



CREATION OF A NEW CLASS OF NANOCOMPOSITE COATINGS OF THE INCREASED CRACK RESISTANCE AND HARDNESS ON THE BASIS OF INNOVATIVE BEAM TECHNOLOGIES

Grigoriev Sergey N.

Moscow State University of Technology "STANKIN", Moscow, Russia

E-Mail: science@stankin.ru

ABSTRACT

Article is dedicated to development of the scientific and technological principles allowing by means of innovative beam and plasma technologies to receive a new class of heterogeneous nanocomposite coatings having the increased cyclic crack resistance and hardness on the conductive and dielectric complex-shaped products. On the basis of the received results the source of metal atoms and beams of high-energy molecules with a rectangular target was developed and new installation for coating deposition is made.

Keywords: nanocomposite coating, glow discharge, electrostatic confinement, electrons, fast neutral gas molecules.

INTRODUCTION

Analytical study of the experience of coating deposition showed that the crack-resistant nanocomposite materials and superhard coatings with a wide interface and excellent adhesion to the products have so far been obtained only on a flat conductive substrates using magnetron sputtering by feeding the substrate by voltage pulses of negative polarity with a duration of 20 μ s and an amplitude of 30 kV at a repetition frequency of 25 Hz [1, 2].

High-energy ions knock atoms of the material from the nodes of the crystal lattice in the surface layer with thickness of a few tenths of a micrometer and mix the atoms of the substrate and the coating in this layer. This mixing allows synthesizing superhard and crack resistant nanocomposite coatings with a thickness of 0.1 mm and with an interface width of 1 μ m or more.

Indeed, when to the substrate spaced on 100 mm from the titanium target of the magnetron filed is applied a negative voltage 80 V, synthesizes a known coating which has golden color and a composition of TiN with a microhardness of 2500 HV, columnar structure and the width of the interface less than 0.1 μ m. If the substrate is fed with pulses of 20 μ s and an amplitude of 30 kV with a repetition rate of 25 Hz, deposits a dark purple nanocomposite coating with the width of the interface noticeably greater than 1 μ m, and a hardness of 5000 HV, consisting of grains of TiN and Ti₂N (more recent) with sizes less than 10 nm.

The above-mentioned articles were published about 10 years ago. However, so far their results have not been used in industry for the synthesis of the considered coatings on the products made of dielectric materials with complex geometric shapes.

In the synthesis process on the dielectric products it is necessary to create the same physical conditions, that on a flat conducting substrate when applying to them high-

voltage pulses. First, is better to bombard the dielectric products with the neutral molecules with high energy [3-5]. Secondly, the trajectory of the slow metal atoms and high-energy molecules must be the same. To do this, it is necessary to develop a source of metal atoms, coincident with high-energy molecules pulsed beams with the total for particles of both sorts emission surface [5]. The most important task is the generation of ion emitter, optimal for such a source.

STUDY OF THE GENERATION OF ION PLASMA EMITTER BY USING GLOW DISCHARGE WITH ELECTROSTATIC CONFINEMENT OF ELECTRONS

To confine the electrons were used broad emission grids and the hollow cathodes having an internal volume from 0.005 to 0.12 m³. In the latter case, the emission grid was absent, and as a hollow cathode, which is an electrostatic trap for all electrons, was used a vacuum chamber with a diameter of 0.5 m and a length of 0.6 m with the anode located inside. When between them was applied a voltage of 200-500 V at a gas pressure of 0.1 to 1 Pa, the glow discharge is ignited, the electrons emitted by hollow cathode and fast electrons formed in the cathode layer, are reflected hundreds of times from different sites of the cathode surface S and become isotropic. Before getting on the anodic surface they make inside the trap the path with length

$$L = 4V/S_a, \quad (1)$$

which is hundreds of times greater than the width of the trap

$$a = 4V/S. \quad (2)$$



They manage to visit all parts of the trap and therefore are distributed in its volume V sufficiently homogeneously. As a consequence, the probability of ionization of gas with these electrons is distributed uniformly, and the plasma filling the trap is also fairly uniform. Contribution to the ionization of electrons formed in equipotential plasma is negligible, because their average energy does not exceed 1 eV.

