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Deposition of wear-resistant coatings using a combined source of metal atoms and fast gas molecules

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Abstract – A source of metal atoms accompanied by fast gas atoms has been developed and studied. The fast particles are produced due to charge exchange collisions of ions accelerated by potential difference between a plasma emitter inside the source and an emissive grid. The metal atoms are produced due to sputtering a target placed at the hollow cathode bottom by ions with energy up to 3 keV from the plasma emitter. Sputtered atoms cross the emitter and the grid, together with accelerated ions enter the chamber and deposit on the products. Two different discharge modes can be used to produce the plasma emitter and bombard the growing nitride coating interruptedly by atoms and molecules with energy variable from 10 to 300 eV.

Key words: Strengthening / wear-resistance / hard coatings / gas discharge / plasma / fast molecules

1 Introduction

Ion-plasma processing is widely used in industry for strengthening the surface of various products. It can be plasma immersion ion implantation [1, 2] or deposition of wear-resistant coatings [3]. When dielectrics or semiconductors are to be coated, then broad beam sources of ions [4–6] or fast molecules [7–10] could be used to bombard the growing coating. In this case incidences to the piece surface of metal atoms taking part in the coating synthesis and fast particles bombarding the coating are defined by geometrical shape of the product and its position relative to the source of metal atoms and the source of fast molecules. When rotating products have deep cavities, then slow metal atoms and fast particles arrive to side walls of the cavities by turn, which cannot ensure high quality of synthesized coatings.

To solve the problem, it was proposed by the authors of the present paper to combine the sources of slow and fast particles in one and the same device. The present paper gives some results of such a device investigation.

2 Experimental setup

To produce the above source a target may be placed at the bottom of a hollow cathode [11] and sputtered by argon ions from the glow discharge plasma filling the cathode, the ions being accelerated by negative voltage up to 3 kV applied to the target. Such a device with 80-mm-diameter target being uniformly sputtered in magnetic field, which prevents fast electrons from leaving the hollow cathode through its broad aperture, enables at argon pressure 0.04 Pa coating deposition on substrates distant at 0.1–0.2 m from the target with the deposition rate of 5–10 μm.h^{-1} [12].

Figure 1 presents schematic of a source with 160-mm-diameter target 1 placed at the bottom of 300-mm-diameter and 100-mm-deep hollow cathode 2 and cooled with water. The 200-mm-diameter output aperture of the cathode is covered by grid 3 made of 0.8-mm-thick titanium sheet with 4.6-mm-diameter apertures the distance between the aperture centers amounting to 5 mm. Anode 4 is made of 3-mm-diameter and 60-mm-long molybdenum rod its vertical segment being distant at 60 mm from the target and the hollow cathode bottom.

The source is equipped with DC power supplies 5, 6 and 7 all three of them being simultaneously switched off when cathode spots of vacuum arc appear on target 1, cathode 2, grid 3 or chamber 8 and potential difference between anode 4 and one of these electrodes drops lower than 100 V. Voltage up to 0.7 kV of power supply 5 between anode 4 and cathode 2 and voltage up to 2 kV of power supply 6 between anode 4 and grid 3 are automatically switched on in 10 ms after the general cut-off, but voltage up to 2 kV of power supply 7 between

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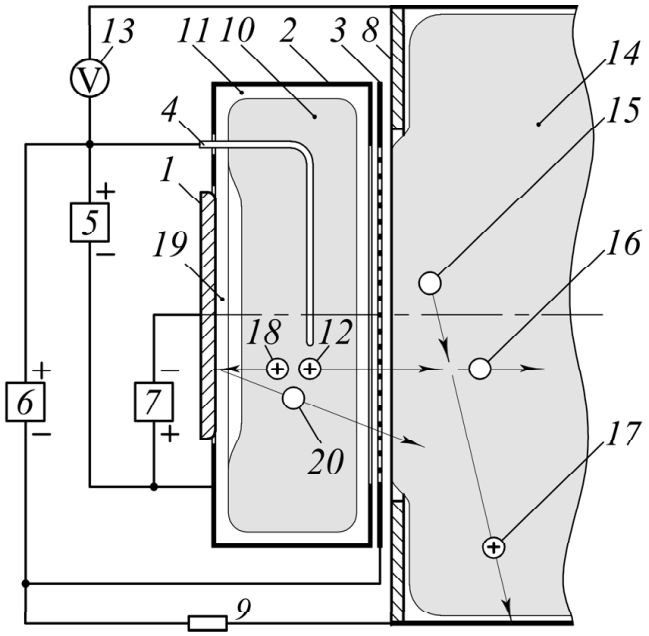

