Nanostructures were formed in a chaotic manner in the form of closely woven threads. Since the nanotubes are twisted together, it can be assumed that they have a complex structure. In addition, multilayer nanotubes of the “Russian nesting doll” type were observed. A nanotube has a diameter of 44.04 nm, a nanotube similar to it, which is inside it, has a diameter of 18.46 nm.

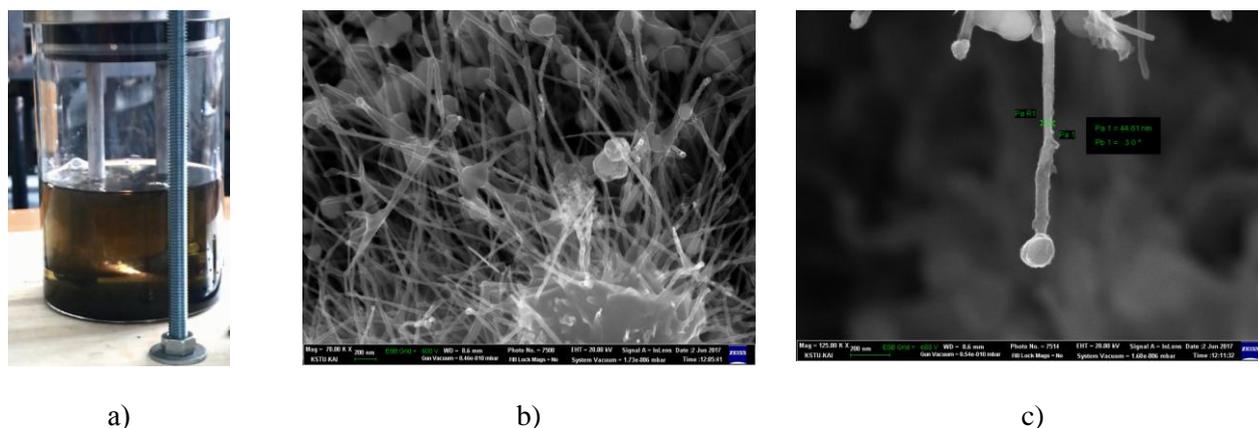


Fig. 1. a) Photograph of the arc discharge during the experiment with diesel fuel; Electron microscopic picture of carbon deposits at the anode: b) Increase of 70.000x., C) Increase of 125.000x.

The light and volatile fractions of oil produced during the plasma-chemical treatment of fuel oil were analyzed on a Chromatek Kristall 5000.2 chromatograph. Chromatographic analysis showed that the main products of the decomposition of hydrocarbons are: ethylene more than 40%, hydrogen 24%, methane 7%.

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PLASMADYNAMIC SYNTHESIS OF ULTRAFINE TITANIUM OXIDES

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Over the past decade, interest has increased in the creation of nanoscale materials due to their unique properties. One of such materials is titanium dioxide due to its characteristics, which is widely used in modern fields of science and technology, in particular, medicine, pigment production, microbiology, photocatalysis, etc. [1, 2]. Moreover, titanium dioxide is used in the coatings form serving to increase the mechanical strength, specific surface area and selectivity of catalysts obtained on their basis.

There are many different ways to produce nanosized titanium dioxide [3, 4], but they have several disadvantages: high cost of raw materials, multistage. This paper shows the synthesis of ultrafine titanium dioxide by the plasmodynamic method. The method allows to obtain material in a fraction of a second (10^{-3} sec.), is one-step and does not require any preliminary preparation, besides its implementation takes place in atmospheric conditions.

Plasmadynamic synthesis is realized in system, in which the main element is a pulsed high-current coaxial magnetoplasma accelerator (CMPA) of the erosion type with metal titanium electrodes. The power supply of the CMPA is provided from a partitioned capacitive energy storage with a capacity of up to $C_{ch} = 28.8$ mF and a charging voltage up to $U_{ch} = 5$ kV.

Figure 1 shows the SEM-image of the product obtained plasma-dynamic method. This study was conducted using a scanning electron microscope Hitachi TM-3000. The synthesized powder is sufficiently agglomerated, which is characteristic for electrophysical methods of dispersion [5]. Estimating the brightness and contrast of the image, we can conclude that the product consists of materials of similar density and is characterized by a fairly wide distribution in the range from 100 nm to 3 μ m. There is the presence of single spherical objects with a size of 5 microns.

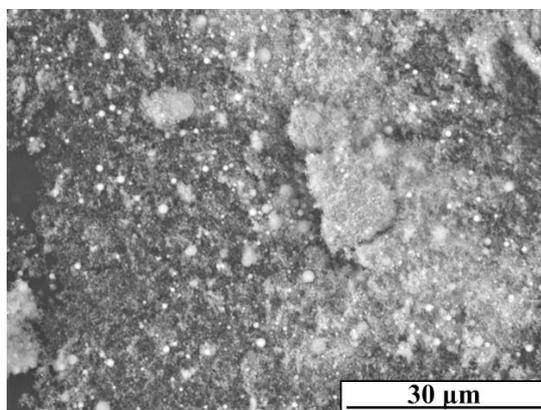


Fig. 1. The SEM-image of synthesized material

Powder of Ti-O phases were obtained by the synthesis in a supersonic plasma jet. The results of scanning electron microscopy have shown that the particles have a spherical form with their sizes varying from 100 nm to 5 microns.

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CONTROL OF THE PARTICLE SIZE DISTRIBUTION AND THE INVESTIGATION OF THE CRYSTAL STRUCTURE OF THE TITANIUM OXIDE POWDERS, OBTAINED BY PLASMADYNAMIC METHOD

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At the present time, titanium oxide (IV), such as TiO₂ (titanium dioxide) is widespread in many industries. It is used in the production of solid films in photocatalysis and solar energy; in addition, titanium dioxide has found its application in both chemical and pharmaceutical production [1-4].

An important problem is the development of methods for the direct synthesis of a finely dispersed phase, since it is possible to achieve improved characteristics of titanium dioxide only in a nanoscale form [5].

This paper shows a method for obtaining ultrafine powder by plasmodynamic synthesis. The main advantages of this method are the speed of the process (10⁻³ sec.), the absence of the need for preliminary preparation of the material and its constant dosing. Also, the method is environmentally friendly and safe.

The synthesized product without any preliminary preparation was investigated by x-ray diffractometry. The fig. 1 shows the diffraction pattern of the synthesized material and cards of the proposed phases. The analysis was performed using a Shimadzu XRD7000 X-ray diffractometer (CuK_α radiation) equipped with a counting monochromator. The full-profile analysis was carried out in the "PowderCell 2.4" software environment and the PDF4+ structural data base. Two crystal modifications of TiO₂ have been identified: anatase aTiO₂ with tetragonal syngony (no. 21-1272) and rutile rTiO₂ also with tetragonal syngony (no. 21-1276). There is a broadening of the peak in the range of 53.8 ÷ 54.5 degrees. However, its separation into 2 phases is clearly visible: aTiO₂ and rTiO₂.

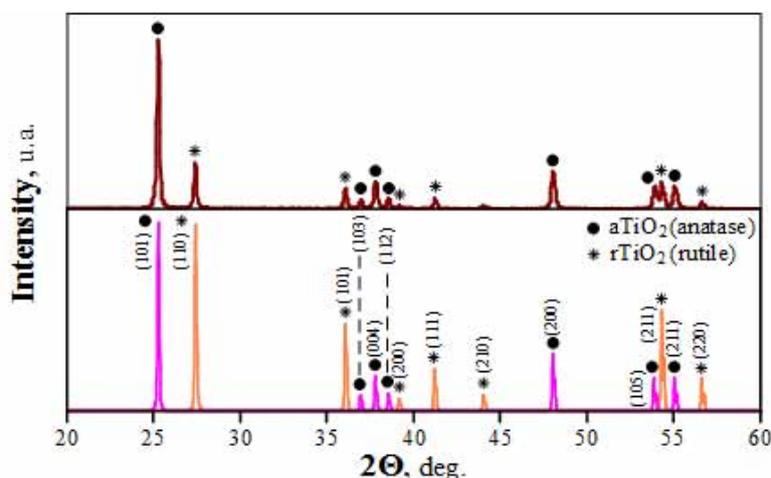


Fig. 1. Diffraction patterns of obtained material and proposed phases

The paper has shown the results about the synthesis of ultrafine titanium dioxide. X-ray phase analysis has determined the presence of 2 crystalline modifications of titanium dioxide: anatase with tetragonal syngony and rutile with the same syngony.

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CASCADE VOLUMETRIC ACCELERATION OF ELECTROHYDRODYNAMIC FLOWS*

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In this paper, we study the systems of electrohydrodynamic flows based on cascade acceleration [1, 2] - systems with ion injection into the acceleration unit (by corona or dielectric barrier discharge) and their further acceleration by synchronizing the movement of ions with the polarity of accelerating electrodes. To implement electrohydrodynamic flows cascade volumetric acceleration, the experimental setup was created (Fig. 1). It consists of two EHD cascade C_1 , C_2 [3], Spellman SL2000 high-voltage power supply, plasma emitters (PE), current-limiting resistor R of 100 k Ω , high-voltage switch (HVS) that forms rectangular pulses [4]. HVS output voltage was smoothly regulated from 0 to 8 kV and monitored through a high-voltage probe with a multimeter V .

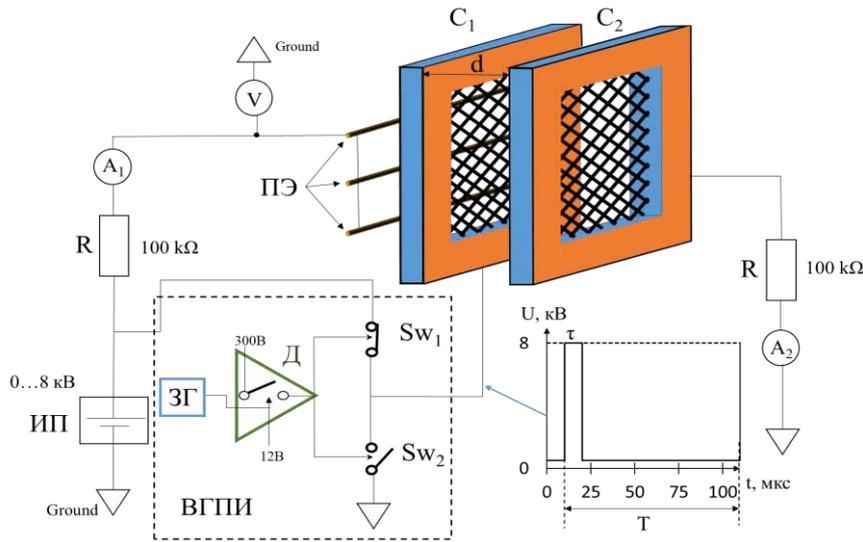


Fig. 1. Experimental setup to study the volumetric acceleration of EHD flows

The discharge current was measured by an A_1 microammeter, and output current by A_2 . The number of emitters was selected on the basis of the range and sensitivity of A_1 and A_2 (10-100 μ A). The impulse corona discharge was formed at the moment when control driver D open the switch Sw_1 and close Sw_2 . Due to this, between the PE and first section grid high voltage difference is forming. The duty cycle of a rectangular signal and pulse repetition rate was regulated by the driving generator (DG). High-voltage switches Sw_1 , Sw_2 are designed for operating at voltages over 10kV and consist of 8 modules of series-connected IGBT without snubber circuits, which reduces losses when working at high repetition frequencies and small pulse width (200 ns). According to the results of the study for a two-section accelerating module, an ion current at second section through A_2 can be achieved of up to 70% at a duty cycle of $D = 0.1$ and of 62% at $D=0.5$. Enhancement of the effectiveness of the EHD flows formation systems is important for creating aircraft [5, 6] on the base of these devices.

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SURFACE PROPERTIES OF POLYLACTIC ACID FILMS AFTER PLASMA TREATMENT

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Polylactic acid is a biodegradable, aliphatic polyester of lactic (2-hydroxypropionic) acid and widely used for medical purposes. Plasma modification of surface allows to change the surface properties (adhesion, wettability) of films and to sterilize the polymer films.

The aim of this work is the study of low temperature plasma influence on the polylactic acid films surface properties.

Materials and methods. The polylactic acid films were obtained by dissolving of polylactic acid PL10 (PURAC, Netherlands) in a solvent of trichloromethane (CHCl₃) (EKOS-1, Russia). The resulting 1% solution in an amount of 10 g was poured into Petri dishes and left for 2-3 days. The films thickness was 20±0,1 μm. The plasma modification of polylactic acid films was done with using the experimental low temperature plasma device. The plasma treatment time of each film surface was 30, 60 and 90 seconds. The surface topography and the roughness of polylactic acid films were studied on atomic force microscopy (AFM) "Solver-HV".

Results. The polylactic acid films had two different sides: front side was more relief, backside was smoother. The inner side of the film had a smoother surface (fig. 1). Ra of polylactic acid films varied from 0.01 to 0.018 μm within the error range from 0.003 to 0.005 μm.

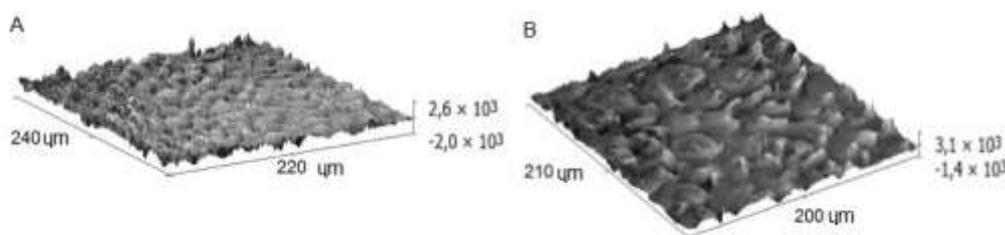


Fig. 1. The polylactic acid films surfaces: a – inner surface; b – outer surface.

The plasma increased the roughness of polylactic acid films by 2.4 times (plasma treatment time was 90 seconds). The asymmetry parameter of all samples was $R_{sk} < |1.5|$.