Fast electrons, oscillating in the trap, ionize the gas not only in the discharge plasma, but also in the cathode layer. The initial energy ε_0 of the electron formed in the layer corresponds to the potential difference between the plasma and the point of its formation. It can amount to hundreds of electron volts, and this electron can form inside the chamber the number of ions $N_\varepsilon = \varepsilon_0/W$, where W is the cost of gas ionization. The ionization efficiency depends on the energy ε averaged mileage $\lambda = 1/n_0\sigma(\varepsilon)$ of the electrons emitted by the cathode, with an initial energy eU_c

$$\lambda_N(p, U_c) = \frac{1}{(eU_c - W)} \int_W^{eU_c} \frac{d\varepsilon}{n_0(p) \sigma(\varepsilon)}, \quad (3)$$

where n_0 is the density of molecules, depending on pressure p and the gas temperature, σ - ionization cross section, a - cathode potential drop, and also on the length of the collisional relaxation of these electrons - the path that they should make in the gas to spend all their energy on the excitation and ionization of the gas.

$$\Lambda = (eU_c/W) \lambda_N. \quad (4)$$

In the optimal pressure range, where $\Lambda < L$ и $\lambda_N > a$, fast electrons spend all their energy on ionization, and the cathode drop does not depend on p . It depends only on the fraction β of the ions incoming to the cathode from the total number of ions formed in the discharge, on the coefficient γ of ion-electron emission of the cathode and the ratio d/a , where d is the width of the cathode layer. Consequently, the cathode drop U_c depends only on the discharge current I , which determines the value of d/a . With increasing current U_c also increases and tends to a limit $U^* = W/e\beta\gamma$.

When $\lambda_N > a$, the number n of ions formed in the trap of fast electrons created by ionization of gas in the cathode layer by one electron emitted by the cathode, equals to

$$n = C(d/a)N^2, \quad (5)$$

where $N = eU_c/W$ - the maximum number of ions formed by the one cathode emitted electron, and the coefficient C depends on N and linearly increases from $C = 0.2$ for $N =$

10 to $C = 0.36$ for $N = 20$ and $C = 0.5$ for $N = 30$. The equation of self-maintained discharge:

$$\beta\gamma(N+n) = 1 \quad (6)$$

and the law of Childe-Langmuir for the current density of ions in the cathode layer allow to calculate current-voltage characteristics (CVC) of the discharge. In the optimal pressure range they do not depend on the size of the trap. Experimental CVC of the discharge with a hollow cathode with volume $V = 0, 12 \text{ m}^3$ and the inner surface $S = 1, 5 \text{ m}^2$ with the area of the anode $S_a = 0.016 \text{ m}^2$ and a pressure of argon of 0.4 Pa, presented in Figure-1 as circles, allow us to determine the coefficient $\gamma \approx 0.06$ for the material of the chamber.

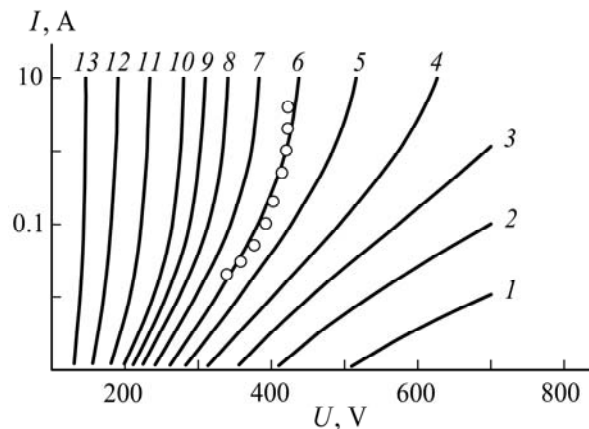


Figure-1. Current-voltage characteristics (CVC) of discharge in argon calculated for $\gamma = 0.01$ (1), 0.02 (2), ... 0.09 (9), 0.1 (10), 0.12 (11), 0.15 (12) and 0.2 (13) and experimental CVC (circles) of the discharge with a hollow cathode with a volume of 0.12 m^3 .

