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High-frequency plasma at atmospheric pressure as a means of deposition of thin films

The main units of the industrial high-frequency generator have been modernized. The design and technical coordination of the plasma torch impedance was developed in order to obtain high-frequency plasma with capacitive coupling for the transmission of electrical energy at atmospheric pressure. The data on the main elements of the structures of adapted units are given, their functional purpose is indicated. Special attention is paid to the principles of operation of the modified units of equipment and their functional relationship when controlling the process of synthesis of films for nanotechnology.

The reasons why nanotechnology is not widespread on the existing high-frequency capacitive plasmatrons are analyzed. New effects of high-frequency plasma and ways to improve the plasmatron for deposition of thin films were discovered. A theoretical justification for the effect of working substance transfer in a plasma channel (cord) of a high-frequency capacitive discharge is found. The possibility of ignition of a single-electrode high-frequency flare discharge from a plasma of a high-frequency capacitive discharge is experimentally proved. An explanation of the structure of high-frequency flare discharge under these conditions is given. The possibility of using the near-electrode spot of the torch for the vapor concentration of the working substance and the deposition of the film is revealed.

The main regularities for the synthesis of nanofilms on the example of zinc and aluminum oxides are investigated. Pyrolysis is chosen among plasma chemical reactions to prepare a pair of working substances. Plasma luminescence of high-frequency capacitive discharge is studied for the purpose of effective plasmochemical reactions in the channel of argon plasma discharge and steam supply of the working substance to the substrate. Thin layers of Al_2O_3 and ZnO are synthesized by a new method using high-frequency argon plasma at atmospheric pressure.

Keywords: *high-frequency capacitive plasmatron; high-frequency flare discharge; synthesis of nanofilms; near-electrode spot of the torch.*

Analysis of research and publications. High-frequency plasma torches are widely used in modern engineering, technology and scientific research. The presence of a high enthalpy of the plasma of a high-frequency gas discharge at atmospheric pressure makes it possible to use it in plasma chemical reactors. A special role is played by high-frequency capacitive plasmatrons. The power supply is connected to the plasma through an electrical capacitance between the electrodes and the plasma channel, which has no direct contact with either the electrodes or the walls of the discharge chamber, which ensures the purity of the plasma. The characteristics of the plasma differ significantly from induction plasma, although the frequency of the supply current can be the same (5–50 MHZ). For high-frequency capacitive plasmatrons, this applies to the current strength of several amperes (1–10 A), the total voltage drop per unit length of the plasma channel of 20–200 V/cm and its diameter up to 1 cm [1]. Therefore, in plasmatrons, on the basis of high-frequency capacitive discharge, high power is realized at sufficiently weak currents (up to 10 A). Plasma is uneven under such conditions, even in molecular gases: the temperature of the electronic component significantly exceeds the temperature of the atom-ion gas. This plays an important role in the selection of plasma chemical reactions that can be efficiently carried out in such a plasma. High-frequency plasma reactors are used for the synthesis of various gases (for example, nitrogen monoxide in atmospheric nitrogen fixation technology), pyrolysis of complex substances or oxidation in the processing technology of various substances, including toxic and radioactive. This use of plasmatrons is associated with a high efficiency coefficient of the input of electrical energy into the plasma, which reaches 95 %.

A significant advantage of the high-frequency capacitive plasmatron is the low level of radiation losses. For example, in comparison with a high-frequency induction plasmatron, the level of radiation losses decreases from 10 to 1 % at the same mid-mass temperature of the oxygen plasma jet [2]. Through the advantages of a high-frequency capacitive plasmatron, it is used to produce nanodispersed powders by plasma chemical synthesis [3–5]. Nanopowders in high-frequency capacitive plasma are also obtained in plasma chemical reactions from water-salt solutions. In this case, the advantages include: single-stage and high speed of the process, homogeneous phase distribution with a given stoichiometric composition, the ability to actively influence the size and morphology of particles, compactness of technological equipment, low cost [6].

A sub-type of high-frequency discharges of the capacitive type is a single-electrode high-frequency discharge, or so-called high-frequency flare discharge. This discharge was discovered by S.I. Zilitinkevych in 1928, and it was one of the first of capacitive discharges, [7]. The first attempt to make this type of discharge the basis of the plasmatron was in 1960 by Bamberg E.A. and Dresvin S.V. Further studies of high-frequency flare discharge were carried out in connection with the solution of applied problems [8]. Plasmatrons, based on high-frequency flare discharge, are successfully used for surface treatment. Plasma processing is widely used in film electronics. As an example of high-frequency plasma at atmospheric pressure, we can study the technology of adjusting the resistors resistance used in microassemblies with irregular structure, including composite ones [9].

