STATE SCIENTIFIC INSTITUTION
"B. I. STEPANOV INSTITUTE OF PHYSICS,
NATIONAL ACADEMY OF SCIENCES OF BELARUS"

UDK 533.924; 533.9.08

SARI Amir Hossein Mohammad Ali

MODIFICATION AND ALLOYING OF SILICON AND STEEL SURFACES BY COMPRESSION PLASMA FLOWS AND ION BEAMS

ABSTRACT
of dissertation for a degree of
doctor of philosophy in physics
in specialty 01.04.08 - plasma physics

Minsk, 2014

The work was performed at the State scientific institution “B.I. Stepanov Institute of Physics of the National Academy of Sciences of Belarus”

Supervisor:	**Astashinsky Valentin Mironovich** Doctor of Physical and Mathematical Sciences, Deputy Director for Science, A.V. Lykov Institute of Heat and Mass Transfer, National Academy of Sciences of Belarus
Official opponents:	**Tarasenko Nikolay Vladimirovich** Doctor of Physical and Mathematical Sciences, Head of the Laboratory of laser plasma diagnostics, B.I. Stepanov Institute of Physics, National Academy of Sciences of Belarus
	Kuleshov Andrew Konstantinovich Candidate of Physical and Mathematical Sciences, Head of the Laboratory of Physics of ion-plasma modification of solids, Belarusian State University
The opposing organization:	Physico-Technical Institute of the National Academy of Sciences of Belarus

The thesis defense will be held on 23 December, 2014 at 14^{30} on the meeting of the Board on thesis defenses D 01.05.01 at the Institute of Physics, NAS of Belarus at address: 220072, Republic of Belarus, Minsk, 68 Nezavisimosti Ave., office 218. e-mail: ponyavina@imaph.bas-net.by, scientific secretary phone: 284-04-50.

The thesis is available in the library of the Institute of Physics of the National Academy of Sciences of Belarus.

Author's abstract is distributed on 21 November 2014

Scientific secretary
of the Council on the thesis defending
Doctor of Physical and Mathematical Sciences A.N. Ponyavina

INTRODUCTION

One of the promising areas to use ion beam sources and plasma flows is the processing of materials for various industrial sectors, such as microelectronics, aerospace equipment, nuclear facilities, in the manufacture of medical devices and implants, etc. In conventional ion implantation, a continuous ion beam is produced by a plasma source and accelerated to a preset energy. In this technique, the irradiation time and the ion current density and energy can be precisely controlled, which makes it possible to accurately manage the concentration and penetration depth of the ions into material. The interaction of ions with the material surface causes microstructural and phase changes and results in mechanical and electrochemical modification of the surface layers. New possibilities for modifying the surface properties of materials are opened by the application of high-energy plasma flows that provide a significant improvement in the performance properties of the surface, which cannot be obtained by conventional thermochemical methods.

The thesis presents the results of experimental and theoretical studies into effects of ion beams and compression plasma flows (CPF) on the widely used materials: silicon wafer and steel plate substrates and the coating-substrate systems (coatings: Cr, Ta, Zr, Ta-Ti; substrates: Si, carbon steel, austenitic stainless steel). Particular attention is given to establishing the dependences of structural and phase changes in the modified layer on the parameters of the ion-plasma exposure for development of a scientific basis for new plasma techniques capable of significantly improving operational properties of materials.

GENERAL DESCRIPTION OF THE WORK

Relation with research programs (projects) and themes

The thesis was carried out in accordance with the tasks comprising the following programs and projects:

1. Sub-program "Fundamental physical interactions and their manifestations in the structure of matter at sub-nuclear and macroscopic levels: fields, particles, nuclei i.e. topologically non-trivial objects, condensed matter, and plasma", the State research program "Interdisciplinary researches and new emerging technologies as a basis for sustainable innovative development (Convergence), "Convergence 2.4.01" task “Plasmadynamic systems of a new generation and physical processes of high-energy heterogeneous plasma flow action on materials, structural / phase evolution and radiation-plasma activation of modified objects, synthesis of low-dimensional structures and coatings "(2011 - 2013, State registration № 20115497).

2. Project BRFFR number F14R-172 "Physical processes of formation of nanostructured coatings of a preset composition for various materials exposed to gas discharges and compression flows in dusty plasma" (2014–2016).

The purpose and objectives of the study

The aim of the thesis is to establish physical regularities in diversity of effects of compression plasma flows and ion beams, generated respectively by gas-discharge magneto-plasma compressor and Kaufman plasma source, on silicon wafers and carbon steel samples, which cause significant changes in their surface properties, including synthesis of nanostructured formations and coatings, and to find dependencies of structural and phase changes in the modified layer on the parameters of the ion-plasma exposure to provide a scientific basis for new plasma techniques intended to significantly improve the performance of materials. To achieve this goal, the following main tasks should be solved:

- Determination of relationships between parameters of the ion-plasma exposure and surface properties (e.g., optical, electrical, mechanical, and electrochemical);
- Investigation of the influence of the ion-plasma impact on the microstructure and phase composition of different systems (steel, Si, Cr/Si, Cr/steel, Ta/Si, Ta-Ti/Si, Ta/carbon steel, Zr/austenitic steel) and analysis of surface properties both on profiles of the element content through the depth and on the microstructural images;
- Experimental measurement of the plasma pressure on the treated sample surfaces using an optical sensor and computation of the plasma pressure by numerical simulation;

Scientific novelty of the work is in establishing new regularities in formation of a modified layer on surfaces of carbon steels and silicon wafers and in management of its structure and properties. It is shown that the action of a single pulse of a compression flow on silicon wafers coated with a thin tantalum layer results in melting of a silicon substrate surface layer and partial melting of tantalum coating, which ensures the formation of a nanostructured layer enriched in tantalum silicides and nitrides and formed by particles 15–75 nm in diameter. The exposure of X18H10T austenitic steel samples with a thin zirconium coating to a series of 5 plasma pulses causes almost a two-fold increase in the modified surface hardness and lowers its friction coefficient by a factor of ~ 5 due to formation of nitrides and intermetallics.

Provisions for the defense

1. Treatment of silicon samples and a Cr/Si system by an ion beam, generated by stationary Kaufman plasma source (working gas is nitrogen), with the energy of ~ 30 keV and a dose in the range of 10^{16}–10^{18} ions/cm^2, leads to implantation of a thin surface layer (~ 0.1 μm) with nitrogen ions that form nitride phases, due to which the silicon surface resistivity of the layer increases to ~ 180 Ohm/square (versus ~ 140 Ohm/square for the unimplanted sample) and microhardness of the Cr/Si-system enhances to ~ 1100 kg/mm^2 (as compared to 800 kg/mm^2 prior to implantation).

2. The exposure of silicon wafers (10×10×0.28 mm^3) coated with a thin (~ 1 μm) layer of tantalum to a single pulse of the compression plasma flow at the density of absorbed energy of 9 J/cm^2 leads both to melting of the surface layer of the silicon substrate and to partial melting of the tantalum coating, which ensures the formation of a nanostructured layer enriched by tantalum silicides and nitrides and comprised of particles with diameters from 15 to 75 nm; with the increase in the number of pulses of the plasma impact to 3 (with other conditions being intact), these nanoparticles additionally merge into submicron clusters ~ 200–500 nm in diameter.