Fig. 1. Schematic of the source: 1 – target; 2 – hollow cathode; 3 – emissive grid; 4 – anode; 5, 6 and 7 – power supplies; 8 – vacuum chamber; 9 – feedback resistor; 10 – plasma emitter of ions; 11, 19 – positive space charge sheaths; 12 and 18 – ions; 13 – voltmeter; 14 – secondary plasma; 15 – gas molecule; 16 – fast molecule; 17 – slow ion; 20 – sputtered atom of the target.

cathode 2 and target 1 is switched on in 20 ms after the cutoff. Shape and position of the anode at an adequate distance from target 1 ensure contact of the anode with a weakly ionized gas from the gas pre-ionization arrangement within the 10-ms-long pause as well as during the high-current glow discharge establishing after power supplies 5 and 6 are again switched on. Chamber 8 and grid 3 are connected to each other through feedback resistor 9 its resistance R being variable from 20 Ω to 20 kΩ.

The pre-ionized gas enters chamber 8 and through grid 3 enters hollow cathode 2 of the source. When voltages ∼500 V are applied from power supplies 5 and 6, low density plasma inside hollow cathode 2 initiates ignition of the glow discharge, and cathode 2 is being filled with plasma 10 of the glow discharge with electrostatic confinement of electrons [13], which is separated from the cathode surface by space charge sheath 11. The voltage of power supply 6 is higher than the voltage of power supply 5 by 50–100 V, and ions 12 are accelerated by voltage between plasma emitter 10 and secondary plasma 14 inside chamber 8. Potential difference φ between secondary plasma 14 and chamber 8 is measured by an emissive probe.

Due to charge exchange collisions with gas molecules 15 ions 12 turn into fast neutral molecules 16. Current in the circuit of chamber 8 of slow ions 17 produced due to charge exchange collisions induces a potential drop across resistor 9 thus lowering potential of grid 3 and preventing electrons of plasma 14 from penetration into plasma emitter 10.

Fast electrons emitted by cathode 2 and produced in the sheath of positive space charge surrounding plasma emitter 10 are confined in the electrostatic trap formed by hollow cathode 2 and grid 3 the latter being negative to cathode 2 and chamber 8. At the room temperature the lower limit of the source operating pressure range is equal to $p_{\min} = 0.14/1.5$ Pa ≈ 0.1 Pa. For this reason at resistance $R > 1$ kΩ, current $I_c \sim 1$ A in the cathode circuit and the anode surface area $S_a = 0.0015$ m^2 exceeding the threshold value $S^* = 0.0012$ m^2 of the positive anode fall existence, the source characteristics at the pressure $p > 0.1$ Pa of argon or its mixture with nitrogen should only weakly depend on p.

3 Experimental results

At argon pressure 0.2 Pa and voltage between the anode and the hollow cathode $U_c = 400$ V current in the cathode circuit $I_c = 1$ A, current in the target circuit $I_t = 0.18$ A and current in the grid circuit $I_g = 0.17$ A. Current in the chamber circuit $I_{ch} = 0.08$ A through resistor 9 with resistance $R = 2$ kΩ induces voltage $U_g = 160$ V between the chamber and the grid, which cuts off the electron current from chamber 8 to emitter 10. In this case equivalent current of fast molecules 16 is equal to $I_b = \eta(I_g + I_{ch}) = 0.2$ A, where $\eta = 0.8$ is geometrical transparency of the grid.