At pressures smaller than the lower boundary p_0 of the optimal pressure range satisfying the equation

$$\Lambda(p) = L, \quad (7)$$

The electrons emitted by the cathode spend only part of their energy on gas ionization, and the rest goes to heating of the anode. In this case, the number of ions formed by them is less than N , and to keep the magnitude of the discharge current is necessary, increasing the cathode drop U_c , to increase the width d and the volume of the cathode layer, and hence the number n of ions formed by fast electrons from the layer.

With decreasing of the gas pressure the cathode fall U_c can grow up to 3 kW, and the number of ions formed by the cathode emitted electrons reduces, and they pass the ionization baton to the subsequent generations of electrons formed in the cathode layer. When U_c increases



dramatically (Figure-2), the pressure tends to a limit, which is the pressure of the extinction of the discharge and which satisfies the equation

$$\lambda_0(p) = L, \quad (8)$$

where λ_0 is the energy-averaged mileage between the ionizing collisions of electrons with energy eU_0 , and U_0 - cathode drop of the discharge at a gas pressure $p = p_0$.

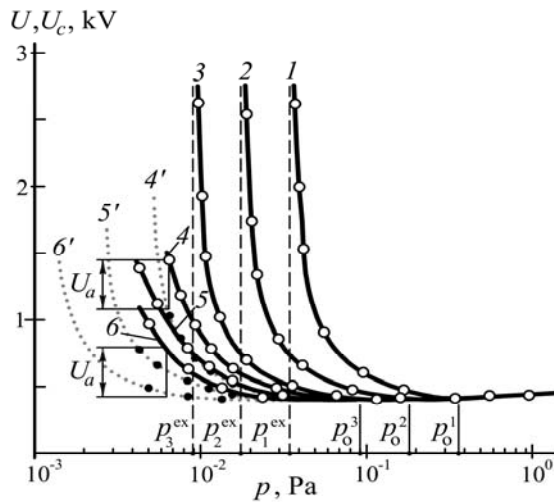


Figure-2. Dependence of the voltage between anode and cathode (solid curves) and cathode potential drop (solid and dotted curves) from the argon pressure p in the hollow cathode with a volume of 0.12 m and the internal surface area of 1.5 m² at a current $I = 0.5$ A and the area of the anode $S_a = 0.064$ (1), 0.032 (2), 0.016 (3), 0.008 (4), 0.004 (5) and 0.002 m² (6).

Pressures p_0 and p^{ex} are directly proportional to the area of the anode S_a and are reducing until S_a decreases to a critical value

$$S^* = (\pi/e)^{1/2} (2m/M)^{1/2} S \approx (2m/M)^{1/2} S, \quad (9)$$

where e - is the base of natural logarithms, m and M - the masses of electron and ion. If $S_a < S^*$ on the anode appears bright glow of the anode plasma, which increases the effective surface area of the anode. In this case, the discharge voltage is equal to $U = U_c + U_a$, where U_a is a positive anode drop of potential, increasing with decreasing of the pressure from 20 V at 0.1 Pa to 0.5-1 kV at 0.01 Pa. Calculated and marked in Figure-2 pressure values p_0 and p^{ex} are in good agreement with the experimental data. Curves 4-6 correspond to the areas of the anode $S_a < S^* = 0.009$ m², and due to the positive anode drop the pressure of the extinction of the discharge exceeds a magnitude determined by equation (8).

It has been shown that it is possible to eliminate the high magnitude of the anode drop U_a and further reduce the gas pressure, if you place an anode in additional vacuum chamber, communicating with a hollow cathode through the hole, cross section of which is considerably smaller than the critical area of the anode S^* . In this case, inside the cathode near the hole occurs the double electrostatic layer with a surface area approximately equal to S^* . This surface plays the role of plasma anode, transparent for fast electrons oscillating inside the hollow cathode. This greatly increases the value L in equations (7) and (8) and reduces the lower boundary of p_0 of optimal pressure range and the pressure of the extinction of the discharge p^{ex} . As for low-energy plasma electrons, they are accelerated in the double layer and fly through the hole in the additional vacuum chamber. These electrons can effectively ionize the gas, maintaining the double layer anode plasma, but the gas must be submitted to the hollow cathode through the auxiliary chamber, and the pressure in it should substantially exceed the pressure in the hollow cathode.

