New-Generation Installation for Material Processing by Metal Ion Beam and Plasma

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Abstract – A new-generation installation for realization of hybrid methods of ion-beam and plasma modification of materials is described. Conditions and examples of practical application of the equipment comprised in the installation are considered.

I. Introduction

Nowadays the majority of technological processes of ion-beam and plasma processing of materials are realized using energy modification of various types. Growing interest to the given scientific and technological direction calls for the development of new universal equipment for realization of a wide variety of material processing modes.

In the present report, a new-generation research-and-production complex intended for realization of perspective methods of ion-beam and plasma modification of materials is described. To develop the complex installation (CI), already well-known approaches to the realization of hybrid methods were used together with recent advances of the Laboratory of Physics and Technique of Ion-Beam and Ion Plasma Processing of Materials of Nuclear Physics Institute (Tomsk) in the field of development of equipment and methods of modification of materials.

2. Equipment

The external view of the CI is shown in Fig. 1. The specifications of the equipment comprised in the installation are given in the table.

To realize modes of plasma vacuum deposition (PVD) of coatings, the CI was equipped with a set of axisymmetric vacuum-arc evaporators (VAEs) based on continuous vacuum-arc discharge (VAD) adapted for processing of refractory materials [1].

To clean the plasma from microparticle fraction (MPF), the VAEs were equipped with plasma filters (PFs) having the venetian-blind structure [2]. The PF is designed as an optically opaque coaxial electrode system. The charged plasma component propagated in $\boldsymbol{E}\times\boldsymbol{B}$ fields created by current running through the PF electrodes under application of a positive potential with respect to plasma. The ratio of ion saturation currents from plasma at the PF input (I_{pl}) and output (I_f) reaches η~58 %. With the use of the PF, the number of macroinclusions on the coating surface decreased by more than 10^2–10^3 times. Additional decrease of the MPF was recorded at distances exceeding the length of the geometrical focus of the converging filter electrodes (~16 cm).

Gas plasma streams were produced using a low-pressure arc discharge from a heated cathode [3]. The PINK plasma generator produced sufficiently uniform plasma with concentration of 10^9–10^{10} cm^3 in volumes exceeding 1 m^3. The plasma produced by the source was not polluted by the cathode material, because the discharge was diffusion in character and formed no cathode spot.

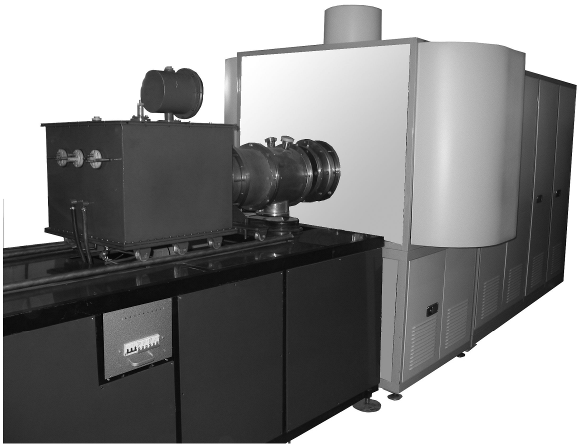

Fig. 1. The external view of the complex installation

A magnetron sputtering system (MSS) was formed by a dual magnetron with direct target cooling by running water [4]. The magnetrons insulated from each other were connected to a power supply unit that generated rectangular voltage pulses with a frequency of ~$4\cdot10^4$ s^{-1}. During one half-cycle, magnetrons alternatively operated as the MSS cathode and anode. In this case, high stability of the discharge was ensured with almost complete absence of the MPF without application of additional arc quench circuits, and high rate of coating deposition was provided for a wide variety of reaction gases. The application of the dual magnetron system ensured that current did not run through the substrate thereby decreasing the probability of microarc occurrence on the substrate.

Accelerated ion streams were formed under application of high-frequency short-pulsed negative potential to parts of machines immersed into the plasma.

Alongside with the conventional advantages of plasma-immersion processing of materials (absence of ion-optical systems, opportunity of processing of large surface areas and parts of complex shape), the suggested approach reduces the power consumption of the high-voltage generator and the probability of microarc occurrence on the surface of processable parts of machines and allows parts with small curvature radius to be processed and modes of ion-beam and plasma processing of materials to be combined. The main advantage of the method of high-frequency short-pulsed plasma-immersion ion implantation or deposition (HFSP^{2}I^{3}D) is the opportunity of effective processing of dielectric materials.