One of the first applications of high-frequency capacitive plasmatrons operating at atmospheric pressure was the synthesis of coatings. The property of the discharge plasma blowing into the cord, which has a high stability at high speeds of the plasma-forming gas blowing, makes it possible to evaporate refractory powders in the plasma without impurities. This process is carried out effectively in a quartz tube, where the discharge channel (cord) passes along the axis, and from the heat transfer on the wall, the plasma is isolated by a separate gas supply. Along the plasma cord, a substrate (product) is placed on which the coating is settled down [10]. Such coatings are used as protective and wear-resistant, for example, in mechanical engineering. Their thickness depended on specific tasks and constituted (0,1–1,5) mm.

Problem statement. As seen above, the use of high-frequency capacitive plasmatrons operating at atmospheric pressure is extensive, also for the synthesis of coatings. But there is no data on their use for coatings in nanotechnology, that is, to create such coatings that are used in electronics, or precision for mechanical engineering. Thus, the purpose of the studies described in this paper was to identify the possibility of coating, which differ from the above, in addition to thickness and quality.

When forming thin films by Physical vapor deposition (PVD), it is important to control the feed rate and the transverse gradient of the vapor density in the settled-down stream. Since these conditions can be met in the absence of turbulent flows, the PVD method is used mainly at reduced pressure or in vacuum. In engineering and technology, high-power high-frequency turbulent flow plasmatrons are mainly used, for which it is difficult to maintain the limitations of the PVD method. But the use of argon and helium as plasma-forming gases makes it possible to regulate the temperature of the discharge channel in a wide range [11]. Thus, to stabilize the temperature fields, eliminate vorticity and mixing of layers, that is, to control the modes of plasma flows. This is a prerequisite for the synthesis of coatings of a significant number of different substances.

Description of the unit. In our studies, we used a classical high-frequency plasmatron, the principle of operation of which is based on the capacitive connection of the power supply with the leading zone of the so-called capacitive electrode-free discharge. Designs of high-frequency capacitive plasmatron share some similarities, that is the transfer of energy to the discharge zone occurred by means of a capacitive current. They are distinguished primarily by the shape and number of electrodes [12]. We used a linear plasmatron (Fig. 1). The high-frequency discharge 6 was maintained inside a quartz tube 4 with an internal diameter of 15 mm, which served as a discharge chamber, using copper coaxial electrodes 3 and 5. Electrode height h = 60 mm. The distance between them L could be increased to 100 mm. Plasma-forming gas (argon) at atmospheric pressure was fed into the discharge chamber (quartz tube) as shown by the arrow, that is, from the bottom up. Argon in our installation played the role of transporting gas. In the quartz tube 4, it was fed through an aerosol generator, which led to the transfer of the working substance to the discharge chamber in the form of an aerosol of an aqueous solution. The aerosol generator worked on the principle of spraying by acoustic vibrations. The resonant frequency of the ultrasonic emitter was 1.7 MHz.

The regulated voltage to the electrodes of the discharge chamber was supplied from the high frequency generator 9, which is developed on the basis of the generator triode GU58B. The operating frequency of the generator varied from 5 MHz to 15 MHz with a maximum oscillatory power of 2.5 kW. The voltage U_c, which was measured by the voltmeter shown in Fig. 1, varied from 1 kv to 7kv by setting the output oscillating circuit. Since this circuit included a plasmatron, after changing the geometry of the plasmatron, the capacitance was corrected by the capacitor C, indicated in Fig. 1. The role of capacitor C was also therein after the spark-discharge of tungsten rod electrode, which is inserted into the area of the high-frequency electrode 5, U_c changes had the desired value. Capacitor C made it possible to either increase or decrease the U_c, depending on the needs of the technological process.

To control the film deposition process, a thermocouple unit 7 of the set for measuring the exhaust gas temperature of internal combustion engines TCD was used, which is labeled as a thermoelectric converter of the type TKH-400U. This meter has a chromel-copel thermocouple embedded in a 1 stainless non-magnetic steel frame.