3. The action of the compression plasma flow at the absorbed energy density of 3 J/cm^2 on silicon wafer with a thin composite Ta/Ti coating (thickness of tantalum and titanium layers is about 1 μm each) results in a modified layer in which a solid tantalum-titanium solution and tantalum nitride phases (TaN and Ta_2N) are formed; with the increase in the absorbed energy density to 9 J/cm^2, nitride and oxide phases disappear, and only titanium silicide (Ti_5Si_3) is formed.

4. The action by a series of 5 compression plasma flow pulses at the absorbed energy density of 9 J/cm^2 per pulse on the samples of industrially used X18H10T austenitic steel with a thin (2.5 μm) Zr coating leads both to an increase in the microhardness of the modified surface by ~ 2 times and to a decrease in the friction coefficient by a factor of ~ 5 due to the formation of hardening nitrides (ZrN and Cr_2N) and intermetallic compounds ($Fe_{23}Zr_6$); increasing the number of plasma pulses from 1 to 5 causes an increase in the depth of the modified layer from 8 to 12 μm and yields a more uniform distribution of alloying elements (as compared with that after a single pulse exposure).

Personal contribution of the applicant

The thesis presents the results of experimental and theoretical studies carried out by the applicant personally as well as the data obtained with the direct participation of the applicant in experiments conducted in collaboration with the laboratory staff and with persons from other organizations. During studies performed in collaboration, the applicant was directly involved both in the formulation of research problems and in carrying out of specific experiments, in analysis and discussion of results. The thesis supervisor, D.Sc. V.M. Astashynski formulated and defined the subject and areas of research, participated in considerations and a choice of experimental methods, in analysis and interpretation of the results. Professor V.V. Uglov and D.Sc. N.N. Cherenda provided methodological and consultative assistance in conducting the experiments and discussing the results. Numerical simulation of dynamics of compression plasma flows, as well as analysis and discussion of the results of experiments to measure the pressure of the plasma were carried out jointly with Ph.D. S.I. Ananin. Ph.D. E.A. Kostyukevich and P.N. Shoronov provided methodological and advisory assistance in conducting experiments to measure the plasma pressure. Ph.D. A.M. Kuzmitsky participated the preparation of the magneto plasma compressor to operation and in sample processing by compression plasma flows. Data obtained by other co-authors are not included in this thesis.

Approbation of the dissertation results

The research results were presented and discussed at the following conferences: VII International Conference "Plasma Physics and Plasma Technology" (Minsk, Belarus, September 17 – 21, 2012); International Conference “Nanomeeting-2013 Physics, chemistry and application of nanostructures" (Minsk, Belarus, 28 – 31 May 2013); International Conference on Advanced applications in nanotechnology – “IBCN12” (27 – 29 June, 2012, Minsk, Belarus); IX Symposium "Physics and diagnostics of laboratory and astrophysical plasma" (Minsk, Belarus, September 16–21, 2012); 10th International Conference on Interaction of radiation with solids (Minsk, 2013); 27th Symposium on the physics and technology of plasma (Prague, Czech Republic, 16–19 June 2014). X Symposium "Physics and diagnostics of laboratory and astrophysical plasma" (Belgrade, 25–29 August 2014); Energy Efficiency - 2014 (Minsk, Belarus, 28 – 31 October 2014).

Publication of the thesis results

On the topics of the dissertation, 18 scientific papers are published including 9 articles in peer-reviewed journals [1–9] (totaling 3.8 copyright sheets), 7 articles in proceedings and materials of scientific conferences [10–16], and 2 abstracts at scientific conferences [17, 18].

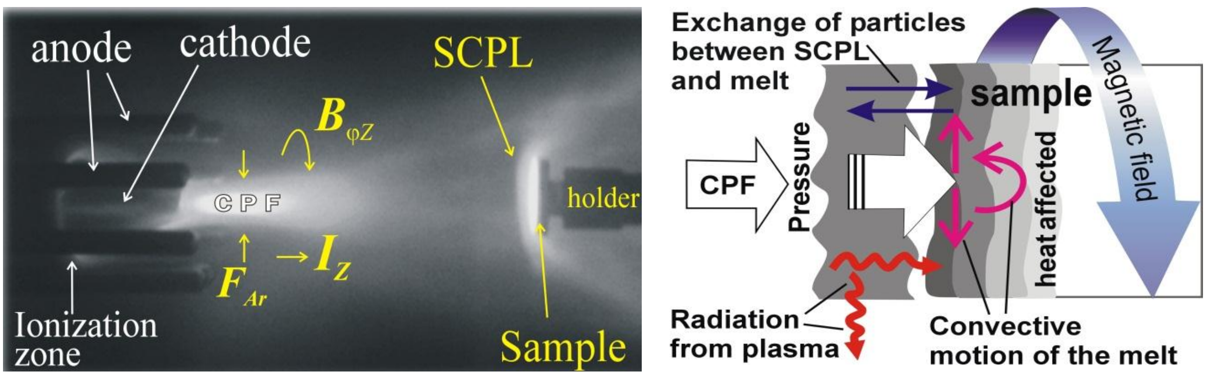
Structure and scope of the thesis

The thesis includes an introduction, general characteristics of the work, four chapters, conclusion, bibliography, and recommendations for the practical use of the results. The total volume of the thesis is 119 pages, the work contains 63 drawings at 55 pages and 13 tables at 13 pages. The References at 16 pages include 212 items.

THE MAIN CONTENTS OF THE WORK

The first chapter provides an analysis of scientific literature on surface modification of materials subjected to compression plasma flows and ion/electron beams. Based on the analysis, the purpose and main objectives of the thesis are formulated.

The second chapter is devoted to the description of experimental setups and diagnostic methods. In the thesis, the methods for surface modification by means of a stationary ion beam and a compression plasma flow were used. The ion beams were produced by Kaufman plasma source. Compression plasma flows were generated by gas-discharge magnetoplasma compressor, figure 1.