Processing of dielectrics is realized through the application of a short-pulsed bias potential to a conducting sample holder. The electric field arising at the dielectric surface is determined by the thickness and permittivity of the dielectric and the dynamics of ion accumulation on the dielectric surface together with the plasma parameters and bias potential [5]. Duration of bias potential pulses is determined by the charge time of the capacity whose plates are formed by the emission plasma boundary and the potential electrode. For the plasma producing the ion current density from a few units to a few tens of mA cm^{-2}, the bias voltage pulse duration used for processing of dielectrics changes, as a rule, from several fractions of a microsecond to several microseconds.

When the bias potential is switched off, the charge accumulated on the dielectric surface is compensated by the plasma electrons. The time of charge compensation does not exceed several nanoseconds, which actually determines the allowable pulse duty factor.

For the case of formation of a rectangular bias potential pulse (τ=1.5 μs, U_{acc}=–3 kV, and n_i=2·10^{15} m^{-3}), it was demonstrated that the mode corresponding to a pulse duty factor of 0.3 involves predominant coating deposition under conditions of pulsed-periodic ion mixing. For a pulse duty factor of 0.66, plasma deposition is almost absent. In this case, the time interval between successive pulses is chosen to be equal to the time of plasma boundary approach to the sample after the pulse termination. The given mode corresponds to high-frequency plasma-immersion implantation from the metal plasma. This mode is most clearly expressed for a pulse duty factor of 0.9, when the dip in the energy spectrum is insignificant with allowance for the presence of ions with different charge states in the stream.

The mode of forming ion beams with energy in the range (20–160) keV is realized using the Raduga-5 source of accelerated ions and plasma. The source combines the continuous mode of generation of metal plasma cleaned from the MPF with pulsed-periodic formation of a metal ion beam in the diode formed by the PF electrodes and the grounded grid electrode. To shield the secondary and plasma electrons from the accelerating gap, an additional grid with a negative potential [6] is included in the source design.

For U_{acc}=40 kV, the field penetrates into the interelectrode gaps of the PF and forms the emission plasma boundary with focusing properties. Due to this, losses of high-energy plasma ions on the PF electrodes are reduced, and the ion beam current at the output from the filter exceeds by 5–10 % the plasma ion saturation current without accelerating voltage.

The combination of the plasma generator with the ion source allows us to combine easily the modes of ion and plasma processing of materials. The average ion beam power can be changed in a wide range by changing the VAD current and the accelerating voltage pulse amplitude and repetition frequency.

The equipment comprised in the CI is mounted on a universal vacuum post comprising a vacuum chamber with a planetary gear mechanism for sample motion with sizes of the working volume 2.65×1.2×2.16 m^3.

VAE with PF*	
Discharge current, A	40–300
Plasma concentration, cm^{-3}	up to 10^{11}
Degree of plasma cleaning from the MPF	>10^2–10^3
Transmission coefficient for the ion plasma component (Ti)	up to 0.58
Rate of coating deposition, m/h	up to 20
Number, units	4–8
PINK*	
Working pressure, Pa	10^{-2}–1
Plasma concentration, cm^3	10^{10}
Discharge current, A	5–200
Ion current density from plasma, mA/cm^2	1–10
Number, units	1–3
DUALMAG Systems	
Target sizes, m	0.8×1×0.1
Working pressure, Pa	0.04–0.1
Discharge voltage, V	350–1000
Discharge current, A	1–30
Frequency of discharge generation, s^{-1}	up to4 10^4
Raduga-5 source of accelerated ions and plasma	
Ion beam current, A	up to 2
Ion energy, keV	up to 160
Accelerating voltage pulse duration/frequency, ms/s^{-1}	400/200
High-frequency short-pulsed voltage generator for plasma-immersion ion and plasma processing of materials	
Pulse amplitude, kV	0– 4
Pulse repetition frequency, s^{-1}	(2–4.4)·10^5
Pulse duration, μs	0.5–2

* Parameters are specified for a single source

The vacuum chamber is equipped with a turbomolecular pump, gas bleed-in system, various diagnostic equipment including infrared and thermocouple sensors for measuring the temperature and pressure, system for heating and cooling of chamber walls, etc. The vacuum post can be controlled automatically or from the operator terminal. The CI can be additionally equipped with a plasma-immersion transit-time spectrometer for investigation of plasma mass and charge composition [7].