Increase in voltage between the cathode and the target up to 2 kV leads to a sixfold increase in energy of ions 18 accelerated in sheath 19 as well as in sputtering rate of target 1. Through apertures of grid 3 sputtered atoms 20 enter the chamber and deposit on pieces placed therein. At the same current $I_c = 1$ A in the cathode circuit the current in the target circuit rises up to $I_t = 0.3$ A due to secondary electron emission. But even at the ion energy of 2.4 keV the coating deposition rate on substrates distant at ∼0.1 m from grid 3 does not exceed 1 μm.h^{-1}.

At $R = 2$ kΩ, argon pressure $p = 0.4$ Pa and cathode fall $U_c = 700$ V current in the cathode circuit rises up to $I_c = 5$ A and current in the target circuit rises up to $I_t = 1.6$ A the ion current component amounting to $I_i \approx 1$ A. Ions 12 accelerated by potential difference between emitter 10 and plasma 14 turn in fast molecules 16 their equivalent current amounting to $I_b \sim 1$ A. However in spite of the above estimation of the lower operating pressure $p_{\min} \approx 0.1$ Pa of the source both currents drop down by an order of magnitude when pressure decreases from 0.4 to 0.1 Pa. This discrepancy may be explained by threefold increase in temperature of the hollow cathode and the gas therein, which leads to a correspondent decrease in the gas molecule density and an increase in the lower operating pressure up to $p_{\min} \sim 0.3$ Pa.

In the above regime of the source at a fixed equivalent current I_b of fast molecules it is possible to increase resistance R of resistor 9 up to 20 kΩ and to reduce the molecule energy from hundreds of elecronvolts down to zero due to increase in current of an additional DC power supply in the resistor circuit up to 20–50 mA. At $p = 0.4$ Pa and equivalent current $I_b \sim 1$ A of 50–200-eV

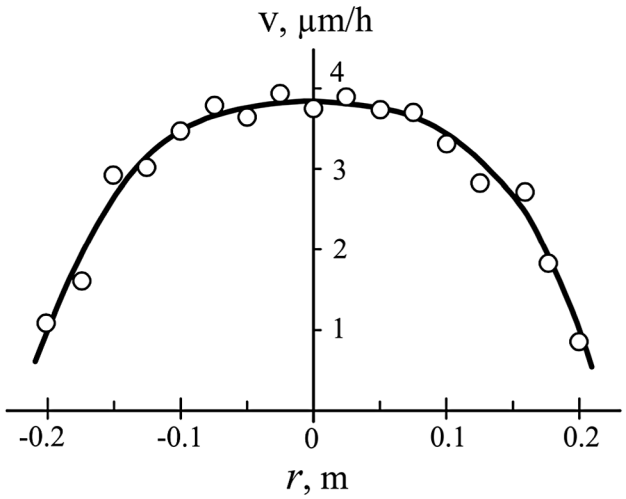

Fig. 2. Radial distribution of the titanium nitride deposition rate v on a titanium substrate distant from the emissive grid at 0.1 m at a voltage between the anode and the chamber $U = 100$ V, voltage between the anode and the cathode $U_c = 0.7$ kV, voltage between the cathode and the target 2 kV, current in the target circuit $I_t = 1.6$ A and pressure $p = 0.4$ Pa of argon (85%) mixed with nitrogen (15%).

molecules, which assist deposition of sputtered atoms, the power of ions sputtering the target amounts to ∼2.7 kW.

The angle β of fast molecules dispersion in the chamber was measured at a distance 0.1 m from the grid. Measurement results showed that β grows up to ∼45° when the molecule energy diminished to ∼100 eV. In this case angular distributions of fast molecules and sputtered atoms of the target are about the same, which enables an uninterrupted bombardment by the fast molecules of coatings being synthesized on the whole surface of planetary rotating pieces their slots included.

When argon is mixed with about 15% of nitrogen, the above source enables deposition of TiN, AlN and TiAlN coatings. At the above current $I_t = 1.6$ A in the target circuit and gas pressure 0.4 Pa the deposition rate of titanium nitride coatings with microhardness ∼2300 HV50 on tungsten carbide substrates distant from the emissive grid center at 0.1 m amounts to $v \sim 4$ μm.h^{-1}. Figure 2 presents dependence on distance r from the source axis of the coating deposition rate v on a titanium strip distanced from the grid at 0.1 m, which was measured using the method described in [12]. In the central zone with radius $r < 0.1$ m the deposition rate amounts to $v \approx 3.75 \pm 0.25$ μm.h^{-1} and at $r > 0.1$ m it rapidly diminishes to $v \sim 1$ μm.h^{-1} at $r = 0.2$ m.