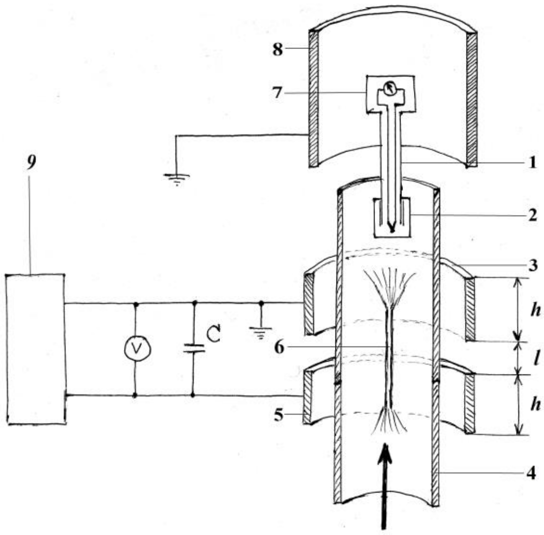

Fig. 1. Diagram of the main units of the installation:
1 – The tubular steel frame of the thermoelectric converter; 2 – Replaceable cap of thermoelectric converter; 3 – The upper grounded electrode; 4 – Quartz tube; 5 – High frequency electrode; 6. – High-frequency capacitive discharge channel (cord); 7 – Thermoelectric converter TKH-400U; 8 – Grounded electrode to maintain high frequency flare discharge; 9 – High-frequency generator

The temperature measurement took place on the surface of the replaceable cap 2, directed to plasma of high-frequency discharge. The cap 2 plays the role of an electrode on which a single-electrode high-frequency flare discharge is ignited. To reduce the parasitic capacitive connections of the lead wires to the ground measuring millivoltmeter was replaced by a miniature and fixed directly on the body TKH-400U. By moving the electrode 8, a predetermined capacitive coupling of the cap-electrode 2 with the ground can be established.

The main results of the research. The experiments, which were carried out under different conditions, showed the possibility of synthesis of thin films from substances introduced into high-frequency plasma at atmospheric pressure. First of all, this possibility is provided by laminar plasma flow in the channel of high-frequency capacitive discharge. When conditions were created under which the Reynolds number was Re ~ 100, the formation of a zinc oxide coating on the inner surface of the quartz tube 4 was observed, as seen in Fig. 2. On the surface, what is below the line of abrupt completion of the coating, i.e. on the level of existence capacitive discharge, the deposition of any substance is not recorded. This fact shows the prerequisites for the effective supply of the working substance to the substrate in the form of steam. Quartz tube 4 can end at the level of the upper end of the electrode 3. Above the electrode, a substrate can be placed where the film will be synthesized.

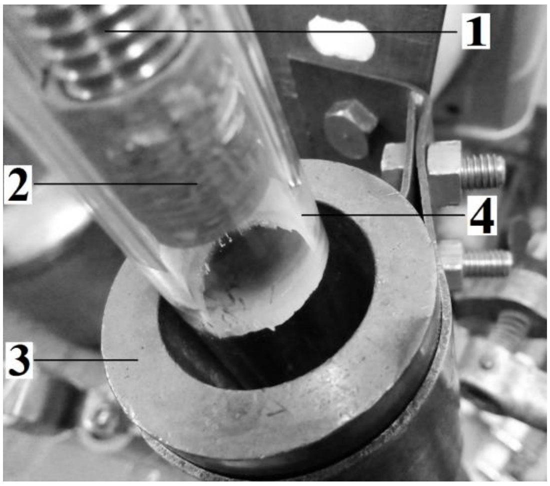

Fig. 2. Zinc oxide coatings on the inner surface of the quartz tube:
1 – The tubular steel frame of the thermoelectric converter; 2 – Replaceable cap of thermoelectric converter; 3 – The upper grounded electrode; 4 – Quartz tube

The temperature of the argon plasma discharge channel, that is, the gas temperature was ~ 900K [13]. At such a temperature and argon feed rate up to 0.015 l/s, an aerosol with an average droplet diameter of 14 mm already a few centimeters from the beginning of the discharge channel is converted into steam, in which the initial composition of the substances that reacted must contain zinc oxide. If there was mixing in the plasma stream, the zinc oxide coating would be fixed on the inner surface of the quartz tube along the high-frequency discharge.