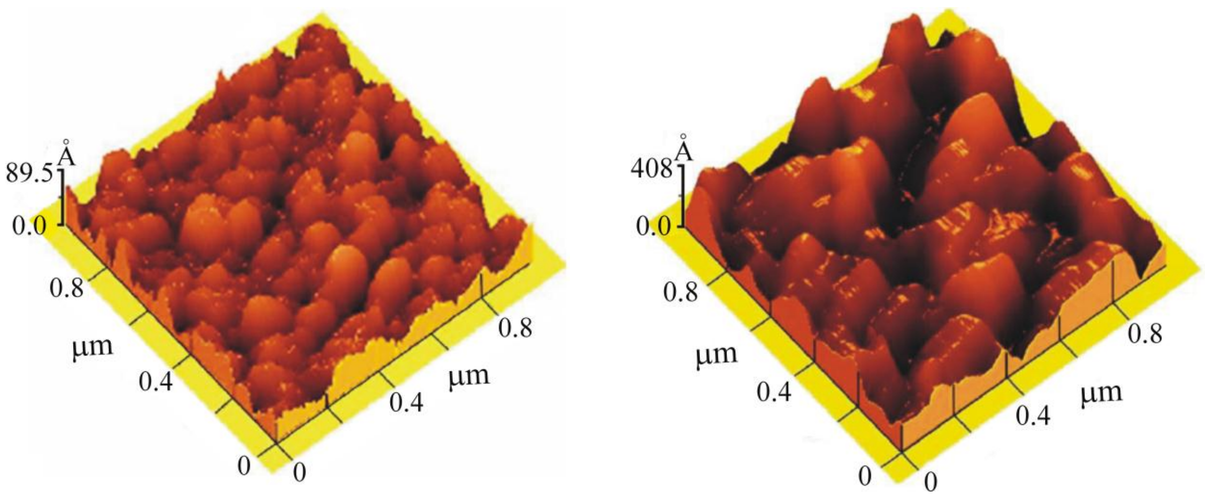
a *b*

a **–photo, *b* – schematic interaction between CPF and a target**

Figure 1. – Action of CPF on a target

The third chapter presents results of studies on irradiation of materials by an ion beam generated by Kaufman stationary plasma source with nitrogen and carbon dioxide as working substances, which allows both to simultaneously accelerate different types of ions, such as $N^+$and N_2^+ in nitrogen or C^+, CO^+, CO_2^+, O_2^+, O^+, etc. in CO_2. It was shown that irradiation of the silicon, chromium film-on silicon, chromium film-on steel, and steel samples by continuous nitrogen ion beam at energy of ~ 30 keV with doses between 10^{16} and 10^{18} ions/cm^2 results both in implantation of a thin surface layer with formation of nitride phases and in the increased surface roughness. The conducted experiments revealed that electrical properties of the exposed silicon samples depend on the irradiation dose: at a nitrogen dose of 10^{18} ions/cm^2, sheet resistivity increases to ~ 180 ohms/square (against ~ 140 ohms/square for unimplanted sample).

Experiments on implantation of nitrogen ions were conducted by using substrates from austenitic steel coated with thin chromium films (600 nm and 1 μm thick) as targets. Implantation of nitrogen ions into the film results in the formation of chromium nitride phases (CrN and Cr_2N). Figure 2 shows the surface of the untreated sample and that of the implanted surface with the enhanced roughness.



***a* – unimplanted sample** ***b* – ion implanted at 6×10^{17} ions/cm^2**

Figure 2. – Three-dimensional atomic force microscopy images of Cr/Si samples

It is shown that the action of N^+ and N_2^+ ions on the Cr/austenitic steel system causes the increase in the hardness and corrosion resistance of the samples depending on the radiation dose. It is found that the maximum value of corrosion resistance for this system is reached at a dose of 2×10^{18} ions/cm^2.

Using secondary ion mass spectrometry (SIMS) method, distributions of nitrogen and chromium nitride were measured into the depth of the samples. These profiles are shown in Figure 3. The figure shows that the maximum penetration of the nitrogen atoms is lower than that of chromium nitride.

By using potentiodynamic polarization measurements, electrochemical properties were defined for the irradiated steel samples and the Cr/steel system (a steel substrate was coated with a 1 μm chromium film). It has been found that the corrosion resistance can be substantially increased depending on the dose of nitrogen ions. Electrochemical tests showed that ion implantation causes the formation of a stable passive layer due to the presence of CrN and γ_N phases. For the N implanted

Cr/steel system, a maximum enhancement (~ 2 times) in the corrosion resistance was reached at a dose of 2×10^{18} ions/cm^2.

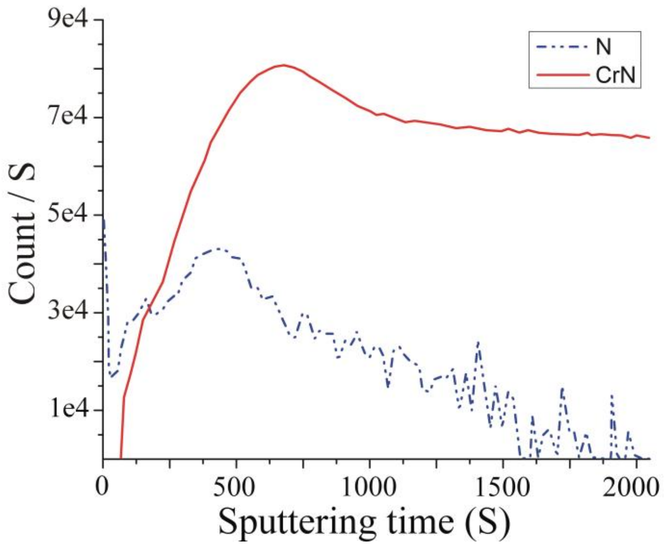


Figure 3. – SIMS depth profile of nitrogen and CrN corresponding to the sample implanted at the dose of 8×10^{17} ion/cm^2

The fourth chapter of the thesis presents the data on surface modification of silicon wafers and carbon steel samples by compression plasma flows depending on exposure parameters. The pressure of compression plasma flows on the target surface was measured using an interferometric optical sensor, which is schematically shown in Figure 4. The sensor includes an acoustic element made of a copper rod, a He-Ne laser, and a photomultiplier. The polished back surface of the rod reflects the laser light beam back into the laser cavity.

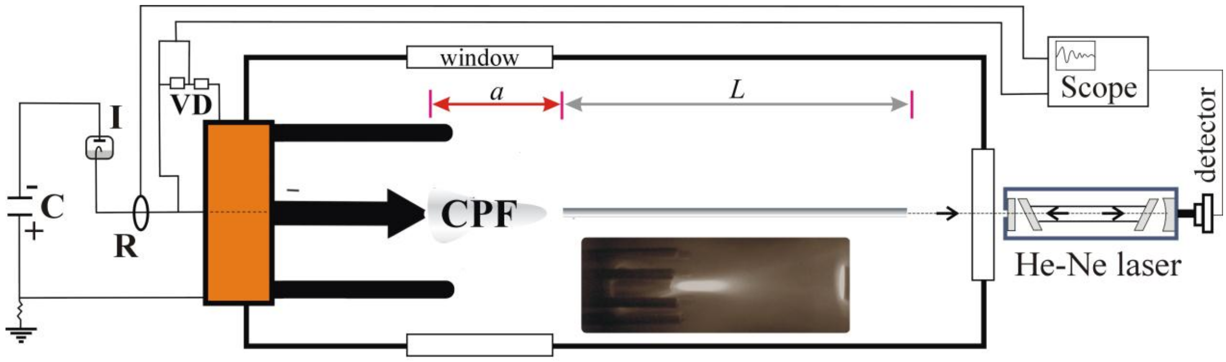


Figure 4. – Experimental setup: capacitor (C), ignitron (I), Rogowski coil (R), voltage divider (VD)

The compression plasma flow impinging upon the front surface of the rod, excites an acoustic wave that propagates along the rod at the speed of sound in copper. The reflection of this wave from the polished surface causes it to shift from its initial position. As a result, the relation between the phases of the beam returned to the cavity and emission in the laser tube begins to vary, and the intensity of the beam incident on the photomultiplier will be modulated with a frequency proportional to the displacement velocity of the rear surface from the initial position.

The plot reflecting changes both in the pressure of the plasma flow and in the discharge current versus time is shown in Figure 5*a* for U_0 = 4.5 kV and a = 12 cm (distance between the target and the cathode tip). As can be seen, the maximum value of the plasma flow pressure is about 16 atm.The results of pressure measurements at the same distance for U_0 = 3.0 kV and 4.0 kV are shown in Figure 5b.

The experiments showed that the maximum pressure of the plasma stream at a = 8 cm and U_0 = 4.5 kV reaches about 73 atm.