It was found that surface of the flat 160-mm-diameter target is being sputtered quite uniformly. A portion of sputtered titanium is deposited on the inner surface of the hollow cathode and for this reason the gas discharge characteristics correspond to a cathode made of titanium. After the titanium target is replaced with an aluminum target and the latter is sputtered during 10 min, the hollow cathode may be considered as made of aluminum. A black coating of aluminum nitride with a very poor electrical conductivity is deposited in this case.

For deposition of TiAlN coatings two targets are simultaneously used: an inner 90-mm-diameter aluminum disc and an annular target with internal diameter 90 mm and external diameter 160 mm. When the inner disc is replaced with a conical target, the target directs electrons emitted due to bombardment of its surface by 2.7-keV ions to the inner surface of the hollow cathode. As a result the fast secondary electrons do not bombard the pieces inside the chamber and, secondly, they contribute to a decrease in the cathode fall U_c, operating gas pressure and an increase in the coating deposition rate, because the electron bombardment of the cathode raises its electron emission current.

After power supply 7 is switched off and voltage of power supply 6 is increased up to 1–2 kV, it is possible to bombard the pieces with fast argon atoms thus heating them and activating their surface. When argon is replaced with nitrogen the source enables the gas ionization in the processing chamber by 2-keV nitrogen molecules, and a non-self-sustained glow discharge between the chamber and an additional anode immersed in the reactive nitrogen plasma produced by the molecule beam may raise the plasma density by an order of magnitude and reduce its nonuniformity down to ±10% [10].

When tools made of high speed steel (HSS) are being processed, the beam power is quite sufficient for maintenance due to bombardment by the fast molecules of the tools temperature 500 °C, which is quite enough for an effective thermodiffusion of nitrogen into the HSS. In this case the tools immersed in the plasma may be isolated from the chamber, which eliminates sputtering by ions extracted from the plasma. Usually the ion current density distribution near the cutting edge is highly nonuniform and it results in an intensive sputtering of the edge and a substantial growth of its radius that is in the tool blunting. But when the tool is heated by fast molecules its surface is sputtered quite uniformly and for this reason the cutting edge radius does not rise but on the contrary diminishes.

Deposition of 3–5-μm-thick wear-resistant coatings with microhardness ∼2500 HV50 and higher on a preliminary hardened by means of nitriding HSS surface with microhardness 1350 HV50 gradually diminishing to 900 HV50 at the depth of 50–100 μm prevents a brittle rupture of the deposited coatings caused by elastic and plastic deformations of the tool bulk.

In order to increase the current I_i of ions sputtering the target at the resistance $R > 1$ kΩ of feedback resistor 9 (Fig. 1) it is needed to raise the cathode fall of potential U_c and the gas pressure p. But at $R < 1$ kΩ an appreciable growth of the current I_i may be reached without increase in the cathode fall U_c. Figure 3 presents dependencies on the argon pressure p of the currents in circuits of the cathode I_c (curves 1), the chamber I_{ch} (2) and the target I_t (3) as well as potential φ of the secondary plasma relative to the chamber (4) at a voltage between the cathode and

the target amounting 2 kV and voltages of power supplies 5 and 6 equal to 400 V.

At $p = 0.2$ Pa reduction of R from 2 kΩ to 200 Ω (dashed curves) leads to increase in the plasma potential from $\varphi = 4$ V to 16 V and growth of currents in the circuits of the cathode from $I_c = 1$ A to 1.4 A, the target from $I_t = 0.3$ A to 0.45 A and the chamber from $I_{ch} = 0.08$ A to 1 A. At $R = 200$ Ω all mentioned values grow with argon pressure rising up to $p = 0.4$ Pa to $\varphi = 24$ V, $I_c = 2.2$ A, $I_t = 0.8$ A and $I_{ch} = 1.5$ A. Potential difference between the plasma inside the chamber and the grid amounts to $R \times I_{ch} + \varphi = 324$ V, which is 2 times higher than at $R = 2$ kΩ. In spite of increase in retarding potential of the grid, the ratio of the current in the chamber circuit to the current in the cathode circuit amounts to $I_{ch}/I_c = 0.7$, which is by an order of magnitude higher than at $R = 2$ kΩ. It may be explained by a substantial rise of electron current from the chamber into the source and an equal surplus of the current in the chamber circuit of ions from the secondary plasma.