All samples had the left side asymmetry.

The analysis of the obtained data showed that the films had a wetting angle $\theta = 80^\circ$ and their properties were close to hydrophobic. The surface energy of the films varied in the range of 26-27 mJ / m². The contribution of the dispersion component was more significant than the polarization one. The polarity of the polylactic acid films was 0.36. The plasma decreased the wetting angle of the polylactic acid films by 1.5 times.

Conclusion. The polylactic acid films have two different sides: front side was more relief, backside was smoother. The low temperature plasma modification contributes to increase the surface roughness and decrease the wetting angle of the polylactic acid films.

The research was conducted with the financial support of the Russian Foundation for Basic Research (RFBR) as part of the project № 18-315-00048.

MULTILAYER CHROMIUM NITRIDE/CARBON COATINGS DEPOSITED BY MAGNETRON SPUTTERING

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Multilayer chromium nitride/carbon coatings ($\sim 0.9 \mu\text{m}$) were deposited by magnetron sputtering in Ar atmosphere at 0.2 Pa. This study focuses on the effect of the layer thickness (50, 100, 150 and 225 nm) on structural and mechanical properties of the multilayer coatings.

The crystal structure of the deposited coatings was investigated by X-ray diffraction and Raman spectroscopy. A cubic structure of CrN and an amorphous carbon phase could be identified within the coatings. The increase in layer thickness results in an increase in grain size of CrN from 11 to 53 nm as well as in the occurrence of strains inside the CrN phase. The ratio of I_D to I_G measured by Raman spectroscopy slightly changed from 1.045 to 1.103.

The film morphology is significantly improved (R_a from 12.8 to 23.5 nm) for the thin coatings.

Hardness and elastic modulus of the CrN/C coatings were measured by nanoindentation at a penetration depth up to 0.3 μm . Highest hardness and lowest elastic modulus were measured for the CrN/C coating with a layer thickness of 225 nm.

The coating adhesion to stainless steel substrate was determined by a scratch-test. The resistance to coating chipping of the CrN/C films could be improved from 10.7 to 41.9 N with the decrease in layer thickness.

SYNTHESIS OF NANODIMENSIONAL CARBON FILMS IN HOLLOW CATHODE DISCHARGE

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The plasma sources based on the hollow cathode discharge (HCD) are used in a wide variety of applications. The term HCD is applied to plasmas in a cathode with negative curvature geometry. The hollow cathode effect used in the HCD is characterized by electron trapping by the geometry of the cathode resulting to plasma density higher than a common glow discharge (from 10^{11} to 10^{13} cm⁻³). HCD excites when the distance between cathode surfaces is reduced, while the applied potential and gas pressure are kept constant, or conversely for a given cathode geometry (the gas pressure is increased such that “dp” product is 1 - 10 Torr cm depending on the gas used). Features of the HCD allows to use it, for example: for elemental analysis [1], nitriding [2], vacuum welding [3], plasma-activated pre-treatment and coating processes [4-5], also as efficient sources of light [6], electrons [7], ions [1, 8] etc.

Here, we describe the method of the amorphous carbon films deposition, characterized by the simplicity of the plasma source design. The source is based on the planar HCD for easy variation of the film thickness and crystalline structure. The cathode/sample holder design and the I-V curve of the discharge for various pressures are shown in Fig. 1.

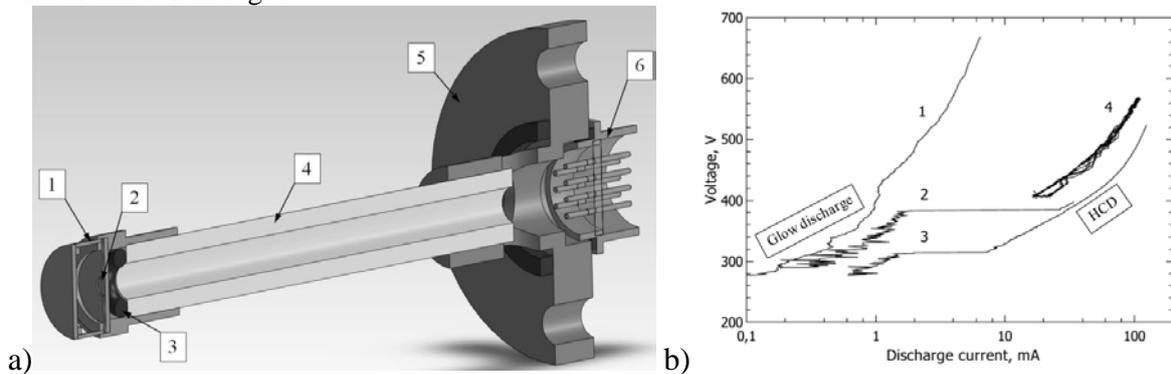


Fig. 1. Section view of the cathode/sample holder (a; 1 - cathode/sample holder, 2 - sample, 3 - heater, 4 - ceramic tube, 5 - flange, 6 - electrical feedthrough) and I-V curve of the discharge (b; 1 - 3 - Ar discharge at 9 Pa (1), 12 Pa (2) and 16 Pa (3); 4 - C₃H₈ (27 Pa)).

The I-V curve of the HCD is almost independent of the pressure over a wide range [9]. It allows us to develop the carbon deposition method with a good reproducibility (in this work it was confirmed by Raman spectroscopy and atomic force microscope measurements). Also, HCD allows to deposit a graphite layers with controlled SP²/SP³ ratio by three types of discharges [10] with different working discharge voltage (energy of bombarding ions).

As example of the PECVD application of the HCD we represent the synthesis method of nano-crystalline graphite films on Al₂O₃/Ni(111) samples. These films later can be used for testing the modes of heteroepitaxial synthesis of structurally homogeneous graphene for nanoelectronics [11]. The field emission of nano-crystalline graphite was measured. The presence of vertically aligned graphene is revealed.

This work was supported by the Russian Foundation for Basic Research (project no. 19-07-00432).

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THE INFLUENCE OF ION IRRADIATION ON STRUCTURE AND MECHANICAL PROPERTIES OF PRESSED PROFILES OF V95 ALLOY AFTER ARTIFICIAL AGING*

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The effect of 10-20 keV Ar⁺ ion irradiation on the mechanical properties and structural-phase state of hot-pressed thin profiles (1.5 mm thick) of V95 (Al-Zn-Mg-Cu) alloy after artificial aging ($T = 140^{\circ}\text{C}$, 16 h) was studied.

Irradiation of samples with continuous beams of Ar⁺ ions on the equipment for ion-beam implantation (ILM-1) with an ion source PULSAR-1M was carried out. ILM-1 equipment based on a glow discharge with a cold hollow cathode. The ion current density was 300 $\mu\text{A}/\text{cm}^2$ with the ion fluence of $2 \cdot 10^{15}$ and $1 \cdot 10^{16} \text{cm}^{-2}$.

Electron microscopy data (Transmission electron microscope – JEM-200 CX) of thin foils samples were obtained. Foils were prepared from cross sections parallel to the irradiated surface at a distance of $\sim 150 \mu\text{m}$ from the irradiated and non-irradiated surface.

It was shown that ion-beam treatment of the alloy in the used irradiation modes in the absence of significant heating of the samples ($T \sim 35\text{-}50^{\circ}\text{C}$) was not lead to a change in the tensile strength and yield strength, while the relative elongation increases slightly (by 1-2 %). This result is interesting, because the improvement of plastic properties while maintaining the strength can have a positive impact on the resource characteristics.

Electron microscopic study showed that under the influence of irradiation in the entire volume of samples there is a change in the grain structure. The elongation of the grains disappears, they become equiaxed. In addition, the irradiation leads to the transformation of the form of (Cu,Fe,Mn)Al₆ intermetallic crystallization origin. This is occurred in a decrease of the length of the slats to 0.1-0.2 μm and an increase of the number of equiaxed shape particles with a diameter of 50-70 nm. Irradiation of the samples were not affected on the size and volume fraction of fine particles of strengthening η' - and η - phases.

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EFFECT OF IRRADIATION ON THE STRUCTURE SUBJECTED TO SEVERE PLASTIC DEFORMATION OF THE 1461 ALLOY OF THE AL-Cu-Li- Zn SYSTEM*

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The effect of argon ion irradiation on structural and phase transformations of the 1461 alloy based on the Al-Cu-Li-Zn system subjected to severe plastic deformation (SPD) was studied by transmission electron microscopy.

The samples of the 1461 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 was ~ 400 μm . The samples were irradiated in continuous mode with Ar^+ ions ($E = 10$ keV, $j = 100$ $\mu\text{A}/\text{cm}^2$, $F = 6.25 \cdot 10^{15}$ и $2 \cdot 10^{16}$ cm^{-2}) by the ILM-1 implanter with the PULSAR-1M source. During the irradiation, the temperature of the samples was controlled, which did not exceed the value 180 °C at a maximum fluence.

The study of the structure and phase composition of the alloy was carried out by the thin foils method by JEM-200CX and Philips CM 30 Super Twin electron microscopes in the IPM UB RAS Center. Foils were made from samples by electrolytic thinning them on both sides. Thus, the information about the structural condition of the original and irradiated samples from their central part with the distance ~ 200 μm from the surfaces was obtained.

Electron microscopic study was showed that the combined (nanocrystalline + nanofragmentation) structure by SPD was formed. The nanocrystalline structure occupies almost the entire volume, the nanofragmentation structure occurs on separate parts of the sample. Nano-grains were the equiaxed shape with the either straightened or convex-concave boundaries. The diameter of recrystallized nano-grains is comparable to the diameter of nanofragments and varies from 20 to 50 nm. The contrast in the form of arcs or loops near the nano-grains with convex-concave boundaries was revealed. The heterogeneous nucleated particles of the T_2 (Al_3CuLi_5) phase on nano-grains boundaries and on remained fragments of dipole boundaries were observed. The diameter of the particles was not exceed of 5-10 nm.

The transformation of the structure of strongly deformed the 1461 alloy by the irradiation ($E = 10$ keV, $j = 100$ $\mu\text{A}/\text{cm}^2$, $F = 6.25 \cdot 10^{15}$ cm^{-2}) was caused. Large-scale dislocation clusters in the alloy structure were observed. In some areas, uniformly distributed dense tangles of dislocations was revealed, in others areas occurs plexuses with insignificant density. The diameter of nano-grains (from 20-30 to 50 nm) in areas with a high dislocation density by the electron microscopic images was obtained. In areas with low density, there is an almost homogeneous nano- and submicrocrystalline structure with a structural element size of 100-120 nm. Thus, the structure with the bimodal size distribution of nano-grains with low fluence irradiation was formed. The irradiation in this mode was not have a noticeable effect on the decay of the supersaturated solid solution of a strongly deformed alloy.

The increase in ion fluence up to $F = 2 \cdot 10^{16}$ cm^{-2} leads to the formation of homogeneous submicrocrystalline structure with the individual submicrocrystalline diameter of ~ 200 -500 nm on the 1461 recrystallized alloy. The sub-microcrystals with straightened the boundaries and the equilibrium triple junctions were obtained. In this case, there is almost complete disappearance of dislocations. The small increase of the size of T_2 -phase particles up 10 to 15 nm and the increasing of the density distribution on the volume of the sample with the increasing fluence were occurred.

Thus, it was found, that short-term (10-32 s) Ar^+ ions irradiation leads to the transformation of the microstructure of the 1461 alloy after SPD. The compare of the obtained results with the microstructure formed in this alloy after the traditional low-temperature annealing and the choose of the irradiation modes in order to form the optimal structure and properties of the alloy will be planned.

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THE INFLUENCE OF ION IRRADIATION ON MECHANICAL PROPERTIES AND LOW-CYCLE FATIGUE OF PRESSED PROFILES OF THE V95 ALLOY AFTER ARTIFICIAL AGING*

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The results of previous studies on the effects of ion gas beams on aluminum alloys [1] indicate that ion-beam treatment in addition to the replacement of intermediate annealing can be used to improve the mechanical and resource characteristics of aluminum semi-finished products at the final stages of processing. The favorable impact of ion-beam treatment on the resource characteristics of various materials is evidenced by some literature sources [2-5]. In this regard, it is important to study the laws of modification of the structure, phase composition and functional properties of industrial aluminum alloys in a different initial state (including the state of supply) under the influence of variable modes of ion-beam treatment.

In this paper, the hot-pressed profiles (PR100-23, thickness 6 mm) of high-strength alloy of the Al-Zn-Mg-Cu system after the hardening and artificial aging ($T = 140\text{ }^{\circ}\text{C}$, 16 h) were selected.

The irradiation of the massive billets of profiles (30 cm long) with continuous beams of Ar^+ ions was carried out by the equipment for ion-beam implantation (ILM-1) with an ion source PULSAR-1M was carried out. The irradiation was carried out on both sides of the samples when they were moved under the ion beam at different speed. When irradiated in mode 1, the energy of ions E was 20 keV, the ion current density j was $400\text{ }\mu\text{A}/\text{cm}^2$, the speed of the target v was 2.5 cm/s, the width of the collimator d was 2 cm, and the ion fluence F was $1 \cdot 10^{16}\text{ cm}^{-2}$. In mode 2: $E = 40\text{ keV}$, $j = 500\text{ }\mu\text{A}/\text{cm}^2$, $v = 1\text{ cm/s}$, $d = 10\text{ cm}$, $F = 9.4 \cdot 10^{16}\text{ cm}^{-2}$. The specimen temperature by the chromel-alumel thermocouple was controlled which was welded to the same sample-witness, and connected to the computer system for the measurement of digital signals by the ADAM-4000 module. At low ion fluence of $1 \cdot 10^{16}\text{ cm}^{-2}$ the temperature did not exceed of $40\text{ }^{\circ}\text{C}$, at ion fluence of $9.4 \cdot 10^{16}\text{ cm}^{-2}$ the samples were heated to $180\text{ }^{\circ}\text{C}$.