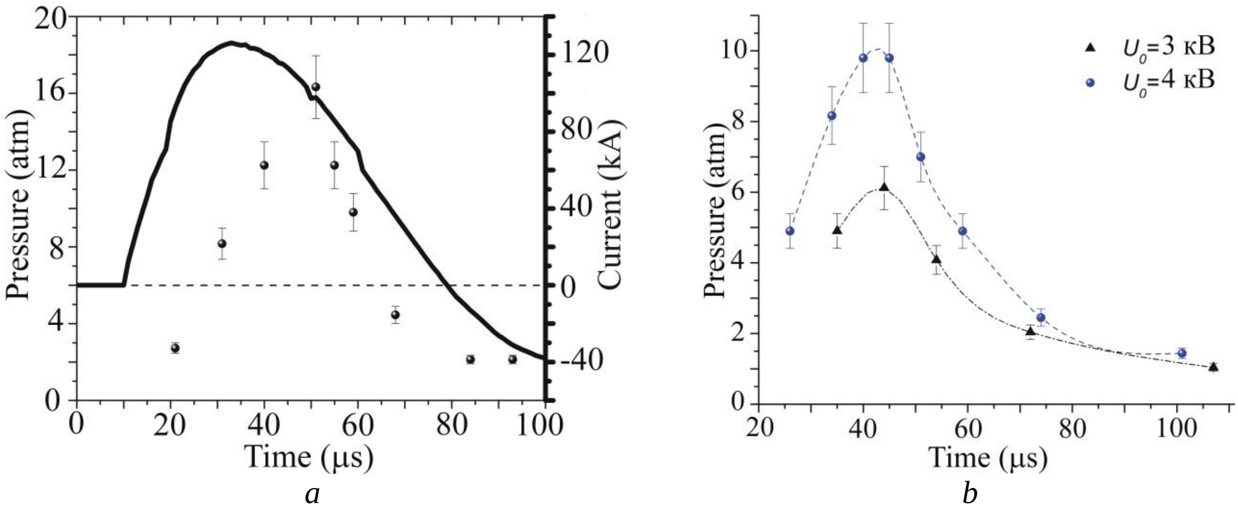
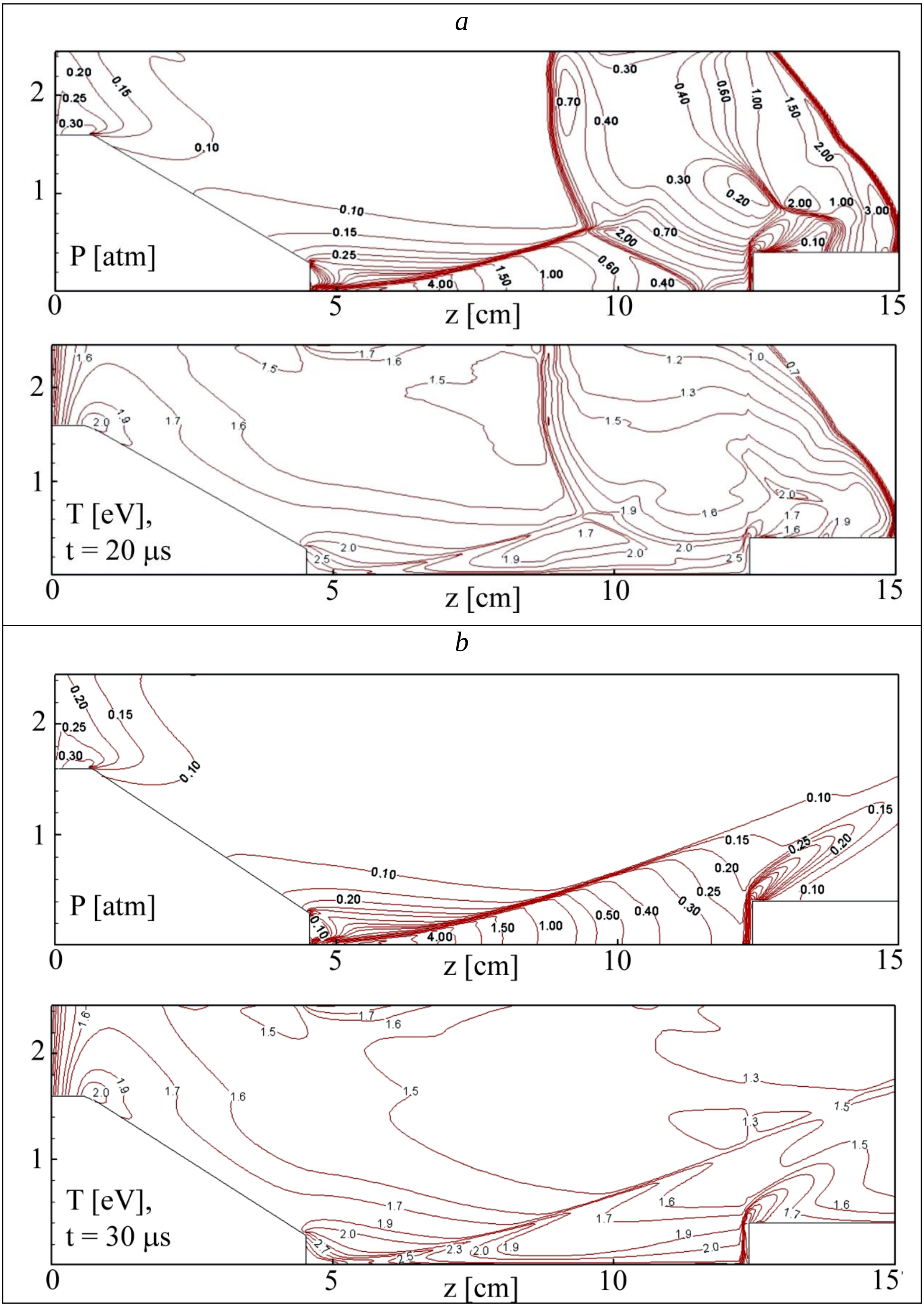


Figure 5. –Plasma pressure and discharge current (solid line) versus time for U_0 = 4.5 kV (*a*), and plasma pressure on target forU_0 = 3.0 and 4.0 kV (*b*)

To validate experimental data on the pressure measurements, numerical simulation of the compression plasma flow parameters was performed using a large-particle method with introduction of the magnetic field and the transfer of energy by radiation. A system of nonstationary equations of radiative magnetogasdynamics was used. Boundary conditions were set in accordance with those in the experiment. The results of calculations are presented in Figure 6.

Figure 6*a* shows distributions of pressure and temperature in plasma at t = 20 μs. At this point, the maximum values of pressure on the target surface, P_{max}, temperature, T_{max}, and electron density, $n_{e\,max}$, reach ~ 80 atm, ~ 5 eV, and ~ 2×10^{18} cm^{-3} respectively. By the time t = 30 μs, P_{max} ~ 16 atm, T_{max} ~ 5 eV, and $n_{e\,max} = 2\times10^{18}$ cm^{-3}.