When the pressure exceeds the upper limit of the optimum pressure range that satisfies the equation

$$\lambda_N(p) = a, \quad (10)$$

the penetration depth of fast electrons in the cathode layer and the intensity of their reproduction are reduced. Because of this cathode fall U_c increases by 100-200 V when the pressure increases to a value p^{eff} , satisfying the equation

$$\Delta_N(p) = a, \quad (11)$$

When $p > p^{eff}$, the energy of the electrons coming back from the plasma to the cathode layer is not sufficient for the formation of fast electrons in it, and the electrostatic trap has no effect on the glow discharge.

The obtained results allow to conclude that in the entire pressure range of the electrostatic trap $p^{ex} < p < p^{eff}$, where the glow discharge is very different from the discharge with a planar cathode, reproduction in the cathode layer of fast electrons, returning to it after each passage through the discharge plasma, plays a crucial role in maintaining the discharge. Contribution of the fast electrons formed in the layer to the ionization of the gas increases with decreasing of the pressure and at the pressure $p \sim p^{ex}$ becomes determining.

Similar regularities were observed in the discharge with trap volume of 0.005 m³. It was formed of a cylindrical hollow cathode with a diameter of 0.26 m and a length of 0.1 m, and a grid with high transparency, overlapping the outlet of the cathode with a diameter of 0.2 m. To prevent the withdrawal of the electrons emitted by the cathode from the trap through the holes of the grid, it is supplied with a negative voltage of 100-200 V relative



to the cathode. If the pressure was optimal for reloading accelerated ions of a gas of 0.2-0.4 Pa, the positive anode drop was less than 20 V, which does not cause serious overheating and melting of the anode. Therefore, the trap with a cathode with volume of 0.005 m³ was chosen for the generation of a plasma emitter of the experimental source of metal atoms, followed by the high-energy gas molecules.

EXPERIMENTAL STUDIES OF THE SOURCE OF SLOW METAL ATOMS AND FAST NEUTRAL GAS MOLECULES

Figure-3 shows a diagram of the experimental particle source, mounted on a vacuum chamber with a diameter of 500 mm and a length of 600 mm. Water-cooled target 1 with a diameter of 160 mm is located at the bottom of the hollow cathode 2 with a diameter of 260 mm and length 100 mm. Grid 3 overlaps the outlet of the cathode with a diameter of 200 mm. Anode 4 is made of a molybdenum rod with a diameter of 3 mm and length 160 mm. The source is equipped with power sources 5, 6 and 7. From the pre-ionizer through which gas is fed into the chamber, the cathode through the grid 3 is supplied from the chamber with weakly ionized plasma. The pressure of argon, nitrogen and their mixture and other gases is measured by a vacuum gauge with sensor BARATRON 626B.1MQF and regulated by dual-channel gas delivery system with controllers 1179G22 CR 1BK (produced by MKS Instruments Inc., USA).

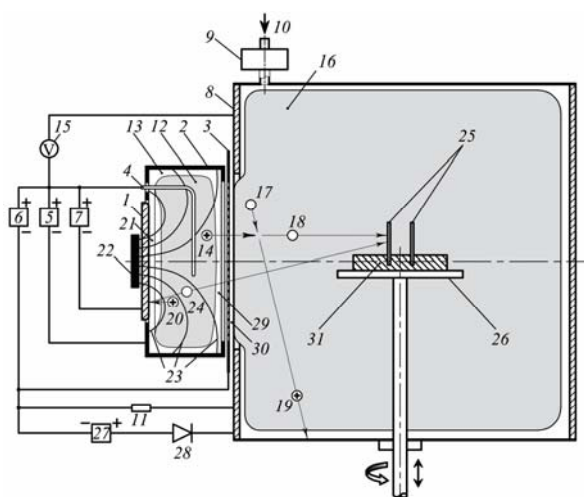


Figure-3. Scheme of the experimental installation.

Grid 3 is connected to the chamber 8 through a resistor 11 with adjustable resistance. After turning on the power sources mentioned plasma of low concentration initiates inside the hollow cathode 2 the ignition of the glow discharge and cathode is filled with discharge plasma 12, separated from the cathode surface by the layer 13. The voltage of the power source 6 is on 100 V-200 higher

than the one on the power source 5 and this prevents the electrons emitted by the cathode of flying out through the holes of the grid 3. Ions 14 are accelerated by the potential difference between the plasma emitter and the secondary plasma 16 formed in the chamber 8 during the injection of the ions into it.