In determining the mechanical properties of the profiles in the initial state and after ion irradiation, standard tensile test methods were used (GOST 1497-84). Cyclic tests on the sinusoidal cycle with a loading frequency of 3 Hz were carried out (cycle asymmetry coefficient was -1). The Weller curves in the range of low-cycle fatigue on the basis of up to 10^5 cycles in order to assess the durability of the investigated aluminum alloys were constructed.

It was shown, that the irradiation of Ar^+ ions with the fluence of $1 \cdot 10^{16}\text{ cm}^{-2}$ was not lead to a change in the tensile strength and yield strength of the alloy, while the relative elongation increases slightly. There is no effect on low-cycle fatigue at the specified irradiation regime. The Weller curves constructed from the test results of the initial and irradiated samples are similar.

The increase in ion energy, ion current density and ion fluence, which leads to the heating of the samples during the irradiation, leads to a significant decreasing in the strength characteristics of the alloy, which was not meet the regulated requirements.

In the future, it is planned to conduct electron microscopic studies by the transmission electron microscopy of the structure and phase composition of the irradiated samples. And also continue to work of the search for optimal modes of the irradiation, which will allow to improve the resource characteristics while maintaining the regulated strength properties of the alloy.

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INFLUENCE OF ELECTROLYTE-PLASMA SURFACE HARDENING ON THE STRUCTURE AND PROPERTIES OF STEEL 40XH *

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Electrolyte-plasma hardening (EPH) is one of the methods of high-speed heating, in which the workpiece is a cathode or anode relative to an aqueous electrolyte. Depending on the heating mode, electrolyte composition, design parameters of the equipment, it is possible to produce hardening, chemical-thermal and thermocyclic processing of materials [1,3]. At the same time electrolytic-plasma hardening is the most economical and productive method. It is characterized by less energy consumption, simplicity of technological equipment and large size of the hardened zone. The advantages of the method are a sufficiently high productivity of the process and the ability to strengthen the details of a large mass and complex profile [3].

We studied the effect of electrolytic-plasma surface hardening on the structure and properties of steel 40KhN in this work.

Electrolyte-plasma surface hardening of the samples was carried out in a laboratory setup, designed and manufactured in the Research Center "Surface Engineering and Tribology." Samples were tested for abrasive wear using an experimental installation using the "rotating roller - flat surface" scheme in accordance with INDUSTRY STANDARD 23.208-79. University using the standard technique "ball-disc" (international standards ASTM G 133-95 and ASTM G 99) [4]. The wear tracks were studied using the MICROMEASURE 3D station contactless 3D profilometer. The elemental composition of the sample treated in electrolytic plasma was examined on a JSM-6390LV scanning electron microscope (JEOL, Japan), with the addition of an energy dispersive microanalyzer INCA Energy, from OXFORD Instruments. Measurements for microhardness were carried out on the device PMT-3 in accordance with INDUSTRY STANDARD 9450-76.

It was established that after ESP a modified layer with a thickness of 1-1.2 mm with high hardness and wear resistance is formed, consisting of a hardened layer of fine-grained martensite, an intermediate layer of perlite and martensite. It is established that after ESP microhardness and wear resistance of steel 40XH increases, depending on the processing mode. After an ESP with a heating time of 3 second, the microhardness increases up to 2 times, the wear resistance increases up to 30 times. High wear resistance of steels after ESP is associated with the formation of fragmented martensite with dispersed carbides.

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CALCULATION OF HEAT REGIMES FOR A NI-AL SURFACE ALLOY FORMED ON A CARBON STEEL SUBSTRATE WITH A LOW-ENERGY HIGH-CURRENT ELECTRON BEAM

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Heat resistant alloys based on nickel-aluminum are widely used nowadays in engineering technologies, for example, for the aircraft industry. They have high thermal conductivity combined with high strength at elevated temperatures. Abovementioned properties as well as its low density make NiAl alloy ideal for special applications like coating blades in gas turbines and jet engines^{1, 2}. Moreover, NiAl alloy possesses the high module of elasticity and high corrosion resistance, which gives an opportunity to use it like corrosion resistance coating.

To form the Ni-Al surface alloy on carbon steel the films of Ni and Al are deposited alternatively on a substrate of carbon steel (0.14-0.22% C; 0.15-0.3% Si; 0.4-0.65% Mn, 0.3%Ni; 0.3% Cr; Fe – balance, wt.%). Then this multilayer system is exposed to action of a low-energy, high-current electron beam (LEHCEB) which induces a liquid-phase mixing of deposited elements. The purpose of the work and, consequently, the calculations carried out was to determine optimal parameters of a LEHCEB for Ni-Al surface alloy formation on carbon steel and to investigate the effect of different thicknesses of layers of Ni and Al on the heat regime of a target. The latter is important because in terms of liquid-phase mixing the thin layers are more preferable, and for simplification of technological process the thicker layers are better.

Calculations were carried out for various multilayered systems with different thickness and numbers of the layers. For instance, the multilayered system of type 1 was three layered system Ni (0.5 μm)- Al (1.52 μm)- Ni (0.5 μm). The multilayered system of type 2 consists of 10 layers of Ni (0.11 μm each) and 9 layers of Al (0.167 μm each). The total thickness of coating deposited in both cases was 2.5 μm .

The melting thresholds for Ni, Al and carbon steel during LEHCEB irradiation were determined by the calculation. The phase diagrams obtained in calculation showed that process of melting occurs similarly for all types of multilayered systems. Melting starts in aluminum layers, then the melt appearing in substrate and finally - in the nickel layers. Layers of aluminum melt successively starting from the next to the irradiated surface. Melting of layers of nickel happens similarly. The calculations demonstrated that the melt thickness on the surface after irradiation with LEHCEB is about 3 μm , and the average lifetime of the melt is $\sim 1 \mu\text{s}$ (Ni- layers), and $\sim 10 \mu\text{s}$ (Al- layers).

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ELECTRICAL MODEL OF MICRO-ARC OXIDATION PROCESS*

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Micro-arc oxidation (MAO) is a complex plasma-chemical process of forming protective oxide coatings on metals and valve group alloys. Such coatings are used in many industries: aviation, rocket and space, textile industry, railway and transport engineering, instrument making, medicine, etc. At the moment there are several problems that impede the widespread introduction of this technology, in particular, the lack of knowledge of the phenomena occurring in the MAO process [1, 2].

Currently, there are a large number of works devoted to the mathematical modeling of the MAO process [3] - [6], but all of them describe only certain of its aspects, in particular, the phenomena occurring during the action of micro-discharges are not taken into account. In this paper, a model of the micro-arc oxidation process in the form of an equivalent replacement electrical circuit is proposed, which takes into account both the electrochemical and plasma-chemical processes that occur during MAO treatment, and an analysis of its work is carried out (Fig. 1).

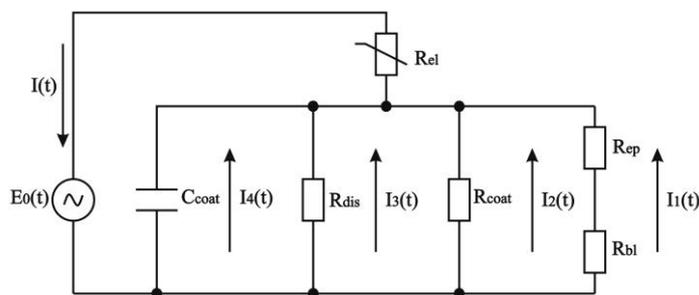


Fig. 1. The modified equivalent circuit of the MOE system: C_{coat} - coating capacity, R_{coat} - coating resistance, R_{bl} , R_{dis} , R_{ep} and R_{el} are the resistance of barrier layer, micro-discharges, electrolyte in the pores and thickness of the electrolyte, respectively; $E_0(t)$ - technological current source voltage, $I_1(t)$ and $I_2(t)$ are the electrochemical oxidation currents, providing growth of the barrier and porous layers, respectively; $I_3(t)$ - micro-discharges current, $I_4(t)$ - coating capacity charge current, $I(t)$ - technological current.

In Fig. 1 C_{coat} is the coating capacity, R_{coat} is the coating resistance, R_{bl} , R_{dis} , R_{ep} and R_{el} are the resistance of the barrier layer, micro-discharges, electrolyte in the pores and the electrolyte thickness, respectively, $E_0(t)$ is the technological current source voltage, $I_1(t)$ and $I_2(t)$ - electrochemical oxidation (ionic) currents, ensuring the growth of the barrier and porous layers, respectively, $I_3(t)$ is the micro-discharges (electronic) current, $I_4(t)$ is the coating capacity charge current, $I(t)$ is the technological current. R_{el} resistance is variable, which allows to take into account the concentration of electrolyte ions change with time (the phenomenon of the so-called “degradation”).

The values of the equivalent circuit parameters are calculated on the basis of the fundamental laws of physics and chemistry.

The proposed model, which takes into account both electrochemical and plasma-chemical processes, makes it possible to simulate the MOE system response to various technological parameters changes: current, voltage, shape and frequency of polarizing pulses, MAO process time and electrolyte components concentration.

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ACCELERATION OF HEAVY IONS IN THE HALL ACCELERATOR

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The process of acceleration of heavy ions in the hall accelerator is studied. Numerical solutions of one – dimensional hydrodynamic equations describing a three-component system-neutral particles, electrons and ions-are obtained. Ions move in a collisionless manner and are accelerated by a self-consistent electric field, electrons diffuse across the magnetic field. The self-consistent field is calculated using the Poisson equation, and it is shown that there is no singularity when the ion velocity coincides with the ion sound velocity. This is due to the refusal to use the exact quasineutrality condition (i.e.), see [1,2]. It is also shown that there is a critical magnetic field above which it is impossible to propagate the ion flow.

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EFFECT OF COMBINED TREATMENT ON MICROHARDNESS AND STRUCTURE OF HYPOEUTECTIC SILUMIN*

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Many scientific teams are working to improve the physical and mechanical characteristics of silumin. Multiphase composites based on the Al-Si system are being created [1]. The problems of the influence of plastic deformation on the tribological characteristics of the alloy AlSi9Cu3 are investigated [2]. In article [3], the influence of alloying elements and crystallization rate on the structure and mechanical properties of eutectic silumin was established.

The scientific work uses methods of the modern material physics to provide an analytic interpretation of the changes occurring in structure and mechanical properties of hypoeutectic AK10M2N silumin after combined treatment.

The as-cast AK10M2H silumin with the following elemental composition: ((9.5-10.5)Si, (2.0-2.5)Cu, (0.8-1.2)Ni, (0.9-1.2)Mg, (up to 0,6)Fe, (up to 0,05)Mn, (up to 0,05)Ti, (up to 0,05)Pb, (up to 0,06)Zn, (up to 0,01)Sn; the rest is Al, weight %) was used as the research material. The specimens had the shape of a parallelepiped with sides 20×20×10 mm. We used yttrium oxide powder Y₂O₃ as coating material.

The silumin specimens were exposed to the combined processing. The first stage involved application of composite coating of the system Al – Y₂O₃ by the method of electroexplosive alloying using the unit EVU 60/10. During the second stage the resulting coating was treated by high-current pulsed electron beam with parameters as follow: energy of accelerated electrons – 17 keV energy density – 25 j/cm² and 35 j/cm², pulse duration – 150 μs, number of pulses – 3. Modification of the surface was made using the unit ‘SOLO’.

The research material appears to be multiphase and morphologically diverse in its original state, aluminium and silicon-based solid solution being its principal phases. Surface modification of AK10M2N silumin by electroexplosive alloying results in formation of a composite coating of the system Al – Y₂O₃. Thickness of the modified layer is inhomogeneous, the average thickness of the coating varied in the range from 50 μm to 70 μm.

Subsequent treatment by electron beams results in the formation of a homogenized layer. The layer's thickness varies depending on parameters of electron beam treatment and electroexplosive alloying and achieves maximum value of 80 μm (fig. 1). It was shown that microhardness of surface layers of hypoeutectic AK10M2N silumin can increase by 5-8 times depending on the treatment mode.

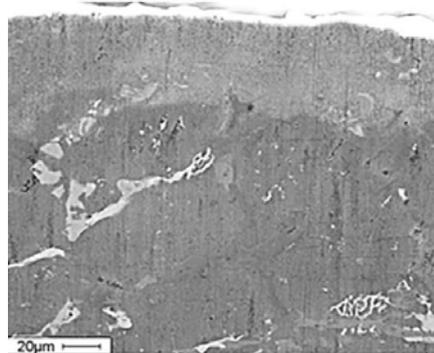


Fig. 1. Electron microscopic image of silumin structure after complex treatment

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COATINGS BASED ON CHROMIUM CARBIDE, DEPOSITED BY ARC SPUTTERING OF GRAPHITE AND CR-AL(SI) TARGETS*

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In the case of joint sputtering of graphite and composite M_xA_y targets, where M - transition metal of IIIB-VIB groups, A - element of the IIIA-IVA groups of periodic system, coating with an amorphous structure is formed [1]. The strong metal-carbon bonds increase internal stresses, reduce ductility and viscosity of coatings. In contrast, doping of carbon coatings with metals that do not form strong bonds with carbon (Cu, Al [2]) is accompanied by the formation of metal phases in the carbon matrix, which improve the viscosity, but reduce the hardness. Nevertheless, it is possible to obtain thermally stable coatings with low residual stress, high hardness and toughness by doping with non-metallic elements, for example, silicon [3]. This paper presents the results of study of CrAlC and CrAlSiC films deposited with physical vapour deposition (PVD) technique.

Transmission electron microscopy (TEM), scanning electron microscopy (SEM) and Raman spectroscopy were applied to investigate the structure of CrAlC and CrAlSiC coatings. The mechanical characteristics (hardness, elasticity modulus), friction and corrosion behavior of coatings were studied.

