Abstract – The results of investigations of applicability of the method of high-frequency short-pulsed plasma-immersion ion implantation and (or) coating deposition using vacuum-arc and ablation plasma to conductive and dielectric substrates are presented. It is shown that ion implantation with the ion sputtering compensation by coating deposition from plasma and ion-assisted coating deposition can be realized for the metal and dielectric samples through alteration of the negative bias potential within 0–4⋅10³ V, with the pulse repetition rate of (1–4.4)⋅10³ pps, pulse duration 0.5–2 µs and duty factor of 0.1–0.9. It is experimentally established that at coating deposition from ablation plasma obtained by the influence of the high-intensity ion beam (j = 3 ⋅ 10² A/m², E = 350 keV, τ = 90 ns) on the target, the micro-arc effects on the substrate surface are observed at a dc bias potential of more than –60 V. The transition to pulses of 0.5 µs duration enabled to increase the bias potential up to –4 kV. The possibility of application of the high-frequency, short-pulse bias potentials for the formation of coatings from vacuum-arc and ablation plasma with high adhesive strength and improved exploitation characteristics is discussed.

1. Introduction
Plasma-immersion ion implantation (PI³) with the application of the gas-discharge plasma was suggested in papers [1, 2] and has been studied presently in detail both for the pulsed and dc plasma generation modes [3–7]. The problems of realization of the methods of PI³ from plasma of conductive materials [8–16] were investigated to a lesser degree. In paper [9], PI³ combined with coating deposition was realized for the metal plasma with the bias potential pulse duration of 3–10 µs and the duty factor of 0.1–0.5. The plasmamersion approach at the realization of the method of high-concentration ion implantation with the surface ion sputtering compensation by plasma deposition was elaborated in papers [17–19]. The pulse arc discharge with duration of 50–400 µs was used in the investigations. The ion implantation was provided by the dc bias potential source, while the mode of high-concentration ion implantation was achieved using the bias potential pulse duration less than the plasma generation pulse duration. The single reference to the PI³ method realization on dielectric materials was given in paper [20], where the conductive grid electrode with high transparency was applied to the substrate surface for the ion flow formation.

The presented paper is devoted to the investigation of the possibility of the high-frequency short-pulse plasma-immersion ion implantation and (or) coating deposition (HFSP³CD) [21, 22] method realization with respect to the metal vacuum-arc and ablation plasma.

2. Physical model of the HFSP³CD method
The processes of the ion flows formation from the vacuum-arc and ablation plasma have similar physical principles and specific peculiarities for conductive and dielectric materials. When using metal substrates the load of the bias potential pulse generator has both the capacitive and active components. The capacitive component appears only during the transient process, when the plasma sheath grows to the stationary state, determined by the Child–Langmuir law or fixed by the distance between the samples and the grid electrode forming the emission boundary of plasma. PI³ from the vacuum-arc plasma on the metal substrates, taking into account the active component of the pulse generator load and the presence of directed velocity of the plasma flow propagation, can be realized both at dc and pulsed bias potentials on the holder and correspondingly on the sample surface [22].

The situation is different when dielectric substrates are used. If the dielectric sample entirely covers the conductive holder, the active component of the load is completely excluded and the processes of the ion flow formation are determined by the thickness and dielectric permeability of the substrate, dynamics of the charge accumulation on the sample surface, parameters of plasma and bias potential pulse [22]. If plasma density is high, charging of capacity, formed by the dielectric substrate surface and the potential electrode (holder), will be finished quickly enough. Therefore, the application of the bias potential pulses of duration exceeding the dielectric surface charging time seems inappropriate. In this connection it is important to determine the parameters of plasma, substrate and bias potential pulse required for the realization of the ion flow generation modes optimal in terms of energy. In case when the bias potential pulse duration is less than the ablation plasma pulse duration, the electric field...
occurs between the plasma and the charged surface of the dielectric sample after the bias potential shutdown on the holder. As a result, not ions but electrons are extracted from plasma. The current and mobility of electrons are much greater than that of ions, so the charge compensation on the surface will happen almost instantly. Simulation show that time of charge compensation does not exceed several nanoseconds. In practice the charge compensation time determines the acceptable duty factor for realization of HFSP$^{2+}$CD modes. Application of the bias potential pulse of microsecond duration and the pause duration of a nanosecond second allows to increase the duty factor practically to the unit. On the other hand it means that for high values of $f/τ → 1$ (where $f$ is the bias pulse repetition rate, $τ$ is the bias potential pulse duration) not only the plasma deposition mode, but also the modes of conventional and high-concentration ion implantation can be realized [23].