3. Operating conditions for the CI equipment

The capability of hybrid operation of the CI equipment is determined in many respects by the pressure in the vacuum volume. According to the data presented in the table, the working pressure is (10^{-5}–1) Pa depending on the equipment type and technological conditions of its operation. For pressures in the range (10^{-3}–10^{-1}) Pa, the CI units operate simultaneously. This allows a wide variety of hybrid methods of ion-beam and plasma processing of materials to be developed, including processing of materials in a reaction gas medium and realization of unconventional operation modes. For example, the plasma formed by VAEs provides a working medium for MSS at reduced pressure.

To provide long continuous CI operating lifetime (up to several tens of hours) and to simplify operation and service of the Raduga-5 source and VAEs, the unified PFs, massive conic cathodes, and ignition elements were used. The special features of the vacuum chamber design allowed its volume to be increased and shutters and transfer chambers to be used. Depending on technological problems, up to 8 VAEs can be placed in the vacuum chamber.

4. CI application for hybrid modes of ion-beam and plasma processing of materials

Fig. 2 shows modes of energy modification of the materials realized with the use of the CI. Under conditions of successive or joint ion-beam and plasma modification, the developed technological complex allowed us to realize modes of surface cleaning and activation, high-intensity and high-concentration ion implantation, formation of transition layers between the base and coating, and deposition of coatings under conditions of intensive ion mixing.

The feasibility of qualitative improvement of traditional PVD methods of deposition of TiN and TiAlN coatings on HSS samples with the use of the VAE equipped with PF and the PINK plasma generator was demonstrated in [8].

Joint application of metal and discharge plasma cleaned from the MPF contributed to an increase in the adhesive strength, wear resistance, and hardness of coatings, decrease of the friction coefficient and surface roughness, and formation of uniform coating structure with the preset stoichiometric structure. The pressure in the vacuum chamber was reduced at the expense of effective ionization of a reaction gas.

The mode of forming transition layers between the coating and substrate was realized in the variant of separate and joint application of HFSP^{2}I^{3}D and the Raduga-5 source.

Fig. 3 demonstrates the effect of the HFSP^{2}I^{3}D mode on the increase in the adhesive strength of the Ti coating deposited on HSS steel and ceramics. Investigations were carried out using the scratch test [9]. The object of research was the critical indenter load at which the coating was destructed. The results obtained vividly demonstrate an increase in the adhesive strength for both metal and dielectric samples. The decrease of the surface roughness of the metal samples with increase in the bias potential was also observed.

The feasibility of application of the Rduga-5 source for realization of the mode of intensive ion-assistant modification is illustrated by the example of Cu film deposition on teflon. The formation of a transition layer and coating deposition in the thermal mode close to the melting temperature allowed us to create the structure whose adhesive strength could not be measured with the use of the scratch test.

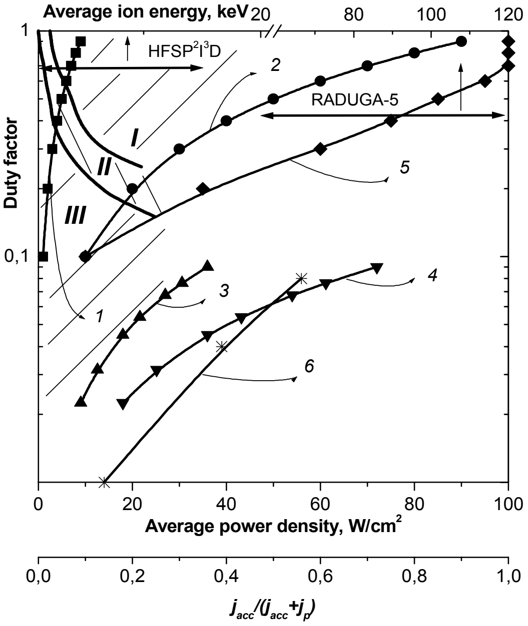

Fig. 2. Variation range: average ion energy for HFSP^{2}I^{3}D and Raduga-5; average ion power density (1–4) for j_{acc}=10 mA/cm^2 and ratio of accelerating ion current density to jacc to plasma ion current density j_p (5,6) of the duty factor. 1,2 – HFSP^{2}I^{3}D (E_i=1 keV and 10 keV respectively), 3,4 – Raduga-5 (E_i=40 keV and 80 keV respectively), 5 – HFSP^{2}I^{3}D, 6 – Raduga-5 (E_i=80 keV at a distance of 50 cm from the ion source). Range of the processing modes realization for HFSP^{2}I^{3}D (Cu plasma): I – Ion implantation without plasma deposition, II – High concentration ion implantation, III – Ion assisted coating deposition