At the close carbon content in the films, the content of aluminum in CrAlSiC is higher. Despite this, the hardness and H^3/E^2 ratio of CrAlSiC is higher than these values for CrAlC. Friction coefficient of CrAlSiC is 0.04, CrAlC – 0.26. The feature of structure of CrAlSiC coatings leads to improving their mechanical and tribological properties. Structure of both coatings is amorphous-nanocrystalline. The formation in the films of Cr_2Al , $Cr_{1-x}Al_x(Si)C$ and the metastable CrC phases is possible. $Cr_{1-x}Al_x(Si)C$ phase is a result of Cr atoms partially replacement by Al due to the large its amount in coatings. The adding of silicon leads to the formation of silicon carbide nanograins. Nanograins of silicon carbide with a size of up to 30 nm were detected in CrAlSiC using transmission electron microscopy. Clusters of nanocrystalline graphite as spherical inclusions and plates, probably, of several graphene layers were found in CrAlC using Raman spectroscopy. Aluminum oxide, found in both coatings, makes their corrosion-resistant coatings.

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THE AXIAL VUV RADIATION INTENSITY DISTRIBUTION OF A GLOW DISCHARGE AND ITS APPLICATION FOR CREATION LUMINESCENCE CENTERS IN CRYSTALLINE MEDIA *

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The axial VUV radiation intensity distribution (<130 nm) of an air glow discharge at pressure 50 – 530 Pa was investigated by the thermoluminescent method using the CaSO₄·Mn thermoluminophor. The formation of thin layer of luminescent defects on the faces of planar lithium fluoride crystals located in the positive column (low-temperature plasma) and Faraday dark space of a glow gas discharge was studied.

The purpose of the study was to investigate axial VUV radiation intensity distribution of glow discharge at various pressures and voltages for creation color centers in wide-gap crystals. It was necessary to determine the types of the formed color centers, to study the spectral-kinetic characteristics of their luminescence, to reveal the mechanism of their formation and to identify the glow discharge zones in which defect formation was most effective.

Scanning confocal fluorescence microscope MicroTime 200 with picosecond time resolution with a spatially-selective time-correlated single photon counting was used to determine spectral-kinetic characteristics of photoluminescence of lithium fluoride (LiF) irradiated samples bandgap width, which is about 14 eV. Spectra of photoluminescence measured under excitation by picosecond laser with a wavelength of 470 nm were recorded by the spectrometer Ocean Optics 6500.

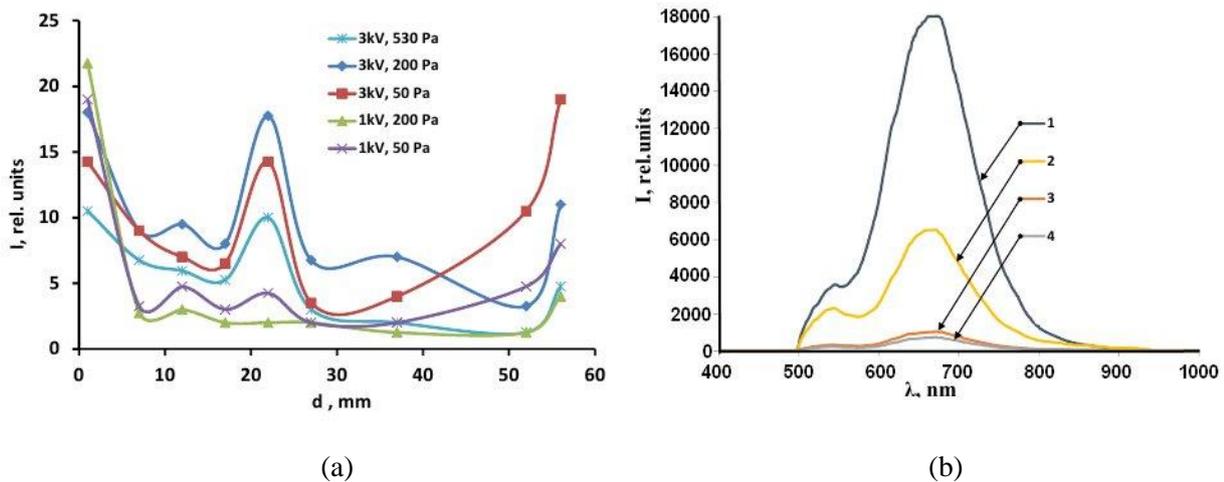


Fig. 1. Axial VUV radiation intensity distribution (a) (the anode was located on the left, the cathode on the right) and luminescence spectra (b) of irradiated crystals. (Excitation with picosecond laser pulses at a wavelength of 470 nm, light filter with a transmittance from 500 nm. The curves (1) and (3) contribute to a specimen in the positive column. (2) and (4) – in the Faraday dark space, (1) and (4) – for the anode side (turned towards the anode) and (2), (3) – for the cathode side.)

Using the thermally stimulated luminescence method we obtained the VUV-radiation distribution of the discharge (Fig. 1a). The obtained results give a picture of the distribution of VUV radiation with a wavelength shorter than 130 nm. The VUV radiation intensity distribution has maximum values in the near-electrode regions of the discharge in the pressure range of 50–530 Pa and voltages of 1–3 kV. The stratified positive column of glow discharge is also a region of intense VUV radiation at high voltage.

As follows from Fig. 1b, there are two types of luminescent centers formed in crystal during irradiation in a glow discharge, namely, F₃⁺ - and F₂⁻ color centers with luminescence band maxima at 540 and 680 nm and decay time constants of 7.1 and 17.9 ns. By using a MicroTime 200 time resolution laser confocal scanning luminescence microscope (PicoQuant GmbH) it was shown that the highest concentration of luminescent defects corresponds to the sides of the crystals that were irradiated near the electrodes.

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TWO-STAGE SUBNANOSECOND PLASMA SWITCH*

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An experimental investigation of high-current high-voltage switches based on the "open" discharge with the generation of counter-propagating electron beams – kivotrons is carried out. The possibility of operation of such gas discharge devices in a two-stage of nanosecond pulses compression circuit at a voltage up to 25 kV and a repetition rate up to 12 kHz pulses with forced air cooling in the regime of regular pulses at the resistive load of 20 Ohm is demonstrated. The total pulse compression degree in two stages reached 300-600 with pulse edge less than 200 ps. The average power at the active load after the second stage of the compressor reached 500 watts. High efficiency of nanosecond pulse compression in comparison with analogues based on other physical principles is demonstrated. In this work the main operation parameters of the kivotrons in the regime of increased pulse repetition rate are considered and optimized, as well as circuitry features of their operation with cascade connection and a different type of load for each stage of the pulse compressor.

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EFFECT OF ADDITIONAL ION BOMBARDMENT ON THE QUALITY OF VACUUM ION-PLASMA COATINGS

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The characteristics of multilayer vacuum ion-plasma coatings Ti-TiN are investigated. It is shown that additional ion bombardment contributes to obtaining high-quality coatings. Ion bombardment has a significant effect on the state of the surface layer of the metal. In the process of ion bombardment, conditions are created for the formation of active adsorption centers and fine-grained structure, nanoscale grains and layers [1,2].

The characteristics of the surface quality with a vacuum ion-plasma coating under conditions of ion bombardment largely depend on the initial surface, as well as on preliminary preparation before deposition of coatings [2, 3].

The purpose of this work is to study the effect of additional ion bombardment (AIB) at the stage of preliminary surface cleaning and deposition of coatings on the adhesion strength of synthesized coatings of the Ti-TiN system.

To implement additional ion bombardment, a «PINK» plasma source was used, which implements a non-independent high-current diffusion discharge. It was used both at the stage of pretreatment and surface activation, and for the synthesis of protective coatings in the plasma assisting mode. The use of «PINK» allows you to significantly increase the adhesive strength and form a better coating, as well as to reduce the temperature of the formation of layers.

The formation of multilayer coatings is implemented by sequential deposition of Ti and TiN from arc plasma.

Conducting AIB plasma source of «PINK» allows you to: provide high energy efficiency of the process of generating low-temperature bulk plasma; reduce the fraction of the micro-droplet fraction in the plasma flow of vacuum electric arc evaporators; perform plasma cleaning, etching and surface activation without spraying with cathode material vapors; realize complex processing of products in a single vacuum cycle, including the processes of final cleaning, activation, plasma-assisted spraying of functional coatings; to ensure the formation of micro- and nanostructured coatings with high hardness, increased wear resistance, improved corrosion resistance.

The adhesion of the coating material to the base was investigated by two methods: - by bending of witness samples and - by the imprint of a diamond pyramid when measuring microhardness. Tests showed excellent adhesion of the deposited coatings. In the first case, when bending a stainless steel plate, there were no chipping and peeling of the surface layer at the bend site. The interaction of the diamond pyramid with the surface on which the coating was based on carbon and silicon was also not observed detachment and chipping.

The resulting surface layers have high adhesion and are able to withstand repeated bending, which was confirmed on samples of witnesses.

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DESIGNING OF EQUIPMENT FOR THE SYNTHESIS OF COATINGS FROM NITRID AND CARBIDES OF INTERMETALLIDE TI - AL SYSTEMS BY CONDENSATION OF PLASMA FLOWS GENERATED WITH VACUUM ARC

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The formation of new materials of coatings with high physicochemical properties on the surface of parts and the development of technologies for their deposition is one of the promising areas of industrial development. Such materials include multicomponent materials based on intermetallic compounds, which have unique properties and retain an ordered structure up to the melting temperature. The Ti – Al system is one of the most promising in the field of creating nanostructured metal-intermetallic composites, primarily due to the low density of raw materials and their wide use in engineering.

The article discusses the results of research on the production of coatings formed by a vacuum arc with an integral cold cathode. The results of the interaction of titanium and aluminum flows on cold and hot substrates in the environment of various gases are analyzed.

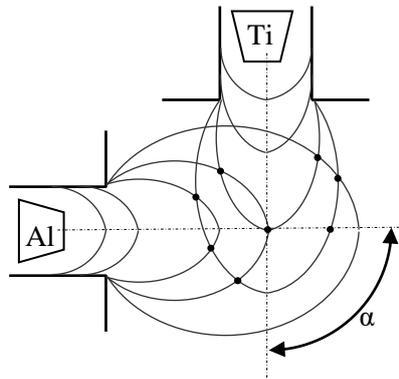


Fig. 1. The scheme of the experiments

Analysis of the research results [1-6] showed that the phase composition of the coatings on the samples, namely, the percentage of intermetallic, as well as their carbides and nitrides in the coating depends on (Fig. 1):

- spatial location of samples (distance to electric arc evaporators, angle between plasma axis and substrate surface);
- additional bombardment of the substrate surface with ions of the reaction gas;
- processing modes (the ratio of the arc current Ti and Al evaporators, respectively, pressure, ion energy).

The obtained data will make it possible to formulate a technical assignment for designing installations for the synthesis of coatings from nitrides and intermetallic carbides of Ti-Al systems of the required phase composition.

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GENERATION OF THE COLD PLASMA JET OF ATMOSPHERIC PRESSURE IN HELIUM AND ARGON*

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The results of an experimental study of the generation, spectral and temperature parameters of a plasma jet, which was generated in a dielectric channel in a stream of inert gas — helium or argon, propagating in a gas stream outside the channel, are presented. Gas-discharge device consisted of a quartz tube channel 100 mm long with an inner diameter of 8 mm, in which a metal electrode was coaxially placed, and replaceable capillary inserts 6 mm long with an inner diameter 0.9, 1.5, 2.3, 2.6, 3.2 or 3.77 mm. Outside the quartz channel, there was a movable copper ring electrode. The discharge zone was formed by potential – loaded and external grounded electrodes. The gas system provided the flow of working gases with typical flow rates of 0.5–10 l/min with an overpressure in the gas line of 1.5 atm. A sinusoidal voltage with a frequency of 15-30 kHz and an amplitude of up to 6 kV was used. When the working gas was pumped and the voltage U was applied between the loaded and grounded electrodes, a breakdown was observed at a positive half-wave and with further increase of U over 1 kV in helium and 3 kV in argon a plasma jet was formed, which was a sequence of streamers that fell out of the dielectric channel and propagated in a free space.

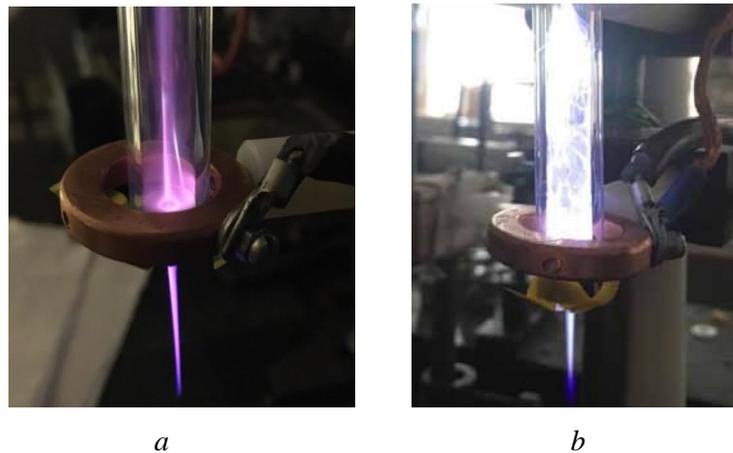


Fig. 1. Photos of plasma jet in helium (a) and argon (b)

Studies were carried out to the dependence of the geometric parameters of a plasma jet in space on the geometry of the dielectric channel and capillaries, the flow rate and voltage in order to increase the volume of plasma formation. Spectral studies measuring the emission of a free jet of helium or argon showed the presence of intense N_2 , N_2^+ , NO, OH lines, Balmer series lines of hydrogen and helium or argon lines in plasma volume. The spectral composition of the mixture varied with the interaction of the jet with objects of different nature. Temperature measurements showed that the jet raised the temperature in the zone of contact with the surface by a fraction of a degree and it didn't not exceed 40°C.

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EFFECT OF COLD PLASMA JET OF ATMOSPHERIC PRESSURE IN HELIUM ON LUNG HUMAN ADENOCARCINOMA CELLS*

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The results of an experimental study of the effects of a cold plasma jet generated in a dielectric channel in an inert gas flow - helium on human lung adenocarcinoma A549 cells are presented.