As a result of charge exchange in collisions with molecules 17 ions 14 turn into fast neutral molecules 18. The current in the circuit of the chamber 8 of slow ions 19, which are formed when recharging, induces a potential drop on the resistor 11, the grid 3 becomes negative relative to the chamber 8 and prevents the penetration of electrons from the plasma 16 into the plasma emitter 12. Changing the voltage of the power source 6, it is possible to adjust the energy of the fast molecules from ~ 0.4 keV to ~ 10 keV.

When the target 1 and the cathode 2 are equipotential, the beam formed by the source of fast molecules can be used for pre-treatment of the products. At voltages of power supply 7 up to 3 kV between the anode 4 and the target 1 the ions 20 from the plasma emitter 12 are accelerated in the layer 21 and bombard the target. To reduce the number of secondary electrons emitted by the target that are entering the chamber through the grid behind the target 1 was mounted the permanent magnet 22 with the field lines intersecting both the target and the hollow cathode.

The scattered atoms 24 pass through the plasma emitter 12, grid 3 and together with accelerated ions 14 enter the chamber. The metal atoms were used for the synthesis of nitride coatings on the surface of products 25, rotating on the table 26 at a speed of 8 rpm. Properties of the synthesized coatings control the continuous bombardment of its surface by fast molecules with optimal energy.

The experiments showed that with the increase of the resistance of the resistor 11 it is impossible to reduce the plasma potential of the emitter 12, which is equal to the potential of the anode 4, below 150-200 V due to the fact that the increase in resistance is accompanied by a decrease of current in the circuit of the chamber. Therefore, to adjust the potentials of the grid and the anode was used the additional power supply 27, connected to the resistor 11 through the diode 28, the current of which through a resistor independently changed the voltage drop on it.

Figure-4 shows the radial distribution of the density of ion current j_i on the probe in the plasma 12 (Figure-3) and speed of the target v , depending on the configuration of the lines of the magnetic field with the induction in the center of the target of 35 mT.

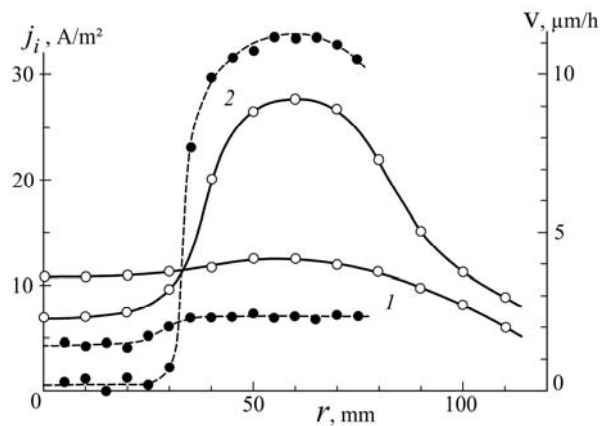
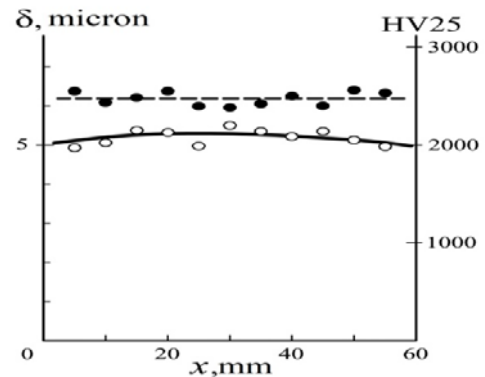


Figure-4. Dependence of the sputtering speed of the target v on the distance r to the axis of the hollow cathode (dashed curves) and the density of ion current to the probe j_i (solid curves) in discharges with magnetic induction at the edge of the target 1 mT (curves 1) and 1 mT (curves 2) at a pressure of 0.4 Pa, the current in the circuit of cathode 0.5 A, the grid voltage of 800 V and the voltage on the target 0.6 kV.