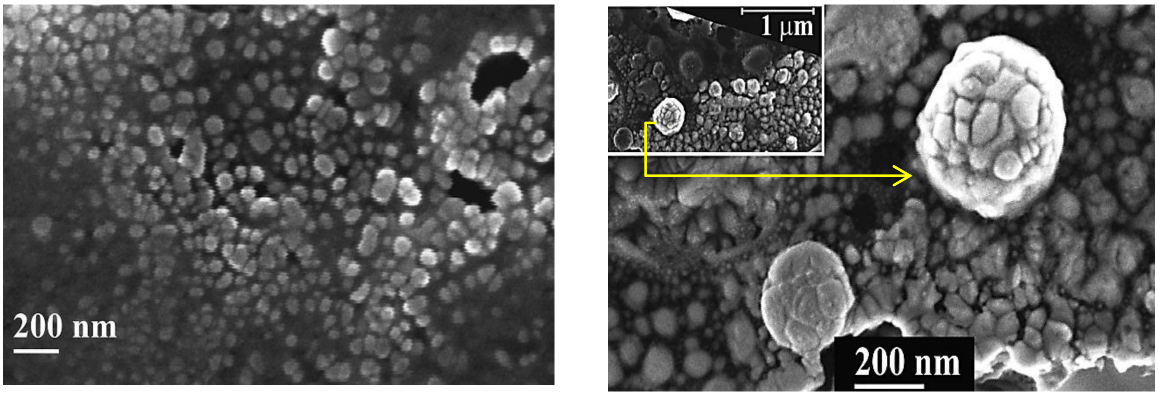


a **– t = 20 μs,** ***b*** **– t = 30 μs**

Figure 6. – Distribution of plasma pressure and temperature

Thus, the calculations showed that for the discharge currents in the range of 100 – 150 kA, the plasma temperature at the target surface is 2 – 5 eV and the electron density is in the range from 3×10^{17} – 5×10^{18} cm^{-3}. The maximum plasma pressure on the target reaches ~ 80 atm at a distance of 8 cm from the target to the cathode tip, which is in a good agreement with experimental data. Such plasma parameters are high enough to melt even very hard materials.

The studies on modification of silicon wafers with thin (~ 2 μm) coatings of tantalum or Ta/Ti compositions were conducted by subjecting them to compression plasma flow (plasma-forming gas – nitrogen). It was shown that the action of the compression flow under 400 Pa initial nitrogen pressure at the 100 μs pulse duration and the 9 J/cm^2 absorbed energy density on the Ta/Si system (tantalum layer is ~ 1 μm thick) results in the formation of a surface layer enriched in tantalum silicides and nitrides. It is stated that increasing the number of plasma flow pulses causes a change in the configuration of nanoscale structures: a single pulse yields single-level nanostructures 15–75 nm in size, whereas at three pulses of the plasma flow, these particles additionally merge into spherical clusters ~ 200–500 nm in diameter, Figure 7. Tantalum nitride is formed due to the interaction between tantalum and plasma-forming gas (nitrogen).



a *b*

a **– single pulse,** ***b*** **– three pulses**

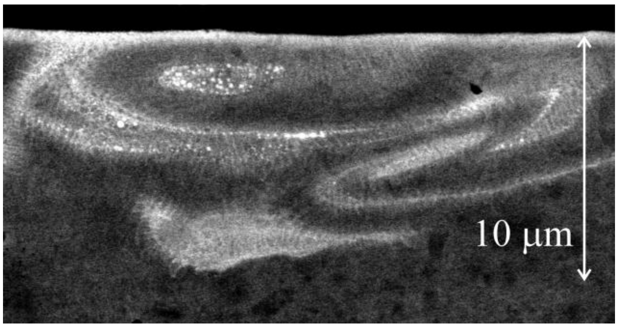
Figure 7. – SEM images of Ta/Si(100) samples after CPF treatment

Studies into modification of silicon with a composition Ta-Ti coating are of interest in context of the possibility to form two types of silicides. It is found that under the action of the compression plasma flow (CPF) on the Ta/Ti/Si system (thickness of tantalum and titanium coatings is ~ 1 μm), a surface layer is formed whose composition is determined by the energy density (w) of the impacting flow: at w = 3 J/cm^2, a tantalum-titanium solid solution and tantalum nitride phases (TaN and Ta_2N) are formed, but tantalum silicide is not detected; at w = 9 J/cm^2, titanium silicide (Ti_5Si_3) is formed, whereas nitride and oxide phases disappear.

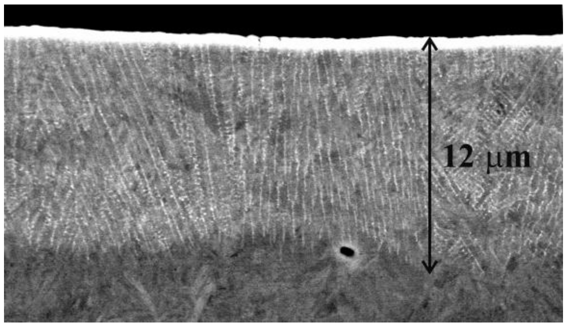
Numerical simulations show that the temperature in the surface layer of the Ta/Ti/Si system exposed to the compression plasma flow exceeds the melting point of silicon, but does not reach that of tantalum, due to which tantalum silicide is not formed.

Experimental studies on modification of steel surfaces by compression plasma flows were conducted by using two types of samples, namely U8A carbon steel pre-coated with a tantalum film (thickness ~ 700 nm) and X18H10T austenitic steel pre-coated with a thin zirconium film (~ 2.5 μm).

The studies made it possible to determine regularities of substantial improvement in surface properties of U8A carbon steel exposed to the compression plasma flow. It is shown that the action of compression plasma flows with a pulse duration of 100 μs and absorbed energy density of 9 – 20 J/cm^2 on the Ta/U8A system results in the formation of the surface layer doped by tantalum atoms. It is stated that the microhardness of the surface can be increased either by enhancing the energy density of the exposure or by increasing the number of impacting pulses (Figure 8).



a



b

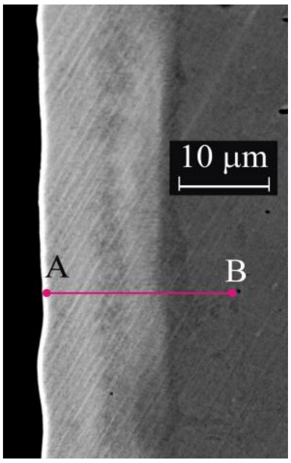
a **–** $\boldsymbol{E}$ **= 20 J/cm^2,** $\boldsymbol{n}$ **= 1 pulse,** ***b*** **–** $\boldsymbol{E}$ **= 9 J/cm^2,** $\boldsymbol{n}$ **= 5 pulses**

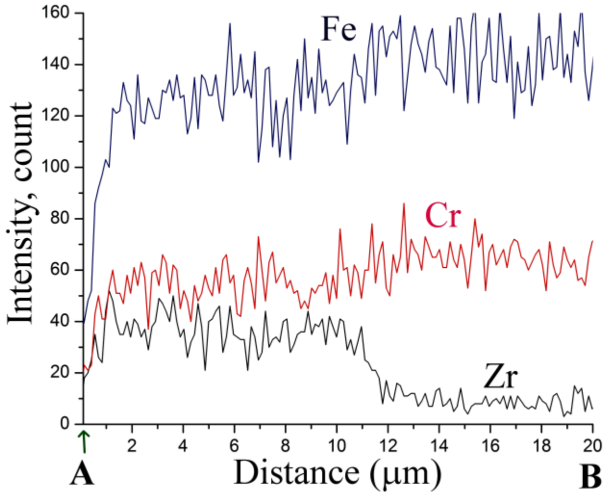
Figure 8. – Cross-sectional SEM images of CPF treated Ta/U8A samples

The increase in the surface layer hardness is due to formation of nitride phases, solid solutions, and intermetallic phases. It has been established that at the energy density, $w = 9$ J/cm^2 and the number of pulses, $n = 5$, a maximum growth in a microhardness value (more than by a factor of 2.5) was reached. It has been shown that with an increase in the number of pulses, the tantalum distribution through the depth of the doped layer becomes more uniform due to repetition of melting, convective mixing, and solidification processes.