The discharge device was a quartz coaxial channel 100 mm long with a diameter of 8 mm with an internal electrode and a capillary 6 mm long with a diameter of 2.6 mm. The discharge zone was formed by potential – loaded and external grounded electrodes. The gas system supplied helium at a gas flow rate of 0.5–5 l/min. When the working gas was pumped and a sinusoidal voltage was applied at a frequency of 25 kHz and an amplitude of up to 6 kV, a breakdown was observed between the loaded and grounded electrodes on a positive half-wave, and with a further increase of U over 1 kV a plasma jet was formed in the helium flow. The plasma formation spectrum was characterized by the presence of intense N_2 , N_2^+ , NO, OH lines, Balmer series hydrogen lines and helium lines in the plasma. The jet increased the temperature in the contact zone with the surface by fractions of a degree and it did not exceed 40°C.

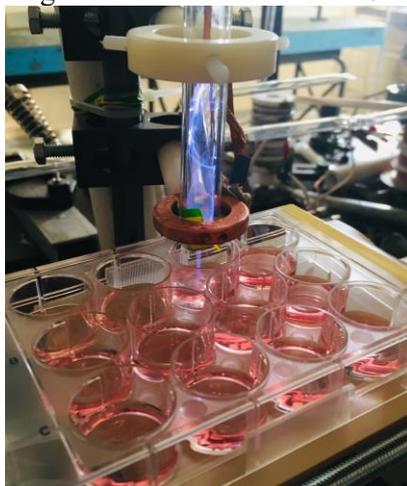


Fig. 1. Photos of the effects of plasma jet in helium on biological objects.

Experiments have shown that human A549 lung adenocarcinoma cells grown in culture dishes were subjected to cold plasma jet ionization (helium) during of 5 to 120 seconds under 3kV and 2.5 L/min argon gas flow. The decreasing of cell viability was observed in plasma-treated cells after 24 h of treatment. This effect was time-dependent with highest value for the cells of 2 min of plasma application.

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RADIATION-AND-THERMAL EXPOSURE OF ACCELERATED IONS ON FERROMAGNETIC ALLOYS OF $\text{Fe}_{100-x}\text{Mn}_x$ and $\text{Fe}_{100-x}\text{Cr}_x$ AFTER THEIR MEGAPLASTIC DEFORMATION IN COMPARISON WITH PURE THERMAL EXPOSURE*

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In [1], an anomalous acceleration of atomic delamination was observed at warm ($T < 0.3 T_{melt}$) ultrahigh megaplastic deformation in rotating Bridgman anvils and a ball mill. The accelerated delamination induced by deformation at 573 K is compared with irradiation by high-energy electrons in a close temperature range. This effect is explained by the mechanism of dynamic aging caused by the continuous generation of a large number of moving point defects in the bulk of the material. In the course of high-energy irradiation, the process of defect formation permeates the entire volume of the studied targets and the classical migration mechanisms of radiation physics can be used to explain.

Recently, numerous studies (see review [2]) have established the important role of nano-scale dynamic effects in the effect of ionizing radiation on condensed matter. These effects, which take place during irradiation with accelerated ions (as well as reactor neutrons, fission fragments), remain outside the field of vision of classical radiation physics. They are not related to the migration process of defects, since, for example, the paths of heavy ions with energies from several units to several tens and hundreds of keV in condensed media do not exceed 1 μm . At the same time, the scale of their impact over the depth of targets reaches values from several tens and hundreds of micrometers to several millimeters. The temperature of the initiated processes decreases by tens and hundreds of degrees as compared with the thermally activated processes and, despite this, their speed increases by several orders of magnitude in the whole volume of the substance, where ions do not penetrate and there are no defects.

The authors of [2, 3] associate these effects with the processes of explosive energy release in the areas of passage of dense cascades of atomic displacements with the formation, within trillionths of a second, of nanoscale zones (thermal spikes) with gigantic temperatures and pressures ($T = 3000 \div 6000 \text{ K}$, $P = 5 \div 40 \text{ GPa}$), in some cases exceeding the theoretical yield strength of materials. As a result, these zones emit post-cascade powerful elastic and shock waves, capable of carrying out liquid flow of condensed media on their front, initiating structural-and-phase transformations. Radiation-shaking of a medium by post-cascade waves can play the role of temperature, increasing the mobility of atoms without heating the medium.

In view of the above, experiments were designed and carried out to study the behavior of highly nonequilibrium (metastable) media under conditions of purely thermal exposure and under conditions of ion beam irradiation with accurate reproduction of thermal exposure modes. Samples of $\text{Fe}_{100-x}\text{Mn}_x$ ($x = 4 \div 10$) and $\text{Fe}_{100-x}\text{Cr}_x$ ($x = 12 \div 22$) alloys were subjected to cold ($T \sim 0.2 T_{melt}$) and warm ($T < 0.3 T_{melt}$) ultrahigh (mega plastic) deformation in rotating Bridgman anvils and ball mill. Comparison of purely thermal and radiation-thermal effects (beams of heavy accelerated radiation: Ar^+ , Kr^+ and Xe^+ with energies of 5 \div 40 keV) using temporal diaphragms ($\tau = 0.001, 0.01$ and 0.1 s) showed that the formation of the short-range atomic order in initially metastable alloys $\text{Fe}_{100-x}\text{Mn}_x$ and $\text{Fe}_{100-x}\text{Cr}_x$ are accelerated many times during radiation-and-thermal of accelerated ions exposure compared to purely thermal.

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MICRODIFFRACTIONAL ANALYSIS OF THE STRUCTURE OF HIGH-SPEED CELLULAR CRYSTALLIZATION OF SILUMINE*

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The purpose of this work was to establish the laws of the formation of the structural-phase state of hypoeutectic composition silumin subjected to irradiation by an intense pulsed electron beam. Silumin AK10M2N was used as the study material. The silumin subjected to electron-beam treatment at the “SOLO” setup [1] with the following parameters: the energy of accelerated electrons is 17 keV; electron beam energy density of 25 J / cm²; irradiation pulse duration 150 μs; number of pulses 3; pulse repetition rate 0.3 s⁻¹. Irradiation was carried out in argon plasma at a pressure of 0.02 Pa. Studies of the elemental and phase composition, defective substructure of silumin samples in the cast state and after irradiation with an electron beam were carried out using transmission diffraction electron microscopy (JEM 2100F).

It has been established that, in the cast state, the material under investigation has a multiphase structure, represented by aluminum-based solid solution grains, eutectic grains, and intermetallic inclusions. The presence in the material of inclusions plate and needle shape significantly reduces the performance characteristics of the material [2]. The irradiation of silumin with an electron beam leads to the melting of the surface layer up to 35 μm thick and to the dissolution of inclusions of the second phase. Subsequent high-speed crystallization is accompanied by the formation of a cellular structure (Fig. 1, a). Nanosized particles of the second phase are located in the volume of cells and along their boundaries (Fig. 1, b – d). It is shown that the formation of a submicro-nanoscale multiphase structure leads to an increase in the wear resistance of the material by ≈1.7 times, microhardness - by ≈1.2 times.

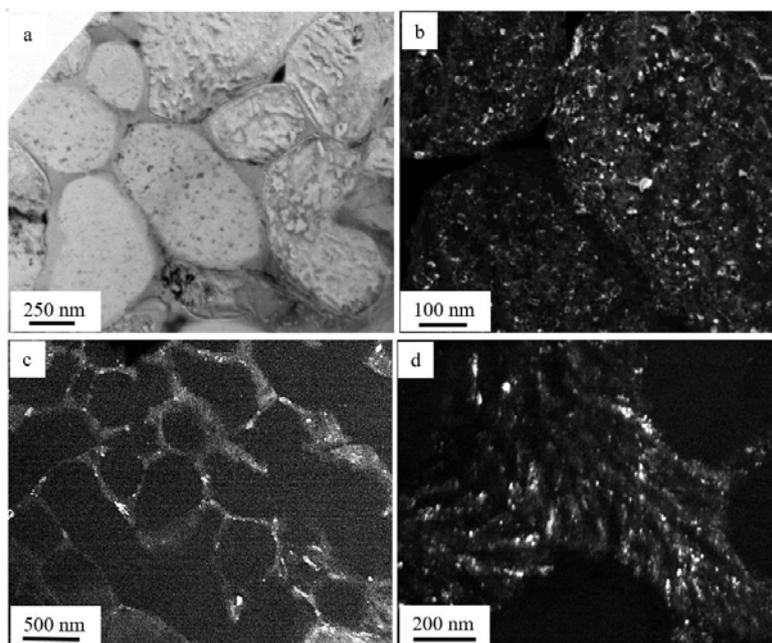


Fig. 1. Electron-microscopic image of silumin structure subjected to irradiation by an intense pulsed electron beam; a - bright field; b – d dark fields obtained in silicon reflexes.

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GASEOUS DISCHARGE PLASMA SWITCHING IN OVERSIZED INTERFERENCE MICROWAVE SWITCHES*

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The report presents the results of the experimental study of gaseous discharge plasma switching in interference oversized switches of X-band active resonant compressors. Switching in the spontaneous breakdown mode was initiated by considerable discontinuity at an electric field antinode in the switching arm. The study was conducted for the interference switch made of the rectangular waveguide with the cross section area of 50×25 mm² [1] and for the switch with the combination of the rectangular waveguide of 50×25 mm² cross section and the circular waveguide having the diameter of 50 mm [2]. At plasma gas-discharge switching the threshold nature of effective acting initiated by discontinuity in the form of a thin copper wire introduced into the arm through the cut-off frequency waveguide was found. The switching efficiency as a function of the wire length was determined. The conditions for effective switching with the sequential cascade of the oversized switches are obtained. It is shown the effective switching in oversized interference microwave switches is possible and may be identical to switching in the single-moded switches.

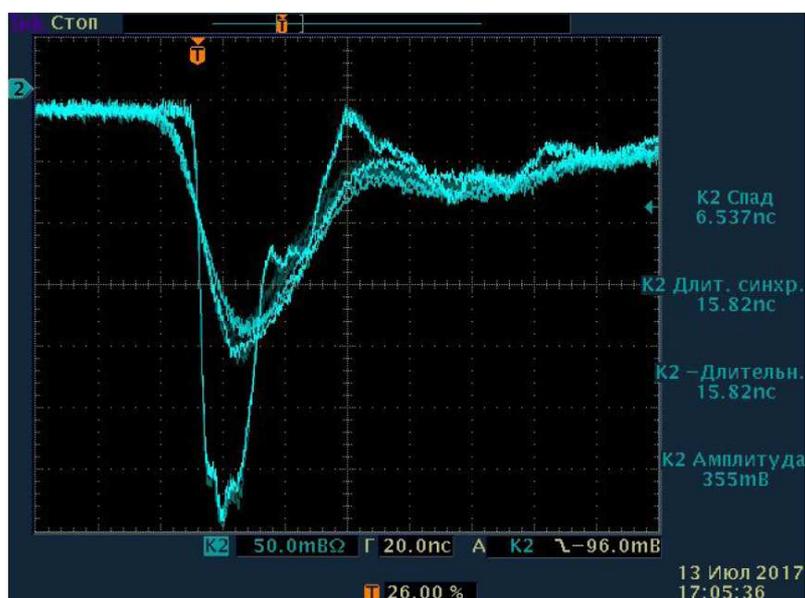


Fig. 1. The oscillogram showing the change in the parameters of the output pulse of the active resonant microwave compressor when the threshold is passed. The interference switch was made of oversized waveguides

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STUDY OF METAL POWDER RECEIVED BY PLASMA SPRAY

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Additive technologies make it possible the production of complex in form and non-technological products at the present level of development of the industry, using universal equipment. Additive technologies are recognized by the leading countries in the world as the main advanced industrial technologies. The Russian Federation has significantly lagged behind in the development of additive technologies from the United States, Japan, Europe, China and Israel. In the medium term, Russia risks becoming dependent on advanced industrial technologies of Western countries and losing its technological sovereignty. In this regard, the development of this technology in our country and the production of raw materials for additive equipment (powders) is today an urgent task.

The problem of obtaining metal powders of various nomenclatures with the required properties can be solved using a highly concentrated heat source, in particular, a plasma jet. A distinctive technological feature of the use of a plasma jet is a higher concentration of the energy of the heating source and its forceful effect on the heating zones. At the same time, the influence of plasma characteristics on the properties of the sprayed materials is obvious.

In this paper, a new method for producing metal and ceramic powders by plasma spraying of rod blanks is proposed. A distinctive feature of this method is the use of a direct-acting plasmatron, where a bar-stock acts as one of the electrodes, and the cooling of the molten particles takes place with a water screen. The water screen is not only in the lower part of the installation, but also flows evenly from the walls, which gives a more efficient cooling of all molten particles, forming a regular spherical shape, and a plasma flow. Water constantly circulates in a closed circuit, the heat is removed in a heat exchanger.

Figure 1 shows of a powder sample obtained by plasma spraying:

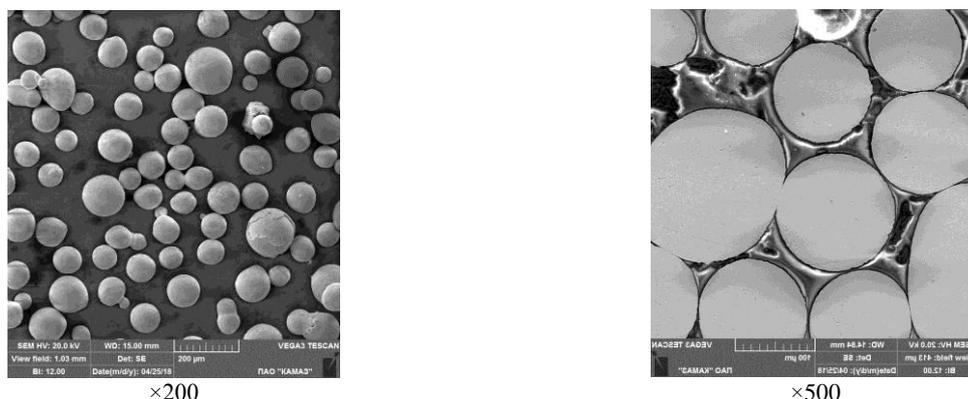


Fig. 1. The powder sample obtained by plasma spray.