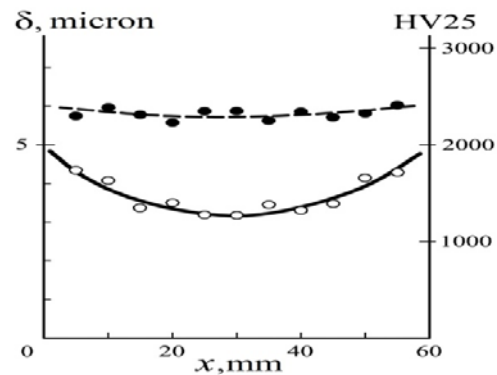
In case if induction on the border of the target $B_0 = 1$ mT heterogeneity of the sputtering speed keeps on the previous value $\pm 7\%$, but the proportion of the ion current from the plasma in the chain of the target increases from 12% to 25%. With increasing B_0 up to 6 mT, the proportion of current in the circuit of the target ions, uniformly diffusing 85% of its surface increased to 56%. When B_0 exceeds 15 mT, sputtering of the target becomes sharply inhomogeneous and differs from the sputtering in conventional magnetron discharge.

To demonstrate the capabilities of a new source of metal atoms and fast gas molecules, the coatings of titanium nitride were synthesized on two plates 25 (Figure-3) of aluminum oxide with a length of 60 mm and a height of 48 mm, installed at a distance of 25 mm from each other on the rotating Table 26. To measure the thickness of coatings, each plate was partially closed with the masks on both sides. First, the surface of the plates was activated for 10 minutes at a pressure of 0.2 PA with fast argon atoms with energy of 1 KeV. Then at a pressure $p = 0.4$ PA of a mixture of argon and nitrogen (15%), the voltage on the sputtering target $U_t = 3$ kV, the current in the circuit of the hollow cathode $I_c = 3$ A, and the density of the equivalent current of ~ 10 A/m² of argon atoms and nitrogen molecules with energy of 150 eV was synthesized for 6 hours on cover plates. After removal of the masks was measured the thickness of the coating δ . On the outer surfaces of the plates it is distributed fairly uniformly, $\delta \approx 5 \mu\text{m}$ (Figure-5a). On the inner surfaces, facing each other, the thickness decreases from $\delta \approx 5 \mu\text{m}$ at the edge of the plate to $\delta \approx 3.2 \mu\text{m}$ in the middle (Figure-5b). X-ray diffraction pattern of the inner surface (Figure-5c) does not differ from the diffraction pattern of the outer surface

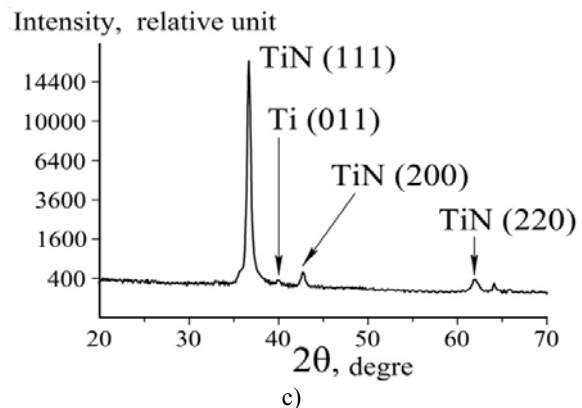
and corresponds to the TiN coatings obtained by standard magnetron technology. Microhardness $\sim 2500\text{HV}_{25}$ and the golden color of the resulting coatings are typical for the stoichiometry of the TiN.



a)



b)



c)

Figure-5. Dependence of thickness of the coating δ on the distance x to the plate edge (solid curves) and microhardness (dashed curves) on the external (a) and inner (b) surfaces, and x-ray diffraction pattern of the inner surface (c).



However, to obtain crack-resistant and super hard materials and coatings, you should replace the continuous bombardment of the synthesized coatings with ions with energy of 50-200 eV with pulsed bombardment with particles with energy of about 30 KeV. For this purpose, it is proposed to use a plasma emitter at a grounded chamber potential and get high-energy neutral molecules, giving a negative high-voltage pulses to the recharge device, consisting of two grids and a hollow cylinder between them (Figure-6). In this case the source contains anode 2, electrically connected with the working vacuum chamber 1, hollow cathode 3, emission grid 4, target 5, the power source of the discharge 6, the positive pole of which is connected to the chamber 1 and the anode 2, and the negative pole is connected to the hollow cathode 3 and the target 5. It also includes the generator of high-voltage pulses between the anode and the emissive grid 4, additional grid 8 parallel to it and the hollow electrode 9 which is electrically connected to both grids and covering the space between them. Behind a target is located a magnetic system 10 with the arched configuration of the field lines.