Results of investigations of TiSiB coating deposition on samples from VT-9 alloy under conditions of successive application of an ion beam (E_i=(20–60) keV, j_{acc}=10 mA/cm^2, τ~400 μs, and f=200 s^{-1}) from the Raduga-5 source and of the HFSP^{2}I^{3}D mode were presented in [8]. The radiation-stimulated diffusion mechanism of forming a doped layer caused a smooth character of doping impurity concentration distribution from the target surface to depth as great as 1 μm.

The coating was deposited with the use of an additional VAE with PF having composite TiSiB cathodes. In the HFSP^{2}I^{3}D mode for coating 6 μm thick, the formation of an amorphous layer was observed in the surface layer of the target at depths up to 25 μm. The presence of the deep modified layer promoted an increase of the ultimate strength under cyclic sample loading by two orders of magnitude. The ion-assisted increase in the coating density resulted in a 20-fold increase in the resistance to salt corrosion of samples in the presence of the chlorine ion at elevated temperatures under conditions of thermal cycling.

The Raduga-5 source comprised in the CI in combination with HFSP^{2}I^{3}D provided wide opportunities for realization of modes of ion modification of surface layers of materials.

In particular, ion implantation without coating deposition can be realized through joint application of the Raduga-5 source and HFSP^{2}I^{3}D. In this case, the time interval between the subsequent bias potential pulses was chosen less than the time of plasma approach to the surface of a processable sample.

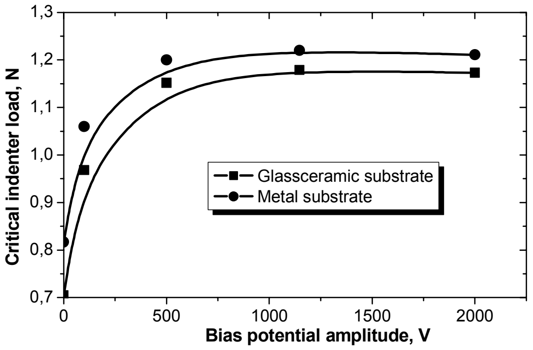

Fig. 3. Ti coating delamination critical force depending on bias potential pulse amplitude. Duty factor is 0,42

High-concentration ion implantation was realized with the use of the Raduga-5 source with compensation of the layer sputtered from the target surface by plasma deposition of the same material as the implanted ions [10]. The concentration of the doping impurity can exceed the irradiation doze and reach 100 at. % near the surface at the expense of incorporation of implanted material atoms as recoil ones from the surface deeper into the targets. As in HFSP^{2}I^{3}D, the plasma interaction with the surface of materials during time intervals between subsequent accelerating voltage pulses allows the accumulation of significant charge on dielectric materials during ion-beam processing to be avoided.

The modified layer depth and the concentration of doping impurity can be increased under conditions of high-intensity ion implantation. The given mode was realized in [12] for processing of a Ti target by an ion beam (j_{acc}=3.5 mA/cm^2 and E=40 keV). The doped layer with thickness greater than 2 μm was formed in 2 h with the formation of the Ti-Al intermetallic systems having the concentration of doping impurity up to 60 at.% and the integral doze of incorporated atoms up to $3.6\cdot10^{18}$ cm^2. The average size of phases generated in the mode of high-intensity ion implantation of intermetallic compounds was (24–65) nm. Investigations of the modified layers confirmed their high tribological characteristics at temperatures from 25 to 400 °C.

5. Conclusions

The new-generation CI has been developed for realization of the hybrid methods of high-intensity and high-concentration ion implantation, HFSP^{2}I^{3}D, and PVD under intensive ion-assisted conditions. The principle of CI design is based on the use of high-frequency short-pulsed potentials for the formation of plasma streams and accelerated ions. The feasibility of forming deep (>2 μm) modified layers with high concentration of doping impurity, creation of wide transition layers between the substrate and coating, improvement of physical and exploitation properties of coatings formed on conducting and dielectric materials with the use of CI equipment and the suggested methods of processing of materials has been demonstrated.

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