The plasma spraying makes it possible to obtain a metal powder size of 5-200 microns.

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APPLICATION OF LOW-PRESSURE GLOW DISCHARGE IN TRANSVERSE SUPERSONIC GAS FLOW

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Article presents application of glow discharge at low pressures in transverse supersonic gas flow limited in part of discharge region. Mathematical model of supersonic flow in a vacuum chamber described. Results of experiments on realization of glow discharge at low pressures due to the organization of a transverse supersonic gas flow are given.

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INFLUENCE OF DIFFERENT PLASMA INITIATION WAYS ON OBTAINING ULTRADISPERSED SILICON CARBIDE

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Silicon carbide (SiC) has been used in many fields of human activity: power electronics, the production of abrasive materials and ceramic products for working in corrosive environments. SiC is used due to its properties: high hardness and wear resistance, wide forbidden zone, refractory [1-2]. The characteristics of the materials can be improved by using nanoparticles in their production.

Silicon carbide synthesis techniques are not effective enough, because they have unsatisfactory dispersity, high duration and other. The synthesis of ultradispersed SiC was carried out on a coaxial magnetoplasma accelerator (CMPA) [3]. This work is aimed at studying influence of different plasma initiation ways on the phase composition of the SiC. It was assumed that the method of initiation of the arc discharge will affect the phase composition of the synthesis product by changing the time and nature of the transition of the mixture of precursors to the plasma state.

The series of experiments were conducted with a different plasma initiation ways: carbon fibers and graphite aerosol (graphitization) . The precursors in experiments were carbon black and silicon powder, which were mixed. 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).

The result of this work is the optimization the plasma dynamic synthesis of nanoscale cubic silicon carbide using different plasma initiation ways. According to XRD and TEM, the SiC plasma dynamic synthesis proceeds more fully in the case of using graphitization as a plasma initiation way due to the more effective sublimation of precursors during preionization time. In this case product contains a phase of cubic silicon carbide β -SiC (~99%). Important advantage of the graphitization way is the high processability, which ensures the simplicity of the accelerator preparation and the reliability of its operation.

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OBTAINING NANO-DISPERSE SOOT FROM ORTHOXYLENE BY THE HIGH-VOLTAGE AC PLASMA TORCH*

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Carbon structures are used as a reinforcing component in the production of rubber and plastics. About 70% of all manufactured soot is used in the production of tires, ~ 20% is used in the production of rubber products. The remaining amount is used as a black pigment; retarder aging plastics; component that gives plastics special properties.

There are several industrial methods for producing soot. The basis of all is thermal (pyrolysis) or thermo-oxidative decomposition of liquid or gaseous hydrocarbons. Depending on the raw materials used and the method of its decomposition, the following types of soot are distinguished: furnace, lamp, thermal and channel. High quality carbon and fullerene-containing soot are the result of plasma pyrolysis of hydrocarbons. In most cases, DC torches are used for this; however, high-voltage [1] and low-voltage [2] AC devices can also be used to solve these problems. These plasma torches showed high efficiency in the processing of biomass [3], natural gas [4] and organochlorine substances [5].

The report discusses the continuous process of soot production using an AC high-voltage plasma torch. It consists of three electric arc channels with three graphite rod electrodes [6]. The plasma torch is powered by an alternating current power source with a 10 kV open circuit voltage [7]. The power supply consists of a high-voltage transformer (380/10000 V), current-limiting inductances, a reactive power compensator and a system for continuous measurement of electrical parameters. The main plasma gas is argon. Evaporated orthoxylene (boiling point is 144.4 °C) was fed to the heated argon stream. The reagent flow rates were as follows: argon - 1 g/s, orthoxylene - 0.1 g/s. In electric arc channels, the hydrocarbon quickly decomposes to soot. The properties and composition of produced carbon structures were studied using a FT-IR spectrometer, a scanning electron microscope, an elemental analyzer (CHNS), and an X-ray diffractometer. The particle size distribution was determined by the method of dynamic light scattering. It was found that in the products there are two fractions: the main fraction with a concentration of about 70% and a particle size of from 80 to 120 nm and the second fraction with a particle size of from 200 to 1500 nm. The first fraction is the product of the thermal plasma decomposition of orthoxylene, and the second fraction is formed as a result of the erosion of graphite electrodes.

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THE INFLUENCE OF THE MAGNETIC FIELD IN ION NITROGENING ON PROBE CHARACTERISTICS, MICRO-HARDNESS AND STRUCTURE OF AISI M2/R6M5 STEEL

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Ion nitriding is one of the effective ways of surface hardening of steels [1]. In the temperature range of 450-550 ° C, the process is carried out with long exposures. This is due to the low rate of diffusion of nitrogen into the material. The diffusion rate can be significantly increased by various methods of intensifying the process [2, 3]. It is known [3] that the use of a magnetic field in nitriding in a glow discharge allows one to increase the energy of charged particles in a plasma. This accelerates the process of interaction of ions and nitrogen atoms with the surface of the material.

In this work, we study the effect of a magnetic field on nitration in a glow discharge on probe characteristics, microhardness, and the structure of AISI M2 steel. Using a magnetic field on nitration in a glow discharge increases the surface hardness and thickness. Experimental probe current-voltage characteristics in a glow discharge with magnetic fields are obtained.

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THE INFLUENCE OF THE MAGNETIC FIELD IN ION NITROGENING ON PROBE CHARACTERISTICS, MICRO-HARDNESS AND STRUCTURE OF AISI 321/08H18N10T STEEL

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Ion nitriding is one of the effective ways of surface hardening of steels [1]. In the temperature range of 450-550 ° C, the process is carried out with long exposures. This is due to the low rate of diffusion of nitrogen into the material. The diffusion rate can be significantly increased by various methods of intensifying the process [2, 3]. It is known [3] that the use of a magnetic field in nitriding in a glow discharge allows one to increase the energy of charged particles in a plasma. This accelerates the process of interaction of ions and nitrogen atoms with the surface of the material.

In this work, we study the effect of a magnetic field on nitration in a glow discharge on probe characteristics, microhardness, and the structure of AISI 321 steel. Using a magnetic field on nitration in a glow discharge increases the surface hardness and thickness of the hardened material layer by 1.5 times. Experimental probe current-voltage characteristics in a glow discharge with magnetic field are obtained.

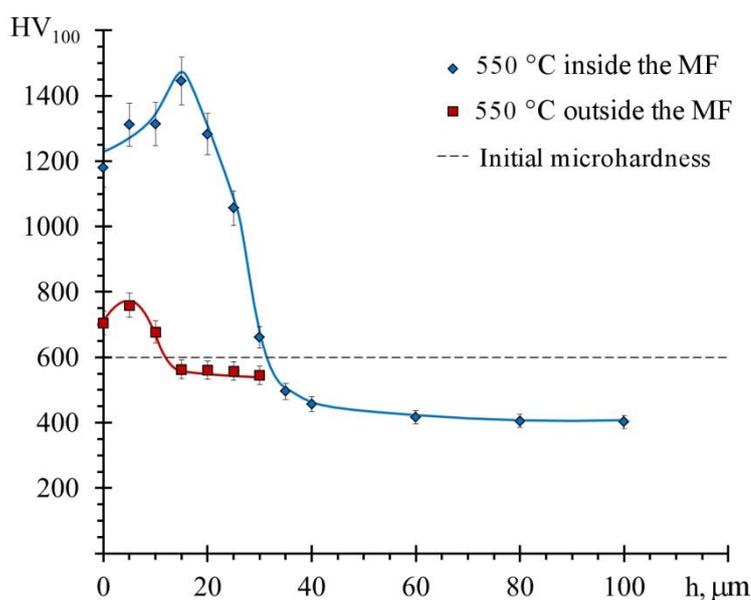


Fig. 1. Graph of microhardness distribution by depth of samples nitrided at 550 ° C

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MODELLING OF MOSSBAUER SPECTRA OF LAYERED METAL SYSTEMS OBTAINED BY ION-PLASMA SPUTTERING

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Ion-plasma deposition allows the formation of layers with high adhesion and the necessary chemical composition. Equilibrium diagrams [1] of the binary systems Fe–Sn and Fe–Zr are characterized by the existence of regions of the solid solution α -Fe(Sn) and α -Fe(Zr) with various intermetallic compounds with increasing of temperature.

High corrosion resistance in combination with mechanical strength, high melting point and low effective cross section for the absorption of thermal neutrons have recently made extensive use of Zircaloy (alloy of Zirconium with Iron and Tin) in reactor engineering. The study of thermally induced phase formation in layered systems Sn-Fe and Zr-Fe is relevant. Simulation of the phase state in layered systems will make it possible to predict changes during thermal treatment.

The layered systems based on Iron, Tin and Zirconium are considered. Using the MSTools software package [2], the spectra of Iron and Tin nuclei were obtained for each position in the phases of binary systems, and taking into account the occupancy of the positions, the standard spectra of each phase were modeled. Next, using the lever rule, the spectra of alloys with different concentrations of the second component were calculated for the reference points of the state diagrams (see Fig.1). A comparison of the simulated spectra with the experimental Mössbauer spectra of two-layer systems (Zr-Fe and Sn-Fe) and the three-layer system Sn-Zr-Fe, obtained by ion-plasma sputtering and subjected to thermal annealing, showed a good correlation.

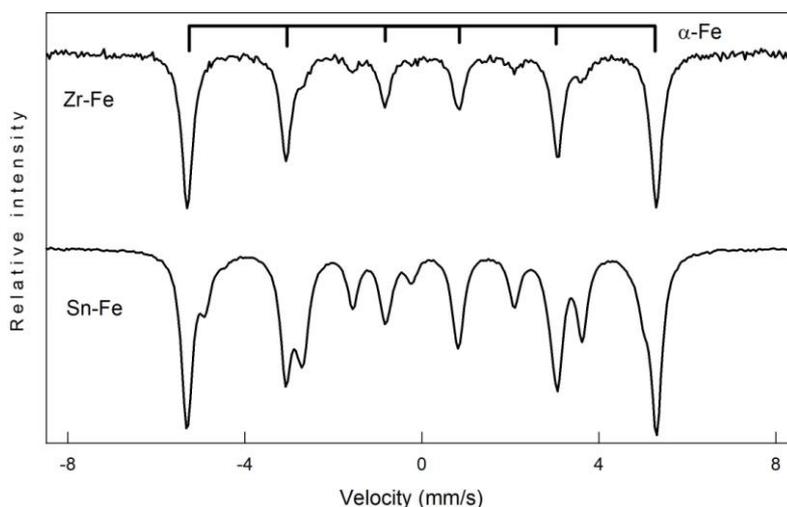


Fig. 1. Simulated spectra of binary system alloys

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EXPERIMENTAL DETERMINATION OF THE OPTIMAL FOCUSING ZONES FOR LASER IGNITION OF BUTANE-AIR COMBUSTIBLE MIXTURES

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Laser ignition is widely discussed and investigated subject today. Recently, in this area of research significant progress has been made. Though laser spark plugs already have practical application, nevertheless the main aspects of ignition of fuel mixtures by means of the laser, for example, the most advantageous position of focus in combustion chamber not fully explored. There are two modes of focusing of laser beam in combustion chamber: focusing in air in the volume of the chamber and focusing on the ablator. Fuel ignition at the first mode of focusing requires high power consumption, increasing the cost and the sizes of laser spark plug, at the second mode of focusing of energy of laser pulse for ignition of fuel it is required 10 times less, however the resource of the ablator is obstacle for realization of this method in practice. Therefore it is necessary to investigate the optimal zone for focusing of laser radiation in which it is possible to realize low energy-intensive ignition and at the same time without destroying the ablator.

In the work results of experiments on ignition fuel mixture (butane based) compositions with various equivalence ratios ($\varphi \sim 0.4-1.1$) and pressures ($p \sim 1-3$ bars) depending on the position of the focus (1 ~ 0-12 mm) in combustion chamber concerning the ablator are presented. The change in the minimum energy of the laser pulse required for ignition the fuel mixtures is fixed and also with use shliren's method dynamics development of burning core and propagation of shock wave at laser ignition (1064 nm, 12 ns) was investigated. It was revealed that the optimum zone of focusing of laser radiation where energy of laser pulse accepts the minimum values, was not on the ablator, and in some removal from it.

The received results are of interest to use of laser ignition in the practical purposes.

ACTION OF SUBNANOSECOND PULSED ELECTRIC FIELD ON SCOV-3 AND JURKAT CELLS*

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The effect of the pulsed electric field of the subnanosecond range on human tumor cells Scov-3 and Jurkat is studied. Studies of the effects of nanosecond electric fields on cells are widely carried out [1], but there is no data on the effect of the duration of the pulse rise time on the processes leading to cell death. The use of pulsed electric fields with a subnanosecond rise time can effectively affect the internal structures of the cell, since the polarization time of the outer cell membrane is ~ 1 ns. These fields can be used in the development of new methods for the treatment of human tumor diseases.

The experimental setup (Fig. 1) consists of a coaxial waveguide with a 50Ω matched load, a transmission line, and a pulse voltage generator. Cell culture with a volume of 0.15 ml in suspension or on a substrate is placed in the end part of the waveguide. The source of voltage pulses is the FID2/25 generator. Pulse duration is 7 ns, amplitude $\pm 12..25$ kV, voltage rise time 150 ps, frequency up to 3 kHz. Trigger generator G5-26 is used. Measurements of the electric field parameters in the transmitting cable and in the working volume were carried out using wideband capacitive dividers. The amplitude value of the electric field strength in a cuvette is $15..30$ kV/cm. The pulse shape is in Fig.2. The absence of electrical discharge phenomena was controlled by spectral radiation registration with use of spectrometer Ocean Flame-S. The geometry of the mismatched part of the waveguide and the cell with the sample provides the passage of high-frequency TEM-wave modes up to 3 GHz into the working volume without noticeable distortions.