When at $p = 0.2$ Pa the resistance is diminished to $R = 20$ Ω, current in the cathode circuit rises to $I_c = 2$ A, current in the target circuit – to $I_t = 1$ A, in the chamber circuit – to $I_{ch} = 1.5$ A and potential of the plasma inside the chamber grows to $\varphi = 50$ V. At $R = 20$ Ω these values grow to $I_c = 8$ A, $I_t = 4.5$ A, $I_{ch} = 6.5$ A and $\varphi = 200$ V with the argon pressure increasing up to $p = 0.4$ Pa. In the latter case potential difference between the secondary plasma and the grid rises to 330 V, but the ratio of the current in the chamber circuit to the current in the cathode circuit $I_{ch}/I_c = 0.8$ is a little higher than at $R = 200$ Ω. It means that the current of electrons injected from the chamber into the source and ratio of this current to the current of ions from its plasma emitter into the chamber keep on growing.

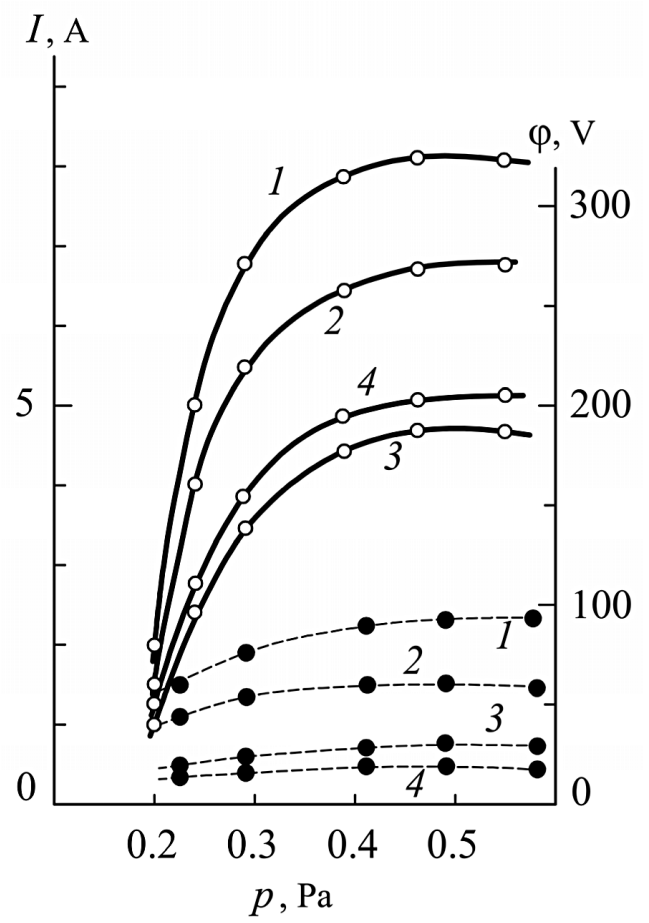

Fig. 3. Dependence on argon pressure p of currents in the circuits of the cathode I_c, the chamber I_{ch}, the target I_t and potential φ of the secondary plasma inside the chamber at a voltage between the cathode and the target 2 kV, voltage between the anode and the cathode $U_c = 400$ V and resistance of the resistor $R = 200$ Ω (dashed curves) and $R = 20$ Ω (full curves).

4 Discussions

The above results reveal two different regimes of the glow discharge, which produces the plasma emitter of the source. For the investigated experimental model the regime depends on resistance R of the resistor connected between the emissive grid of the source and the chamber.

In the first regime with resistance $R > 1$ kΩ the current of electrons from the chamber into the source is totally cut off. The cathode fall U_c of the glow discharge depends in this case on coefficient γ_i of ion-induced electron emission on the cathode and value of the current I_c in its circuit.