The regularities are determined for substantial improvement in the surface properties of widely used industrial X18H10T austenitic steel treated by compression plasma flow. The steel samples coated with the Zr layer (~ 2.5 μm) were processed under the exposure conditions, similar to those for treatment of the Ta/U8A system.

Figure 9 shows a micrograph of the modified sample cross section and the elemental profile recorded by means of energy dispersive X-ray spectral microanalysis along the AB line.





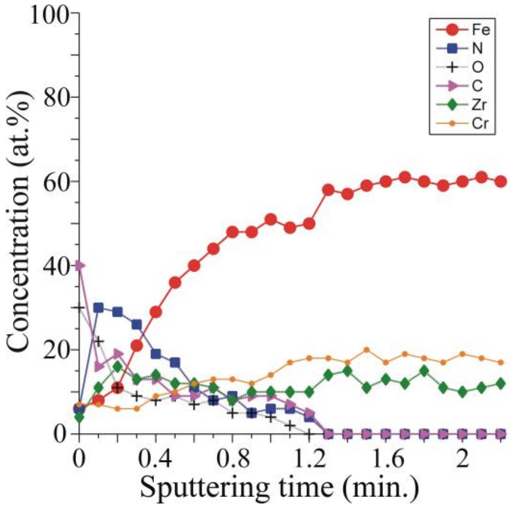
a *b*

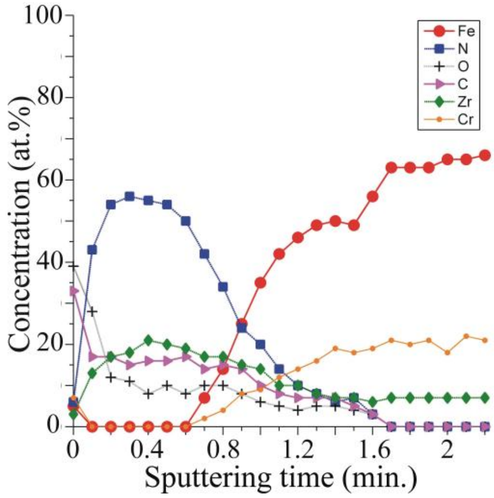
a **– SEM image,** ***b*** **– EDX analysis along AB line**

Figure 9. –Zr/steel system after CPF treatment by five pulses, ***w*** **= 9 J/cm²**

It is shown that the action of CPF at $n = 5$ and $w = 9$ J/cm^2 on the Zr/X18H10T system results both in twofold increase of the surface microhardness and in decrease of the friction coefficient by a factor of more than five.

To study the effects of different regimes of plasma flow treatment on implantation of plasma-forming gas (nitrogen), the alloying element (Zr), and substrate elements (Fe, Cr), a thin surface layer of processed samples was analyzed by Auger electron spectroscopy methods. Results of studies for regime parameters $n = 1$, $n = 5$, $w = 9$ J/cm^2 are shown in Figure 10*a* and 10*b*, respectively.





a **– n = 1 pulse** ***b*** **– n = 5 pulses**

Figure 10. – Auger electron spectroscopy after CPF treatment at 9 J/cm²

It has been shown that increasing the number of pulses of the plasma exposure from 1 to 5 causes an increase in the modified layer depth from 8 to 12 μm and yields a more uniform distribution (as compared with that after single pulse exposure) of the alloying elements (Figure 10*b* shows that with the number of plasma exposure pulses, the nitrogen content in this layer increases).

CONCLUSION

Main scientific results of the thesis

1. Experiments on the exposure of materials to ion beams were conducted using stationary Kaufman-type plasma source with nitrogen and carbon dioxide as working substances, which provides simultaneous acceleration of various types of ions e.g. either N^+ and N_2^+ in nitrogen atmosphere or C^+, CO^+, CO_2^+, O_2^+, O^+, and so on in CO_2 atmosphere. It is shown that irradiation of samples of steel, silicon, a chromium film on silicon, and a chromium film on steel by stationary Kaufman ion beam source with ~ 30 keV nitrogen ions at doses in the range of 10^{16}–10^{18} ions/cm^2 results both in a thin surface layer implantation with formation of nitride phases and in enhancement of surface roughness. It is found that exposed silicon samples show dose-dependent changes in their electrical and optical properties: at a dose of 10^{18} Ni ions/cm^2, the layer sheet resistivity increases up to ~ 180 Ohm/sq. (against ~ 140 Ohm/sq. for an unimplanted sample) [1–5].

2. It is shown, that the Cr/Si and Cr/austenitic steel systems and austenitic steel samples subjected to nitrogen ions, $N^+$and N_2^+, exhibit a substantial dose-dependent increase in their microhardness and corrosion resistance. It is stated that after implanting a dose of 1×10^{18} ions/cm^2, the surface microhardness of the Cr/Si system increases to ~ 1100 kg/mm^2 (from ~ 800 kg/mm^2 prior to implantation), and after annealing of the implanted samples, microhardness reached 1600 kg/mm^2 (versus ~1050 kg/mm^2 for unimplanted samples). It is found that maximum corrosion resistance of the Cr/austenitic steel sample is reached at a dose of 2×10^{18} ions/cm^2 [1–3].

3. By using an optical pressure sensor insensitive to electromagnetic interferences, the pressure was measured on the target subjected to the compression plasma flow generated by the gas-discharge magnetoplasma compressor. It is shown that at nitrogen initial pressure of 400 Pa, initial voltages (U_0) on the capacitor bank in the range of 2–4.5 kV (which corresponds to maximum values of the total discharge current from 53 kA to 126 kA), and at a distance from the accelerator central electrode tip to the target, a = 12 cm and 8 cm, the plasma pressure reaches respectively ~16 atm and ~73 atm. [7, 9, 14, 16, 18].

4. The regularities are determined for substantial improvement in the surface properties of widely used industrial X18H10T austenitic steel treated by compression plasma flow. It is stated that the action of compression plasma flow at $n = 5$ and $w = 9$ J/cm^2 on the Zr/X18H10T system results both in twofold increase of the surface microhardness and in decrease of the friction coefficient by a factor of more than five. It is found that increasing the number of plasma pulses from 1 to 5 causes an increase in the depth of the modified layer from 8 to 12 μm and yields a more uniform distribution of alloying elements (as compared with that after single pulse exposure) [6].

5. It is shown that the action of the 100-μs plasma flow pulse at the 400 Pa initial nitrogen pressure and 9 J/cm^2 absorbed energy density on the Ta/Si system (tantalum layer thickness ~ 1 μm) produces a superficial layer enriched with tantalum silicides and nitrides. It is stated that the number of CPF pulses affects the configuration of nanoscale structures: the single CPF pulse action results in the single-level nanostructures 15–75 μm in size, whereas at the three-pulse plasma impact, these particles additionally merge into spherical clusters ~200–500 nm in diameter [7, 10–12].