The vacuum-arc and ablation plasma differ in concentration by several orders. Thus, for example, concentration of the vacuum-arc metal plasma, filtered from microparticle fraction using shutter-type plasma filters, usually does not exceed $5 \cdot 10^{10} \text{ cm}^{-3}$. Concentration of ablation plasma at distances used for the coating deposition ($0.1–0.4 \text{ m}$) is $10^{12}–10^{14} \text{ cm}^{-3}$. In the last case it means that at bias potential supply to the target, there will be formed a sheath less than 1 mm. The electric field strength in the layer of charge separation and especially near the potential electrode will be largely determined by the surface microrelief. The electric field strength growth near the peaks on the substrate surface will be accompanied by the increase in the electron current and growth of probability of the cathode spot initiation. The thickness of the charge separation layer and the velocity of the plasma torch propagation, formed at a micro arc discharge, determine the time of development of the electric breakdown between the emission plasma boundary and the potential electrode. In this connection, with respect to ablation plasma, the use of a dc bias or bias pulses of high duration does not have the prospects of practical application for realization of methods of the ion-beam and ion-plasma modification of material surface properties.

3. Equipment and methods of experimental investigations

Investigations of possibilities of the HFSP$^{2+}$CD method with regard to the vacuum-arc plasma were performed on the complex installation for realization of hybrid technologies of ion-beam and ion-plasma material treatment [24]. At the experimental investigations, the installation comprised two vacuum-arc evaporators equipped with the shutter-type electromagnetic plasma filters for plasma filtering from microparticle fraction [25] and the vacuum-arc generator of gas plasma with the hollow cathode [26]. Metal and dielectric samples were placed on a conductive holder that was used both in the experiments with the vacuum-arc and ablation plasma. The bias potential short in duration was applied to the holder.

The experimental investigations on application of the HFSP$^{2+}$CD method for realization of the ion-beam and plasma material treatment modes using ablation plasma were performed using the source of intense pulsed ion beams TEMP [27]. The magnetically-insulated diode with the external magnetic insulation is used in the accelerator for the generation of intense pulsed ion beams with the energy of up to 350 keV, current density up to 200 A/cm$^2$ and duration of 90 ns. The type of the diode system being used is characterized by a long lifetime and stability of the ion beam parameters. The high-potential electrode of the accelerator is made of graphite. The nanosecond generator of the accelerator forms two high-voltage pulses of opposite polarity with the amplitudes of 150 and 300 kV. The first pulse is used for generation of the explosion-emissive plasma; the second one of negative polarity is used for the ion acceleration. The beam of accelerated ions contains carbon ions and protons in the ratio of 70 and 30%, respectively.

The ion beam, formed in the magnetically-insulated diode, was focused on the titanium target. The target was located angularly to the incident beam so that vapor and plasma torch propagating along the normal to the target surface, fell on the potential electrode, placed at the distance of 0.1 m from the sputtered target.

4. Results of experimental investigations

The results of experimental investigations of the adhesive strength of coatings, formed at Ti vacuum-arc plasma deposition depending on the amplitude of pulsed bias potential are shown in Fig. 1. The coatings were deposited on substrates of ceramics and stainless steel at the duty factor of 0.42. As is seen from the presented results, the critical force, at which the coating destruction takes place, is increased from 0.5 N for coatings deposited on the metal sample without bias, to 1.2 N for cases, when the amplitude of the bias voltage is $-500 \text{ V}$. The further increase in the bias potential amplitude to $-2 \text{ kV}$ resulted in the growth of the critical force to $1.23 \text{ N}$.

![Fig. 1. Dependence of alteration of the critical force on the bias potential on the sample holder](image)
The coating deposited on the ceramic sample demonstrated similar behavior at destruction by diamond indenter. Thus, for coatings formed without the bias voltage, the critical force was 0.3 N, and with bias potential of 500 V the corresponding coating characteristic increased to 1.16 N with the subsequent growth to 1.18 N for high values of the bias potential amplitude.