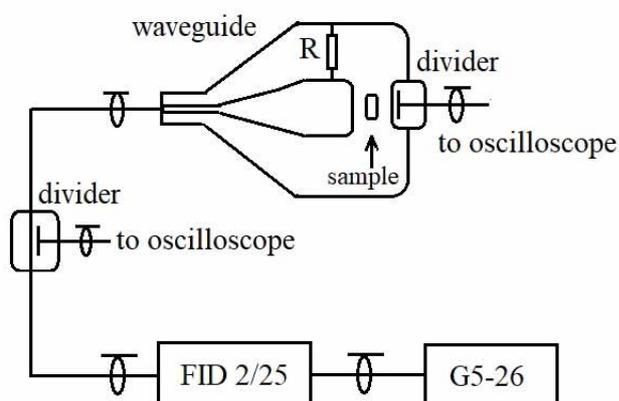


Fig. 1. Experimental setup

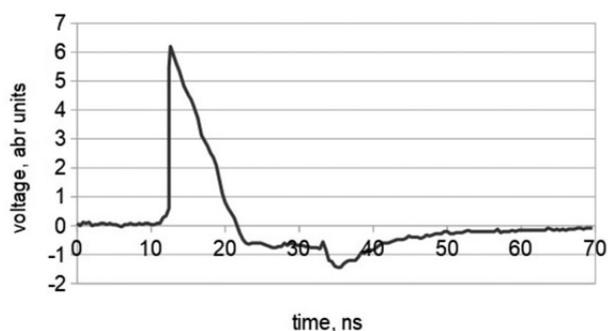


Fig. 2. Pulse shape in sample volume. Amplitude 25 kV/cm

In preliminary experiments, Jurkat and Scov-3 cells were processed at a pulse frequency of 100 Hz. The amplitude value of the electric field strength in the cuvette was 25 kV/cm. The total number of pulses during the processing of each sample was 30000. After the treatment, the stability of the cells was studied using trypan blue staining. The proportion of dying cells was measured after 4 hours, 24 hours and 4 days after treatment.

In experiments on Scov-3 cultures, it was shown that the following day, no more than 50% of living adherent cells remain. In experiments on Jurkat after a day, the concentration of living cells is 90% of the control sample. Authors would like to thank FID Technology for technical help.

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INFLUENCE OF HYDROGEN PLASMA ON THE SURFACE OF A BISMUTH SINGLE CRYSTAL

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Currently, there is an intensified search for new technologies and methodologies that allow creating three-dimensional ordered structures from nanoobjects. Two main parameters are distinguished for create a relief on the surface: injury in plasma and liquid chemical etching. Experience shows that the plasma-chemical effect is always accompanied by the formation of some morphology on an atomically clean surface. In this paper, an attempt was made to plasma processing in chemically active gaseous media in order to modify the nanomorphological surface of bismuth crystals. A bismuth single crystal was a stream of atomic hydrogen (AH). In the experiments was used hydrogen of 99.995% purity. Hydrogen dissociated on the radical with a concentration of active particles is the result of a high-frequency electric discharge. A sample of a bismuth single crystal was treated with atomic hydrogen for an hour.

The crystals surface where smooth and uniform before the visible. As a result of exposure by AH to the surface of bismuth single crystals, formations are formed in the form of protruding submicron and nanoscale particles in the form of single and accumulated triangular pyramids. The angles between the base areas of the pyramidal structure are $\sim 60^\circ$, i.e. they go along the binary axes of the single crystal. The angle of inclination of the single crystal is $\sim 54^\circ - 56^\circ$, which corresponds to the inclination of granite to a less perfect cleavage of the bismuth single crystal.

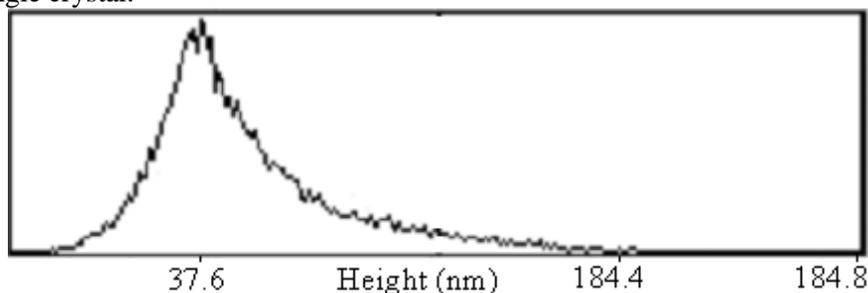


Fig.1. Distribution of formations by height

The distribution of formations by height has the form of a curve with a sharp maximum (Fig. 1) at 37.6 nm. The proportion of formations with sizes less than the most probable is 37%. It is assumed that the modification of nanomorphology is carried out due to self-organization [1, 2]. Process control can be achieved using modern methods of plasma chemical micromachining, which allow modifying the nanomorphology of the surface of crystals, initiating the nucleation of formations of the required size and surface density. Such an approach opens up possibilities for creating quantum-size systems in two- and three-dimensional structures.

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EFFECT OF LOW-TEMPERATURE ION-NITRIDING OF TITANIUM ALLOY (Ti-6Al-4V) ON PARAMETERS OF SURFACE LAYER

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Currently, one of the most effective ways to modify the surface of titanium alloys is ion nitriding. However, it is known that ion nitriding is carried out at high temperatures, which in turn leads to an increase in the structural parameters of the material and the deterioration of its mechanical properties [1-3].

Currently, work is underway to study the low-temperature ion nitriding of titanium alloys, which allows us to maintain a high surface finish and eliminate the deterioration of the mechanical properties of the material [3].

This work is devoted to the comparison of two methods for modifying the surface of a titanium alloy Ti-6Al-4V at low temperatures: nitriding in a glow discharge and nitriding in a non-self-sustained high-current arc discharge. The effect of low-temperature ion nitriding of titanium alloy Ti-6Al-4V on the surface microhardness, the depth of the nitrated layer, roughness and surface residual stresses is considered.

As a result of the study of the effect of nitriding temperature, it was found that treatment, both in a non-independent high-current discharge and a glow discharge at a temperature of 450°C, increases the surface microhardness of samples from titanium alloy Ti-6Al-4V by 1.2 times. As the nitriding temperature rises to 600°C, the surface microhardness of samples processed in a glow discharge increases by 1.3 times. Processing in a non-self-directed high-current arc discharge increases the surface microhardness by 1.8 times.

It has been established that the temperature of ion nitriding affects the surface roughness of the samples. So with an increase in temperature from 450 to 600°C, the roughness value of the surface being treated increases by 2 ... 2.5 times.

It is established that the temperature of nitriding affects the sign and the value of residual stresses. So, with ion nitriding in a non-self-sustained high-current arc discharge and in a glow discharge at a temperature of 600°C, the residual stresses were +12.4 and – 8.4 kgf / mm², respectively. When nitriding in a non-self-acting high-current arc discharge at a temperature of 450°C, the residual stresses amounted to +0.5 kgf / mm², after treatment in a glow discharge - 23.7 kgf / mm².

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INFLUENCE OF HYDROGEN CONTENT ON THE PROCESS OF LOW-TEMPERATURE ION NITRIDING TITANIUM ALLOY Ti-6AL-4V IN CG AND UFG STATES*

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Currently, one of the most effective methods for hardening the surface of parts made of titanium alloys is ion nitriding [1-3]. It is known that the composition of the gas mixture during ion nitriding significantly impact on the structure, properties, phase composition and growth kinetics of the modified layer. The presence of hydrogen in the nitriding mixture increases the diffusion of nitrogen by removing the nanocrystalline layer on the surface of the titanium alloy, which hinders the process of nitriding [4, 5]. However, there are no data on the effect of the hydrogen content in a three-component gas mixture (nitrogen-argon-hydrogen) with low-temperature ion nitriding of titanium alloys ($T \leq 600^\circ \text{C}$) on the technological parameters and properties of the modified layer.

In this work, low-temperature ion nitriding of titanium alloy Ti-6AL-4V in a coarse-grained state (CG) was carried out at a temperature of 600° with different hydrogen contents (0-30% H_2). Based on the results obtained on CG samples, nitriding of titanium alloy Ti-6AL-4V in the ultrafine-grained state (UFG) was carried out at a temperature of 550° with a hydrogen content of 0 and 10%. Microhardness measurements were carried out over the depth of the hardened layer, optical and SEM images of the microstructure were obtained after nitriding. Studies of the current-voltage characteristics of the discharge with different hydrogen contents and gas mixture pressure were carried out.

To assess the effect of hydrogen content on the depth of the hardened layer, and as a consequence on the kinetics of its growth, the curves of the distribution of hardness over the depth of the modified layer were obtained (Fig. 1). Analysis of the plot showed that the addition of hydrogen to the gas mixture leads to an increase in the surface microhardness from 460 $\text{HV}_{0.05}$ with nitriding without hydrogen, to 530 $\text{HV}_{0.05}$ with the addition of 20% hydrogen. A further increase in the hydrogen content to 30% leads to a decrease in the surface microhardness value to values of 460 $\text{HV}_{0.05}$.

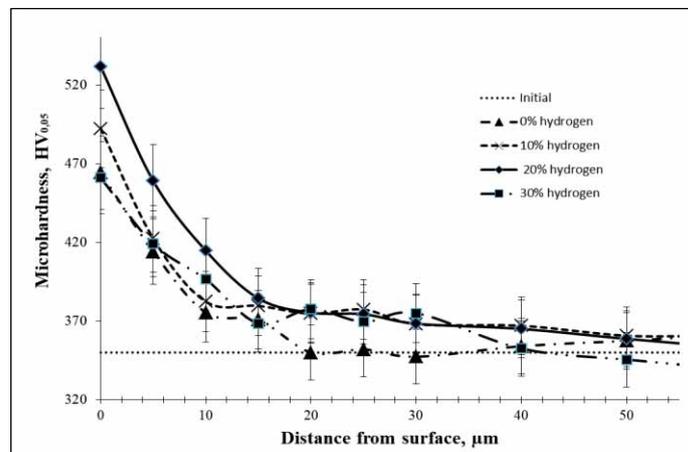


Fig. 1. Plot of microhardness distribution over the depth of the hardened layer

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MODEL CALCULATION OF THE STOICHIOMETRIC COMPOSITION OF THREE-COMPONENT VACUUM ION PLASMA COATINGS*

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A mathematical model of the process of vacuum-arc deposition of coatings of the Ti-Al system in the environment of various gases (nitrogen, oxygen, carbon) has been developed, which allows one to calculate the stoichiometric composition of 3-component coatings. On the basis of the model, a program was created to determine the stoichiometric composition of the coatings depending on the technological regimes (Fig. 1). The samples were deposited with 3 component coatings in the environment of various gases (nitrogen, oxygen, carbon) by calculated regimes (Fig. 2). To determine the adequacy of the mathematical model, the chemical composition of the coatings was determined using energy dispersive analysis. The results of comparing the calculated with the experimental data obtained, it was found that the error of the model is within 10%. The use of the developed model and computer program will allow at the stage of development of the technological process to choose the modes of applying 3 component coatings.

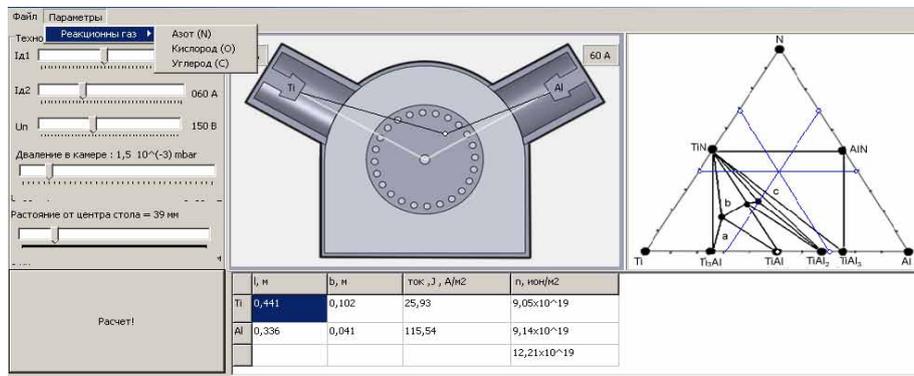


Fig. 1. Interface of the program "Determination of the stoichiometric composition of coatings based on an intermetallic compound Ti-Al system deposited from a vacuum-arc discharge plasma in a medium of various gases"

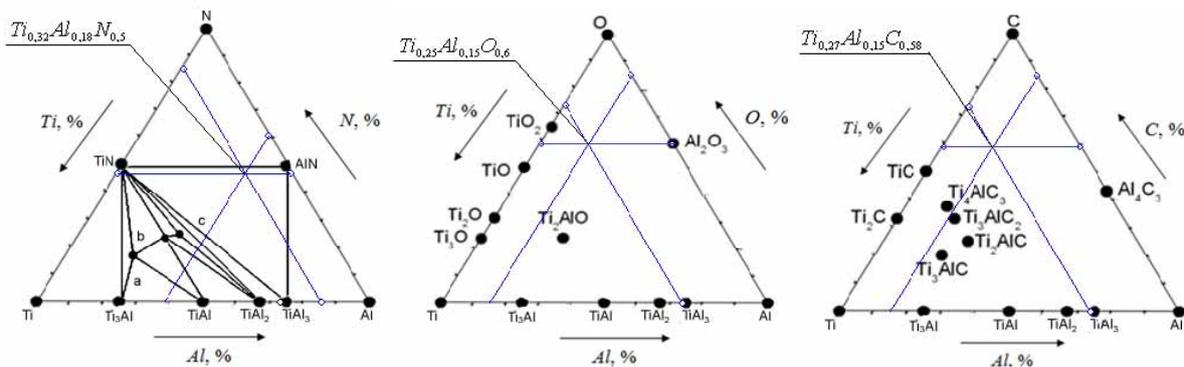


Fig. 2. Interface of the program "Determination of the stoichiometric composition of coatings based on an intermetallic compound Ti-Al system deposited from a vacuum-arc discharge plasma in a medium of various gas"

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EFFECT OF VACUUM ARC PLASMA COATINGS DEPOSITION CONDITIONS ON PARTS QUALITY PARAMETERS

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Hard coatings, synthesized by physical vapor deposition (PVD) are widely used as an inexpensive way to increase the service properties due to their excellent properties. It is highly desirable to deposit a thin coating and increase significantly the service properties. The achievable service properties depend not only from deposited material but from coating structure and surface layer quality. Service properties differ in required values of surface quality parameters for different application. Thus, ensuring the required service properties by controlling the surface quality parameters is highly demand. Therefore, researches aimed at revealing the regularities of surface properties formation under the coating deposition conditions are particularly relevant. Following coating and surface parameters were studied: surface roughness, coating adhesion properties, hardness and wear resistance properties. The following deposition conditions were varied: deposited material, ion source focusing current, substrate negative bias, substrate location and substrate initial condition. Effect of additional ionization sources such as plasma source with filament cathode and plasma source with hollow cathode presence was also studied. The experimental results are highly promising in industrial application through their wide industrial application.