6. It is found that under the action of the compression plasma flow on the Ta/Ti/Si system (thickness of tantalum and titanium coatings is ~ 1 μm), a surface layer is formed whose composition is determined by the energy density (w) of the impacting plasma flow: at $w = 3$ J/cm^2, a tantalum-titanium solid solution and tantalum nitride phases (TaN and Ta_2N) are formed; at $w = 9$ J/cm^2, titanium silicide (Ti_5Si_3) is formed, whereas nitride and oxide phases disappear [8].

7. The numerical simulation of temperature fields in surface layer of Ta/Ti/Si and Ta/Si systems exposed to the compression plasma flow was carried out. It was shown that for absorbed energy density 9–13 J/cm^2, the temperature of surface area exceeds the melting points of silicon and titanium, but does not reach that of tantalum, due to which tantalum silicide is not formed [7, 8].

Recommendations for the practical use of the results

Practical significance of the results presented in the thesis is in development of physico-technological backgrounds for new efficient ion-plasma technologies of essential improvement in performance properties of carbon steels, widely used in industry, and for synthesis of silicon carbides and nitrides and tantalum silicide in surface layers of silicon wafers for needs of micro- and optoelectronics.

LIST OF PUBLICATIONS OF APPLICANT ON THE SUBJECT OF THESIS

Articles in peer-reviewed journals

1. Nitrogen implantation and heat treatment effect on the hardness improvement of the chromium film surface deposited on Si(111) substrate / M. Ghoranneviss, A.H. Sari, M. Esmaeelpour, M.R. Hantehzadeh, H. Savaloni // Applied Surface Science. – 2004. – Vol. 237, № 1–4. – P. 326–331.

2. Microstructural and corrosivity changes induced by nitrogen ion implantation on chromium films / A. Shokouhy, M.M. Larijani, M. Ghoranneviss, S.H.H. Hosseini G, M. Yari, A.H. Sari, M.G. Shahraki // Thin Solid Films. – 2006. – Vol. 515, № 2. – P. 571–575.

3. Corrosion behavior of low energy, high temperature nitrogen ion-implanted AISI 304 stainless steel / M. Ghoranneviss, A. Shokouhy, M.M. Larijani, S.H. Haji Hosseini, M. Yari, A. Anvari, M. Gholipur Shahraki, A.H. Sari, M.R. Hantehzadeh // Pramana Journal of Physics. – 2007. – Vol. 68, № 1. – P. 135–140.

4. Structural and optical properties of silicon nitride film generated on Si substrate by low energy ion implantation / D. Dorranian, P. Azadfar, A.H. Sari, S. Ghorbani, A. Hojabri, M. Ghoranneviss // The European Physical Journal - Applied Physics. – 2008. – Vol. 42, № 2. – P. 103–107.

5. Formation of SiC using low energy CO_2 ion implantation in silicon / A.H. Sari, S. Ghorbani, D. Dorranian, P. Azadfar, A.R. Hojabri, M. Ghoranneviss // Applied Surface Science. – 2008. – Vol. 255, № 5. – P. 2180–2184.

6. Austenitic Steel Surface Alloyed with Zirconium Using Compression Plasma Flow / A.H. Sari, V.M. Astashynski, E.A. Kostyukevich, A.M. Kuzmitski, V.V. Uglov, N.N. Cherenda, Yu.A. Petukhou / High Temperature Material Processes, – 2012. – Vol. 16, № 4. – P. 297–313.

7. Phase transformations in Ta-Si system induced by compression plasma flow / A.H. Sari, V.M. Astashynski, A.M. Kuzmitski, Yu.A. Petukhov, V.V. Uglov // The European Physical Journal - Applied Physics. – 2014. – Vol. 65, № 1. – P. 10801.

8. Effect of Compressed Plasma Flow on Tantalum-Titanium Thin Layer Deposited on Silicon Substrate / A.H. Sari, V.M. Astashynski, A.M. Kuzmitski, V.V. Uglov // International Journal of Materials Science and Applications. – 2014. – Vol. 3, № 3. – P. 92–99.

9. Studies on dynamic pressure of compression plasma flow / V.M. Astashynski, A.H. Sari, S.I. Ananin, E.A. Kostyukevich, P.N. Shoronov, A.M. Kuzmitski // Plasma Physics and Technology. – 2014. – Vol. 1, № 3. – P. 157–159.

Articles published in the Proceedings of scientific conferences

10. Effect of compression plasma flow on structure phase transformations of tantalum–silicon system / V.M. Astashynski, A.H. Sari, A.M. Kuzmitski, Yu.A. Petukhou, V.V. Uglov, S.A. Filatov // Plasma Physics and Plasma Technology: Proceedings of VII international conference, Minsk, 17–21 September, 2012. / ed. by V.M. Astashynski, V.S. Burakov, I.I. Filatova – Minsk, 2012. – P. 449–502.

11. Formation of silicon-based nanostructures by compression plasma flow in Ta/Si system / V.M. Astashynski, A.H. Sari, A.M. Kuzmitski, Yu.A. Petukhou, V.V. Uglov, M.A. Andreev // IX Belarusian-Serbian Symposium on Physics and Diagnostics of Laboratory and Astrophysical Plasmas, Minsk, 16–21 September, 2012. – Minsk, 2012. – P.122–125.

12. Nano–sized spherical clusters of tantalum silicide formed by compression plasma flow / A.H. Sari, V.M. Astashynski, A.M. Kuzmitski, Yu.A. Petukhou, V.V. Uglov // Physics chemistry and applications of nanostructures: Proceedings of international conference Nanomeeting-2013, Minsk, 28–31 May, 2013. / ed. by V.E. Borisenko [et al.]. – Minsk, 2013. – P. 407–410.

13. Mixing of “tantalum–carbon steel” system by compression plasma flow / A.H. Sari, V.M. Astashynski, Yu.A. Petukhou // Interaction of Radiation with Solids: Proceedings of 10th international conference, Minsk, 24–27 September, 2013. – Minsk, 2013. – P. 275–277.

14. Parameters of shock-compressed layer near solid objects exposed to compression plasma flow: experimental studies and numerical simulation / V.M. Astashynski, S.I. Ananin, E.A. Kostyukevich, A.M. Kuzmitski, A.H. Sari, P.N. Shoronov // X Symposium of Belarus and Serbia on Physics and Diagnostics of Laboratory and Astrophysical Plasmas, Belgrade, 25–29 August, 2014. – Belgrade, 2014. P. 7–10.

15. Compression plasma flow treatment of tantalum-steel system / A.H. Sari, Astashynski, S.I. Ananin, E.A. Kostyukevich, A.M. Kuzmitski // Energy Efficiency-2014: Proceedings of international conference, Minsk, 14–16 October, 2014. – Minsk, 2014. – P. 101–103.

16. Time-resolved pressure measurements of compression plasma flows / A.H. Sari, V.M. Astashynski, S.I. Ananin, E.A. Kostyukevich, A.M. Kuzmitski, P.N. Shoronov // Energy Efficiency-2014: Proceedings of international conference, Minsk, 14–16 October, 2014. – Minsk, 2014. – P. 127–129.