The upper limit $p_{max} \sim 1$ Pa of the operating pressure range exceeds by an order of magnitude the lower limit p_{min}, which according to Equation (1) at the room temperature of argon, the cathode fall $U_c = 400$ V and the electrostatic trap width $a = 0.12$ m of the source is equal to 0.1 Pa. The pressure $p \sim 0.1$ Pa ensures an adequate transportation into the chamber through the emissive grid distant from the target at 0.1 m of sputtered metal atoms their free pass length amounting to ∼0.07 m, as well as transformation into neutral particles of 87% argon ions with energy of ∼100 eV and charge exchange length of ∼0.1 m on their 0.2-m-long way from the grid to the substrate.

However the emissive grid and the hollow cathode of the above experimental model are not cooled with water and at the cathode fall $U_c = 700$ V and current $I_c = 5$ A they are heated up to 600–700 °C. The gas inside the cathode is heated up to the same temperature, its density is three times lower than in the processing vacuum chamber, which is cooled with water, and the lower operating pressure of the source rises up to $p_{min} = 0.3$ Pa. At this pressure value efficiency of the sputtered atoms transportation from the target to the grid is not reduced, the charge exchange length of ions in the chamber falls to ∼0.03 m, and percentage of ions in the flow of fast gas atoms to the surface distant from the grid at 0.2 m does not exceed 1%.

In the first regime the source is able to produce a broad beam of fast argon atoms with energy of 2 keV and equivalent current of 1 A for heating and activation of

the pieces, for instance, tools made of high-speed steel. Afterwards it may produce a broad beam of fast nitrogen molecules for production in the chamber of reactive nitrogen plasma and maintenance of the tools temperature ∼500 °C, which is adequate for an effective nitriding.

After a 50-μm-thick surface layer is strengthened due to nitriding without blunting of the tools cutting edges [14] the chamber is again filled with argon and a high negative voltage is applied to target 1 being sputtered by argon ions 18 from plasma emitter 20 (Fig. 1). Sputtered atoms pass through the grid into the chamber and deposit on the tool surface. Increase in resistance R of resistor 9 between the chamber and the grid and the use of an additional power supply connected to the resistor make it possible to increase voltage U_g between the chamber and the grid at a fixed equivalent current of fast gas atoms bombarding the coating thus decreasing energy of the fast atoms to any value ranging from zero to several hundreds of electronvolts. When about 15% of nitrogen is added to argon the source enables synthesis on strengthened due to nitriding surface of superhard wear-resistant coatings with deposition rate up to 4 μm.h^{-1}.

In the second regime electrons from the chamber are injected into the source. Those electrons enter the hollow cathode of the source through the grid from external plasma. In the above source of metal atoms the energy of electrons injected into the plasma emitter is less than the energy eU_c. For this reason injected electrons cannot bombard the cathode as in the sources of fast molecules with energy exceeding 1 keV, where electron bombardment of the cathode results in reduction of the cathode fall U_c from ∼1000 V down to ∼100 V. In the present source the growth of currents in the circuits of its target I_t and cathode I_c with decreasing resistance R of the feedback resistor (Fig. 3) is the result of gas ionization inside the cathode directly by injected electrons.

At the transparency $\eta = 0.8$ and the surface area ∼0.03 m^2 of the grid the total area of its apertures amounts to ∼0.024 m^2, which exceeds 16 times the anode surface area $S_a = 0.0015$ m^2. However the area of each aperture exceeds tens times the effective area of its central zone, through which electrons penetrate into the electrostatic trap of the discharge and then may leave the trap provided they have not lost a part of their energy before leaving. For this reason equal to the anode area S_a output aperture S_o of the electrostatic trap grows due to electron injection only negligibly.

As the energy of injected electrons is less than the energy of emitted by the cathode electrons, which spend all their energy on ionization and excitation of the gas inside the trap, the injected electrons also spend all their energy there. However when the current of injected electrons exceeds several times the electron emission current of the cathode, they substantially contribute to the gas ionization. It is possible to speak in the present case about a non-self-sustained glow discharge sustained by electrons injected into its electrostatic trap.