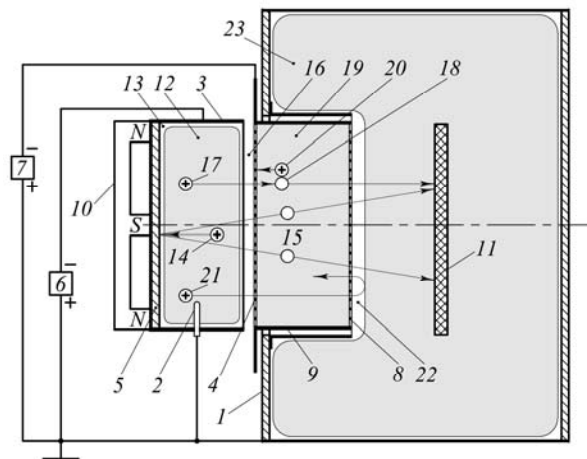


Figure-6. Source of metal atoms and pulse starts of high-energy molecules.

After the pumping of the chamber with the workpiece of a dielectric material installed inside it, the gas, for example, mixture of argon and nitrogen (15%) is let in, until the pressure rise to 0.5 Pa. After turning on the power source 6, the voltage to 500 V between the anode and a hollow cathode initiates a glow discharge and hollow cathode is filled with homogeneous discharge plasma 12, which is separated from the hollow cathode and the target layer 13 with a layer of positive charge.

Ions 14 are accelerated in the layer 13 and with the energy of hundreds of electron volts bombard and spatter the hollow cathode. Metal atoms 15, spattered by ions through the emission and additional grids with a transparency of 90% fly into the chamber and deposit on

the workpiece 11. When to the grids and to the connected with them hollow electrode is applied a negative pulse with voltage amplitude of 30 kV and a duration of 5-20 μ s between the plasma 12 and the grid 4 forms a layer 16 of a positive space charge, and its width increases to about 0.01 m. At a gas pressure of 0.5 Pa and argon ion energy of 30 KeV the length of recharge equal to 0.1 m and is 10 times greater than the width of the layer 16. Therefore, the number of ions turning into the fast molecules in the layer 16 is negligible.

If the distance between the grids is 0.1 m approximately 63% of the accelerated ions in the equipotential space between them become neutral molecules with an energy of 30 KeV. This space is filled with plasma 19, formed as a result of neutralization by electrons emitted by the grids and the hollow electrode, the space charge of the ions 20 formed when recharging. Accelerated ions 21, not turned into a neutral molecule, enter the chamber through the additional grid, slow down in the layer between it and the secondary plasma and return back. Therefore, none of the accelerated ions gets on the surface of the dielectric workpiece. The synthesized coating is bombarded only with fast neutral molecules.

DEVELOPMENT OF AN INSTALLATION FOR THE SYNTHESIS OF CRACK-RESISTANT NANOCOMPOSITE MATERIALS AND SUPERHARD COATINGS

On the basis of the obtained results was developed a source of metal atoms and beams of high-energy molecules with a rectangular (136x356 mm) target 1 (Figure-7) in an inhomogeneous magnetic field at the bottom of a rectangular hollow cathode 2 with a length of 356 mm width 136 mm and a depth of 50 mm. Using 5 ceramic insulators with 3 screens, protected against deposition of metal films, the emission grid 4 with a fixed rectangular hollow electrode 5 with a length of 580 mm, a width of 170 mm and a depth of 85 mm is fastened on the flange of the chamber. The additional grid 6 is attached to this electrode. Bushing 7 is used to feed the grids and the electrode between them with the negative high-voltage pulses.

Inhomogeneous magnetic field with the same distribution of induction between the center of the target and its border as on the round targets in the examined sources must ensure efficiency of the use of the target material higher than conventional.