Abstracts published in books of scientific conferences

17. Dynamics of compression plasma flows interaction with solid targets / S.I. Ananin, V.M. Astashynski, A.H. Sari // Plasma Physics and Technology: Proceedings of 27th international conference, Prague, 16–19 June, 2014. – Prague, 2014. – P. 44.

18. Studies on dynamic pressure of compression plasma flow / V.M. Astashynski, A.H. Sari, S.I. Ananin, E.A. Kostyukevich, P.N. Shoronov, A.M. Kuzmitski // Plasma Physics and Technology: Proceeding of 27th international conference, Prague, 16–19 June, 2014. – Prague, 2014. – P. 87.

РЭЗЮМЭ

Сары Амір Хасейн Махамад Алі

Мадыфікацыя і легіраванне паверхні крэмнію і сталі кампресійнымі плазменнымі патокамі і іённымі пучкамі

Ключавыя словы: кампрэсійны плазменны паток, іённая імплантацыя, мадыфікацыя паверхні, наноструктуры, ўласцівасці паверхні

Мэта працы: ўстанаўленне фізічных заканамернасцяў ўздзеяння плазменных патокаў, генераваных газаразрадны магнитоплазменным кампрэсарам, і іённых пучкоў, генераваных бесперапыннай плазменнай іённай крыніцай Каўфмана, на пласціны крэмнія і вугляродзістых сталей, якое прыводзіць да істотных змен іх паверхневых уласцівасцяў.

Метады даследавання: высакахуткасная фотарэгістрацыя, аптычная інтэрфераметрыя, электронная оже-спектраскапія, сканавальная электронная мікраскапія, рэнтгенаструктурны аналіз, атамна-сілавая мікраскапія.

Атрыманыя вынікі і іх навізна: Установулены заканамернасці фарміравання мадыфікаванага пласта на паверхні вугляродзістых сталей і крэмніевых пласцін, кіравання яго структурай і ўласцівасцямі. Паказана, што ўздзеянне адзінкавым імпульсам кампрэсійнага патоку на пласціны крэмнія з тонкім пластом тантала прыводзіць да плаўлення паверхневага пласта крэмніевай асновы і частковаму плаўленню танталаваго пакрыцця, забяспечваючы фарміраванне ўзбагачанага сіліцыдамі і нітрыдамі тантала наноструктураванага пласта, утворанага часцінкамі дыяметрам 15–75 нм. Уздзеянне серыяй з 5 імпульсаў на ўзоры аўстэнітнай сталі Х18Н10Т з тонкім пакрыццём цырконія прыводзіць да павелічэння цвёрдасці мадыфікаванай паверхні ў ~ 2 разы і памяншэння каэфіцыента трэння ў ~ 5 разоў з прычыны фарміравання нітрыдаў і інтэрметалідаў.

Галіна выкарыстання вынікаў: атрыманыя вынікі могуць знайсці прымяненне ў распрацоўцы новых эфектыўных плазменных тэхналогій для паляпшэння ўласцівасцяў вугляродзістых сталей, якіе шырока выкарыстоўваюцца ў прамысловасці і сінтэзу карбідаў і нітрыдаў крэмнія, сіліцыдаў тантала ў паверхневым пласце крэмніевых пласцін для мікра- і оптаэлектронікі.

РЕЗЮМЕ

САРИ Амир Хоссейн Мохаммад Али

Модификация и легирование поверхности кремния и стали компрессионными плазменными потоками и ионными пучками

Ключевые слова: компрессионный плазменный поток, ионная имплантация, модификация поверхности, наноструктуры

Цель работы: установление физических закономерностей воздействия плазменных потоков, генерируемых газоразрядным магнитоплазменным компрессором, и ионных пучков, генерируемых непрерывным плазменным ионным источником Кауфмана, на пластины кремния и углеродистых сталей, приводящего к существенным изменениям их поверхностных свойств.

Методы исследования: высокоскоростная фоторегистрация, оптическая интерферометрия, электронная оже-спектроскопия, сканирующая электронная микроскопия, атомно-силовая микроскопия.

Полученные результаты и их новизна: Установлены закономерности формирования модифицированного слоя на поверхности углеродистых сталей и кремниевых пластин, управления его структурой и свойствами. Показано, что воздействие одиночным импульсом компрессионного потока на пластины кремния с тонким слоем тантала приводит к плавлению поверхностного слоя кремниевой подложки и частичному проплавлению танталового покрытия, обеспечивая формирование обогащенного силицидами и нитридами тантала наноструктурированного слоя, образованного частицами диаметром 15-75 нм. Воздействие серией из 5 импульсов плазменного воздействия на образцы аустенитной стали Х18Н10Т с тонким покрытием циркония приводит к увеличению твердости модифицированной поверхности в ~ 2 раза и уменьшению коэффициента трения в ~ 5 раз вследствие формирования нитридов и интерметаллидов.

Область применения результатов: Полученные результаты могут найти применение в разработке новых плазменных технологий для улучшения свойств широко используемых в промышленности углеродистых сталей и синтеза карбидов и нитридов кремния, силицидов тантала в поверхностном слое кремниевых пластин для микро- и оптоэлектроники.

SUMMARY

SARI Amir Hossein Mohammad Ali

Modification and alloying of silicon and steel surfaces by compression plasma flows and ion beams

Keywords: compression plasma flow, ion implantation, surface modification, nanostructures

Objective of research: to establish physical regularities in the action of compression plasma flows and ion beams, generated respectively by a gas-discharge magnetoplasma compressor and continuous Kaufman-type plasma ion source, on silicon wafers and carbon steels, resulting in significant changes of their surface properties.

Methods of investigation: high-speed photorecording, optical interferometry, Auger electron spectroscopy, scanning electron microscopy, atomic force microscopy.

Obtained results and their novelty: Regularities are established in formation of a modified layer on surfaces of carbon steels and silicon wafers and in management of its structure and properties. It is shown that the action of a single pulse of a compression flow on silicon wafers coated with a thin tantalum layer results in melting of a silicon substrate surface layer and partial melting of tantalum coating, which ensures the formation of a nanostructured layer enriched in tantalum silicides and nitrides and formed by particles 15–75 nm in diameter. The exposure of X18H10T austenitic steel samples with a thin zirconium coating to a series of 5 plasma pulses causes almost a two-fold increase in the modified surface hardness and lowers its friction coefficient by a factor of ~ 5 due to formation of nitrides and intermetallics.

Scope of results: The obtained results can be applied in development of new plasma technologies to improve properties of industrially used carbon steels and to synthesize silicon carbides and nitrides or tantalum silicides in a surface layer of silicon wafers for needs of micro- and optoelectronics.

SARI Amir Hossein Mohammad Ali

MODIFICATION AND ALLOYING OF SILICON AND STEELS SURFACES BY COMPRESSION PLASMA FLOWS AND ION BEAMS

ABSTRACT
of dissertation for a degree of
doctor of philosophy in physics
in specialty 01.04.08 - plasma physics

Подписано в печать 18 ноября 2014 г. Формат 60x84/16.
Тип бумаги – офисная. Гарнитура «Times». Печать: ризография.
Объем: 1,5 усл. печ. л., 1,1 уч.-изд. л.
Тираж 10 экз. Заказ № 23.

State scientific institution «B.I. Stepanov Institute of Physics,
National Academy of Sciences of Belarus»
68 Nezalezhnasti Ave., 220072, Minsk.
Printed on rizografe in Institute of Physics of NAS of Belarus