OXIDATIVE PLASMA CHEMICAL TRANSFORMATIONS OF C₃-C₄ ALKANES*

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The results of plasma-chemical transformations of C₃-C₄ alkanes as a liquified petroleum gas (LPG) in a barrier discharge plasma in the presence of octane are presented. The transformations of the components of gas-liquid mixture result in the formation of predominantly hydroxyl and carbonyl compounds with the same number of carbon atoms in a molecule as in the starting compounds. The presence of an octane favours an effective removal of the reaction products from the discharge zone due to dissolution of the compounds formed therein.

The main active particles in a non-thermal plasma of the barrier discharge are alkyl radicals, atomic oxygen, and hydrogen. The formation of both oxygenated compounds and hydrocarbons with isomeric structure occurs as a result of their further chemical transformations. The mechanism of conversion of gaseous hydrocarbons is much like that for the conversion of liquid hydrocarbons in a barrier discharge plasma. They both are carried out under similar conditions.

The changes in the initial concentration of the propane and butane in the initial gas mixture from 10 to 75 % wt result in a decrease in the conversion of gaseous hydrocarbons from 4.1 to 0.9 % wt, while the conversion of octane decreases from 2.4 to 0.3 % wt. in one pass through the reactor. The decrease in hydrocarbon conversion is due to decrease in the rate of formation of atomic oxygen in a discharge gap of the reactor.

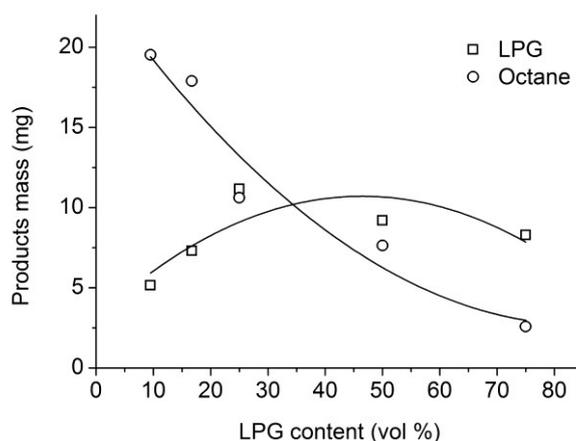


Fig. 1. Mass of the products of the plasma chemical transformation of LPG and octane

Theoretical calculations for the model oxygen-propane mixture have been made using the Bolsig+ software. The results of calculations show that the oxygen dissociation rate constant decreases from $1.87 \cdot 10^{10} \text{ cm}^3/\text{s}$ to $6.71 \cdot 10^9 \text{ cm}^3/\text{s}$ due to a decrease in the mean electron energy from 4.1 to 3.4 eV. It is found out that the mass of products formed in the result of oxidation of gaseous and liquid hydrocarbons depends on the initial concentrations of starting compounds in the vapor-gas mixture. A simple expression is proposed to evaluate the preferential direction of the plasma-chemical reaction depending on the initial concentration of hydrocarbons in a discharge gap of the reactor.

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EFFECT OF ELECTRON-BEAM IRRADIATION ON THE SAFETY AND QUALITY OF HELMINTHOSPORIUM-INFECTED BARLEY

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The fungus *Helminthosporium* (*Helminthosporium sativum* Pam.) is a widely distributed cereal pathogen, but the disease is most harmful in the eastern regions of Russian Federation [1].

In connection with the ecological orientation of agriculture, at present, the efforts of many scientists are aimed at finding new methods of disinfecting seeds without the use of pesticides.

At the end of the last century, it was shown that presowing irradiation of seeds of agricultural crops can be used to increase crop yields [2].

Previously at a low-energy electron accelerator with a plasma cathode and the beam output into the atmosphere it was shown that presowing irradiation of barley seeds of the Vladimir variety reduces of the development of helminthosporiosis in seedlings [3]. Studies on the presowing of seeds at an electron-beam accelerator in an extended dose range from 1 to 8 kGy and a dose rate of 500 Gy per impulse allowed to establish that the progression of the disease depends on both the radiation dose and the post-radiation period.

The most effective was the effect of irradiation during the post-radiation period of 4 days – a decrease in the degree of damage by 33-48 % was observed at doses of 4-8 kGy, and the prevalence of the disease – by 29-46 % (Fig. 1).

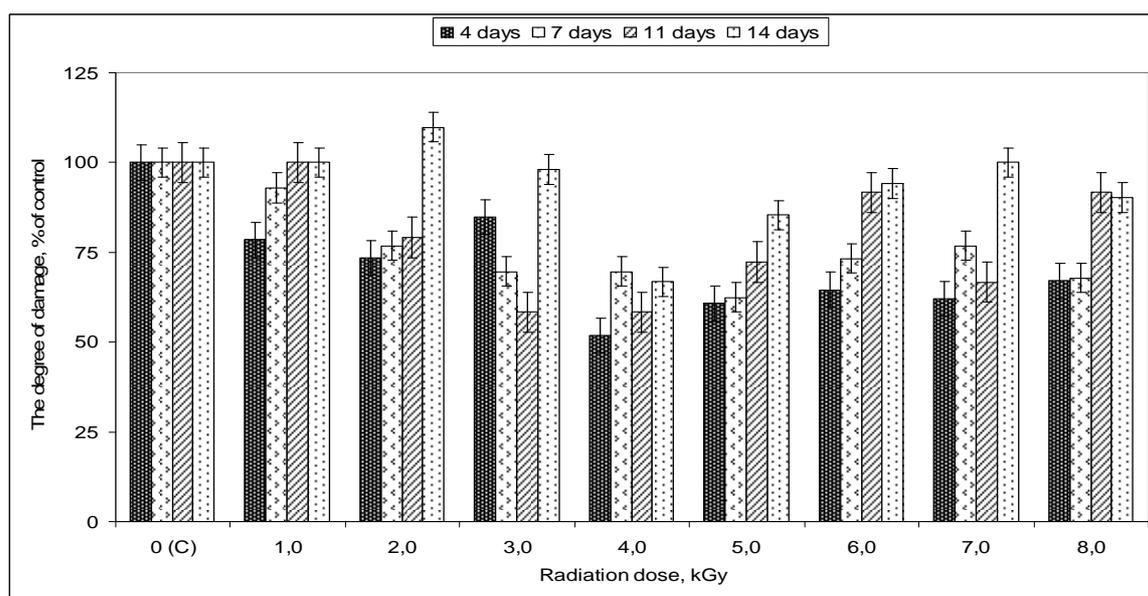


Figure 1. The effect of irradiation on the damage of barley by *Helminthosporium sativum* depending on the post-radiation period

Thus, it was shown that presowing electron-beam irradiation of spring barley of the Vladimir variety reduces the resistance of *Helminthosporium sativum*.

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EFFECT OF TRENCHES ON SHEATH FORMATION NEAR EMISSIVE SURFACE IN LOW PRESSURE PLASMA IN MAGNETIC FIELD*

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The erosion of emissive surfaces during long term operation essentially effects the working characteristics of low temperature plasma devices. The transition in sheath structure near the emissive surface with erosion patterns in low temperature plasma is essentially different for planar and non-planar surfaces [1].

In this work, we study the formation of the plasma sheath near the emissive floating sample with complex topology in magnetic field normal to the sample. From the experimental and calculated data, the spatial potential distribution was derived for a) a sample made from Al₂O₃ with the trenches and b) for a planar control sample. In Fig. 1, the sketch of the experimental setup is shown. Experimental study was carried out in a vacuum chamber filled with Argon with a pressure of 0.05-0.2 mTorr. The cylindrical chamber 1 is made of stainless steel and is closed with the ends of the covers in a flange manner. The diameter of the test volume of 160 mm. The covers have insulated vacuum electrical inputs for supply the thermal emission cathode 5, make potential between the cathode and the chamber, and measuring signals from Langmuir probes 9. The applied voltage ranges from 30 V to 300 V, the external magnetic field is 20 G. The emissive sample with a complex topology is a disk with trenches with depth and width of 5 mm. The simulations of spatial distribution of plasma parameters were performed with Particle in Cell Monte Carlo Collision method for the experimental conditions. Both the experimental and calculation results show considerable difference in the sheath structure transition with increasing the applied voltage for planar and non-planar cases.

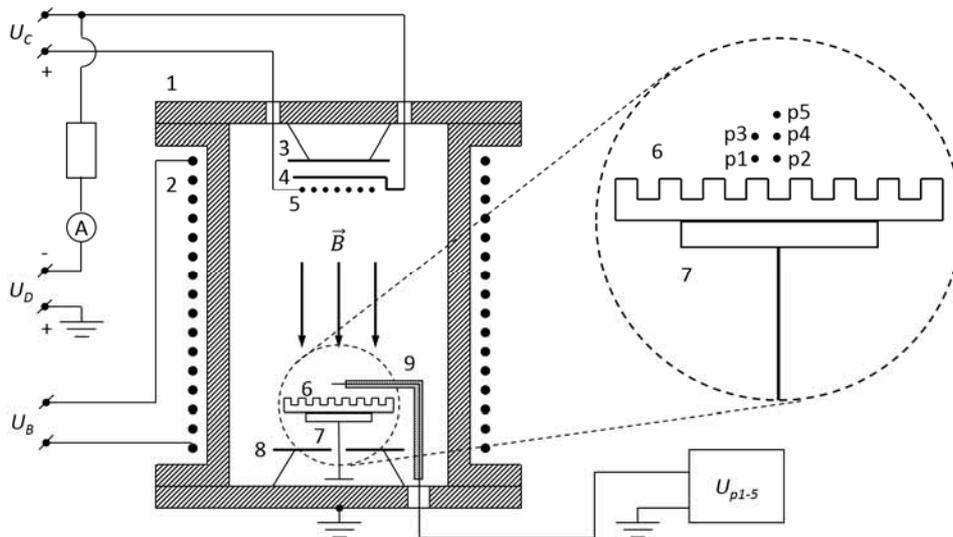


Fig. 1. Work chamber scheme: 1 – vacuum chamber, 2 – magnet system, 3 – screen, 4 – cathode screen, 5 – thermionic cathode, 6 – sample, 7 – foot plate, 8 – screen, 9 – Langmuir probe.

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PARTICLE GENERATION DURING LASER-PLASMA TREATING OF METALS IN EXTERNAL ELECTRIC FIELD

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The radiation of the GOR-100M ruby laser operating in the free oscillation regime (pulse duration ~ 1.2 mc) passed through the focusing system and was directed through the hole in the electrode onto the sample that served as the second electrode and was mounted in air at a pressure of 10^5 Pa. The energy of the laser pulses varied from 5 to 60 J. The voltage was applied to the electrodes from the source, built on the basis of the UN 9/27-13 voltage multiplier of the TVS-110 unit. The source allowed the voltage variation within 25 kV and its stabilization in the course of the experiment. To study the spatial and temporal evolution of the laser plasma torch in the course of laser radiation action on the sample, we used the method of high-speed holographic motion-picture recording. Presence of an external electric field weakly affects the concentration of electrons in the laser plasma plume. When either positive or negative potential is applied to the sample, many small droplets appear on its surface after the laser action. In particular, at the laser pulse energy 20 J, the diameter of the focusing spot 2 mm, and the electric field strength 10^6 V/cm we observed ejection of droplets having the mean characteristic size less than 0.1 mm to the distance up to 2 cm from the crater centrum. The maximal characteristic size of drops was ~ 0.4 mm. In the absence of the external electric field the mean size of the droplets was ~ 0.4 mm. The droplets were seen at the distance up to ~ 1 cm from the crater centrum. The primary plasma formation and the initial stage of the laser plume development, in principle, do not differ from those observed in the absence of the external electric field. The metal is melted and evaporated. As a result of local formation of steam and plasma, the erosion plume begins to form with the fine-dispersed liquid-drop phase. Note, that the bulk evaporation is promoted by the gases, diluted in the metal, and by the spatiotemporal non-uniformity of the laser radiation. At a radiation flux density $10^6 - 10^7$ W/cm² the bulk evaporation is typical of all metals used in the experiments. Obviously, the presence of the external electric field affects (increases or decreases depending on the direction of the field strength vector) the velocity of motion of the plasma front and causes some distortion of the plasma cloud shape. It is essential that the mentioned differences (at the considered parameters of laser radiation) are observed only at the initial stage of the laser plume development, because after the steam-plasma cloud reaches the electrode an electric breakdown (short-circuit) occurs, and the external field in the interelectrode gap disappears. The significant difference in the characteristic size of droplets, observed on the surface of the irradiated sample in the presence of the external electric field (independent of the direction of the field strength vector) and in the absence of the field, is a manifestation of the following mechanism of droplet formation. It is known that at the surface of a liquid (including a liquid metal) the formation of gravity-capillary waves is possible under the action of various perturbations. Undoubtedly, the examples of such perturbations are the spatially non-uniform evaporation of the target material due to non-uniform heating caused by non-uniform energy distribution over the focusing spot, the non-uniform primary plasma formation caused by roughness of the irradiated sample surface, and, in the first place, the slop of the molten metal initiated by each spike of laser radiation, acting on the exposed sample.