As a result of recharge in the equipotential space between the grids 4 and 6, the same as in fig. 6, of the ions, accelerated by voltage pulses with amplitude of 30 kV between the plasma emitter inside the hollow cathode 2 and the grid 4 should be formed the beams of neutral molecules with an energy of 30 KeV and a duration of 5-20 μ s.

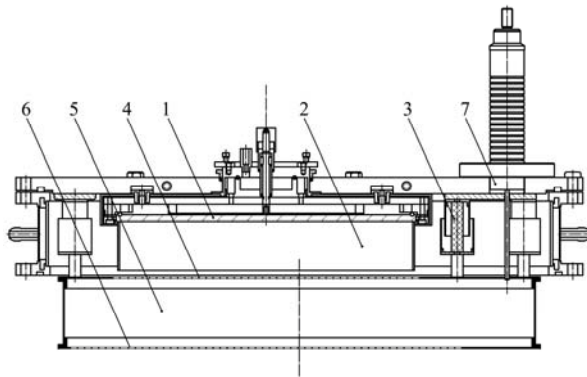


Figure-7. Scheme of the source with rectangular grids, target and a hollow cathode.

The only difference is that the devices used in the studies described above the grid and the target was round, and hollow cathode was cylindrical. Actually the form has no great importance, however long rectangular grids and targets provide higher area of uniform deposition of coatings on workpieces that are installed inside the working chamber on a system of planetary rotation compared with round targets and grids [2-4].

To study this rectangular source and development with its use a synthesis technology of crack-resistant superhard materials and deposition of coatings with high adhesion was developed and manufactured a new installation of coating deposition (Figure-8) [1-3].

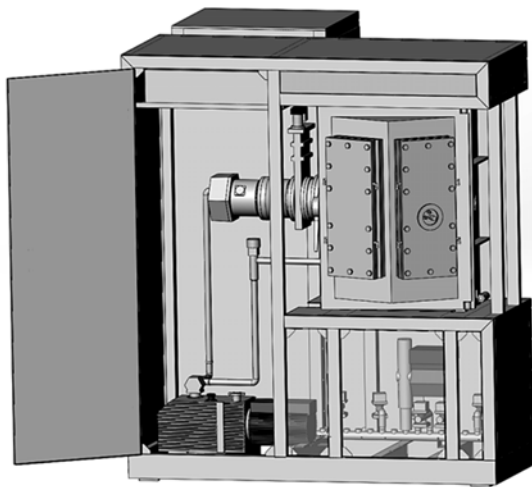


Figure-8. Scheme of installation for coating deposition, designed and manufactured to obtain a new class of heterogeneous materials and nanocomposite coatings with increased cyclic crack resistance and hardness through the use of innovative beam and plasma technologies.

The vacuum chamber of the installation is 800 mm in height and has 4 rectangular windows with a height of 600 mm and a width of 200 mm, which allows mounting the above-described source on the chamber. After the research of the characteristics of the source and development of technology of synthesis of innovative coatings certain changes in the design of installation and source can be made. A proven source design can be replicated, and the installation made for studies can be equipped with four sources, that will improve its performance 4 times.

CONCLUSIONS

In the synthesis of coatings is very important to ensure the continuity of the bombing of the coating on the entire surface of the workpiece, including its cavities. To do this, the trajectories of deposited atoms and fast molecules bombarding the synthesized coating must be the same. Otherwise the metal atoms and fast molecules will come on separate portions of the surface and deposition of the coating without the bombardment of fast particles will interchange with bombardment with high-speed particles without coating deposition, i.e. the conditions to obtain the desired coating properties will not be created. Full coincidence of the trajectories is possible only if both sources are combined in one device common to both sorts of particles emission surface.

To obtain such a device, in the present work the sputtered target is installed at the bottom of the hollow cathode opposite to the emission grid of a source of fast neutral molecules developed in MSTU "STANKIN" and it is supplied by a negative voltage up to several kilovolts relative to the cathode. The atoms of the material of the target, sputtered by ions with energies of a few KeV, through the emission grid source with opacity 90% go to the workpiece surface in the working vacuum chamber with fast molecules that will ensure the constancy of the ratio of the densities of deposited atoms and the molecules bombarding the coating on all parts of the surface of the workpiece with complex geometric shape.

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