When electrons entering the source leave the secondary plasma filling the chamber, the plasma potential rises up to $\varphi \sim 100-200$ V (Fig. 3). Electrons accelerated in the sheath between the plasma and the chamber to the energy of $e\varphi$ oscillate therein and spend all this energy on the gas ionization before they come to a virtual anode composed of central zones of the grid apertures, through which electrons penetrate into the source.

As a result the density of the secondary plasma inside the chamber rises thus enabling a further increase in current of electrons injected into the source. When this current grows up, the plasma density in the hollow cathode also rises and at the current ∼8 A in the cathode circuit the heating power 3.2 kW is comparable with the power heating the cathode in the first regime at $I_c = 5$ A and $U_c = 700$ V. For this reason the lower limit of the operating pressure in both cases rises up to $p_{min} = 0.3-0.4$ Pa due to the heating.

This is confirmed by the curves in Figure 3, which demonstrate a weak dependence of currents in the circuits of the target and the cathode on the argon pressure p at $R = 200$ Ω and $p > 0.3$ Pa (dashed curves) as well as at $R = 20$ Ω and $p > 0.4$ Pa (full curves). When at $p = 0.4$ Pa and fixed cathode fall $U_c = 400$ V resistance R is reduced from 2 kΩ to 200 Ω and afterwards to 20 Ω, current in the chamber circuit rises from 0.08 A to 1.5 A and afterwards to 6.5 A.

At $R = 2$ kΩ the current of injected electrons is practically equal to zero and their energy amounts to 236 eV. When R is reduced to 200 Ω, the energy drops to 75 eV and at $R = 20$ Ω it amounts to about the same value of $\varepsilon = 400-20 \times 6.5-200 = 70$ eV.

At $R = 200$ Ω current of injected electron $I_e \sim I_{ch} = 1.5$ A gives rise to an increment of the ion current from the plasma emitter amounting to 2.8 A. At $R = 20$ Ω the current $I_e \sim I_{ch} = 6.5$ A of those electrons gives rise to an increment of the ion current from the emitter amounting to 11 A. In both cases the estimates of the ion current increments are roughly equal to the sum of currents in the circuits of the cathode and the target. This reveals a substantial contribution of injected electrons into the gas ionization inside the hollow cathode.

In the second regime of the metal vapor source the energy ε of fast molecules assisting the vapor deposition is exactly equal to the energy of injected electrons. This regime does not allow an independent regulation of the coating deposition rate and the energy ε of fast particles bombarding the coating. At the current 0.5–5 A in the circuit of the target being sputtered by ions their energy does not practically change and amounts to 70–75 eV. Nevertheless this value is optimal for synthesis of many superhard coatings. For this reason the second regime with a higher current of ions sputtering the target and a lower cathode fall of the discharge is preferable at the stage of coating deposition.

5 Conclusions

Experimental model investigated in the present work is in fact a universal source enabling successive

execution of all processing stages needed for synthesis of wear-resistant coatings: the piece heating and surface activating, strengthening of a 50-μm-thick surface layer and synthesis on the strengthened surface of a 3–5-μm-thick superhard coating.

The source of metal vapor and fast gas atoms with an ion-sputtered target placed at the hollow cathode bottom ensures an uninterrupted bombardment by fast gas atoms or molecules of coatings being synthesized on planetary rotating inside the processing vacuum chamber pieces with slots. Applied to the target high voltage raises the sputtering rate manifold in comparison with a magnetron target at equal currents in their circuits. High uniformity of the ion current density from the plasma emitter ensures a uniform sputtering of the target surface.

Two regimes of the source are available: with injection into the source of electrons from the processing vacuum chamber and without injection. The first ensures current of ions sputtering the target up to several amperes and energy amounting to ∼75 eV of gas atoms and molecules, which uninterruptedly bombard the coating being synthesized. In the other regime the current of sputtering ions only slightly exceeds 1 A, but the energy of fast gas atoms and molecules may be independently regulated from zero to several hundreds elecronvolts. In order to reduce the operating gas pressure to the optimal value of 0.1 Pa it is recommended to cool with water the hollow cathode and the emissive grid of the source in both regimes.

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