The nitriding modes at an enhanced intensity of the ion flow were realized in the experiments on treatment of the samples of the VT6 alloy. The nitriding pressure in the vacuum bulk was 0.6 Pa, bias potential amplitude was –2.4 kV, pulse repetition rate was \(2 \cdot 10^5\) pps, duty factor was 0.6, average ion current on the samples was 250 mA and the treatment time was 240 min.

In this case, the maximum hardness values \(H_v = 12.1\) GPa exceed twice the level achieved in a less intensive treatment mode \((-1.5\) kV) and are 2.5 times greater than the hardness of the initial alloy. At that, the stable increase in hardness of the sample surface layer, taking into account the diffusion processes, is observed at the depth of more than 2 µm. For this experimental conditions, the dose of nitrogen introduced into the surface layer of the sample \(D_{em} = 10^{19}\) cm\(^{-2}\). The radiation dose reached \(D_{rad} = 7.7 \cdot 10^{19}\) cm\(^{-2}\).

The experiments with ablation plasma showed that the ion-plasma treatment modes without the explosive-emissive processes on the substrate surface can be realized at dc bias potentials not exceeding –50÷60 V. At realization of the HFSP\(^{2I3CD}\) method with regard to ablation plasma, the bias potential amplitude changed in the range of \(0–4 \cdot 10^3\) V, pulse repetition rate varied from 2 to \(4.4 \cdot 10^5\) pps, and pulse duration varied from 0.5 to 3 µs.

Figure 2 shows the oscillograms of the current on the sample in case of application of a pulsed bias potential of 3 µs duration with pulse repetition rate of \(1.6 \cdot 10^5\) pps to it. It follows from the presented data that the current amplitude of various pulses clearly determines the moment of plasma reaching the collector and dynamics of its concentration change in time. The total duration of the plasma flow is 20–30 µs. Time of the plasma reaching the sample after the ion beam generation is about 10 µs. Thus, the velocity of plasma propagation is about \(10^6\) cm/s. At the bias potential of –250 V (Fig. 2, a, b) the current pulses appear quite smooth, and the breakdowns are not observed.

The oscillations indicating the breakdown are observed when the bias potential pulse amplitude increases to –500 V (Figs. 2, c). It is typical that the breakdown begins not from the very start of the bias pulse.

Taking into account this effect, the bias potential pulse duration was reduced to 1 µs in the next series of experiments. Typical oscillograms of the current pulses for this case are shown in Fig. 3. The analysis of the oscillogram shows that even the increase in the potential amplitude to –2 kV does not cause any obvious breakdown during the bias pulse.

![Fig. 2. Ion current on the sample at ablation plasma deposition: \(\tau = 3\) µs, \(f = 1.6 \cdot 10^5\) pps; \(U_{bias} = -100\) (a), –250 (b), and –500 V (c)](image)

![Fig. 3. Ion current on the sample at ablation plasma deposition: \(\tau = 1\) µs, \(f = 2.5 \cdot 10^5\) pps; \(U_b = -2\) kV](image)

The subsequent reduction of the bias potential pulse duration to 0.5 µs allowed the increase in the bias potential amplitude to 4 kV. The results of investigations of the adhesion strength of a Ti coating deposited using ablation plasma in HFSP\(^{2I3CD}\) mode are shown in Fig. 1. It is evident from the presented data that the critical force of the load on indenter required for the coating exfoliation grows as the bias potential amplitude increases both for metal and ceramic substrates.

5. Conclusion

The performed investigations allow to make a conclusion of the possibility of application of high-frequency, short-pulse bias potentials of sufficient amplitude for the realization of efficient plasma-immersion ion implantation and ion-assisted deposition of dense metal vacuum-arc and ablation plasma modes. In case of ablation plasma application, the pulse duration should be less than 1 µs. It is obvious, that for the energy efficiency of such bias potentials it
is necessary to increase the pulse repetition rate to the value providing the ion mixing of the coating deposition. Taking into account that the ablation plasma allows to achieve the ion current up to several hundreds of amperes, the design of the generator operating in a dc mode seems inappropriate owing to the complexity of performance. At the same time the generator capable of formation of a succession of the bias potential pulses of the given duration and repetition rate, adapted for the ablation plasma pulse duration can be realized for practical applications.

References