The plasma formation of a high-frequency capacitive discharge at both ends has areas that give a diffuse glow. The main part of the discharge is the cord, that is, the plasma obtained as a result of compression by its magnetic fields of the conduction current.

The plasma cord, heated to a high temperature, is the main part of the plasma flow of argon gas. The gas that is fed into the quartz tube is drawn into its inner part near the axis and moves vertically upwards. This is because there is no mixing, that is, laminar flows provide for the absence of a velocity component that is perpendicular to the flow, and therefore the substance cannot move from one layer to another. The flow heated in the cord is lighter than that near the walls of the quartz tube. The substance in the cord would move vertically upwards even without the impulse that brings the gas supplied from below into the quartz tube, as a result of the phenomenon of convection. Since the gas near the walls of the tube is relatively cold along the entire gas discharge and has a temperature close to the temperature of the walls, there is no convection in this part. From the above, it can be concluded that in the case of supplying gas to the quartz tube from below, it will compress to the axis and move upwards, heating up in an electric discharge.

Cord 2 ends where the plasma has an expansion (Fig. 3). They are observed near the upper end of the upper electrode 3 and the lower end of the lower electrode. In these parts of the plasma, there are plasma instabilities leading to turbulent flows, which can be seen by the movement of the glowing plasma. That is why zinc oxide is deposited in the direction of flow on the inner surface of the quartz tube above the upper end (Fig. 2). In this case, the flow is mixing layers even if there is no obstacle to the flow in the form of electrode 2. In Fig. 2 electrode 2 is inserted into the middle of the quartz tube only to demonstrate its placement in the formation of the torch.

During our numerous experiments, there were cases when external electrodes 3 and 5 were used (Fig. 1), which had significant irregularities on the inner surface, that is, on the surface that is turned to the quartz tube 4. These irregularities provoked the appearance of plasma instabilities, and then the formation of zinc oxide coatings on the inner wall of the quartz tube was observed.

Introduction of the thermocouple unit 7 (Fig. 1) in the discharge region, that is, the approximation of the cap-electrode 2 to the expanded plasma at the upper end of the electrode 3, made it possible to form a torch from the plasma of the capacitive discharge, so an additional high-frequency flare discharge was formed. The appearance of a new discharge can be explained on the model of a single-electrode discharge [2]. In this discharge, the frame of the thermocouple unit has a capacitive connection to the ground, and the cord of the high-frequency capacitive discharge is connected to the high-frequency electrode 5 (Fig. 1).

In the presence of direct ohmic contact between the earth and the frame of the thermocouple unit, a discharge with a high temperature plasma is formed due to the release of a large thermal power, which reached 1000 watts. In the main experiments, the thermal power of the R_f introduced into the plasma of the torch was regulated by the capacitive coupling of the thermocouple block frame with the ground and varied from 30 W to 150 W. The total thermal power of P_1 was determined by multiplying the values of the voltage at the anode of the generator lamp and the anode current just before the ignition of the torch.

$R_f = P_2 – P_1$, where P_2 is the total thermal power after ignition of the torch.

Calculations of the thermal power that could be released at the electrode 8 (Fig. 1), conducted in accordance with [1]. They showed a value that can be neglected in our experiments. Thus, the R_f can be considered the power of a single-electrode high-frequency discharge.

The torch consists of a near-electrode spot, a thin channel and a diffuse shell [15]. Fig. 4 shows a bright near-electrode spot 1 which is located on the end surface of the metal electrode 2. The photo demonstrates a part of the electrode 2 in the form of a cap, wound on the threaded part of the thermocouple unit. Considering the spot 1 in space, it can be said that it is elongated along the discharge axis. Fig. 4 also points at the diffuse shell of the discharge, which has a wide base 5, glowing against the interior of the electrode 3, and tapers to a near-electrode spot 1. As stated above, the diffuse shell is formed from the plasma of the main high-frequency capacitive discharge when the metal electrode approaches the «tips» of the plasma 1 (Fig. 3) (plasma extensions with acuities that are directed upwards). The plasma is thus collected to a near-electrode spot, which explains the shape of the diffuse shell. The absence of an explicit channel in the high-frequency discharge torch is explained by the existence of a «temperature dip» in the longitudinal profile at low capacities of a single-electrode high-frequency discharge. A description of the structure of a single-electrode high-frequency discharge and thermal fields of plasma under these conditions is given by A.S. Tobolkin [16].

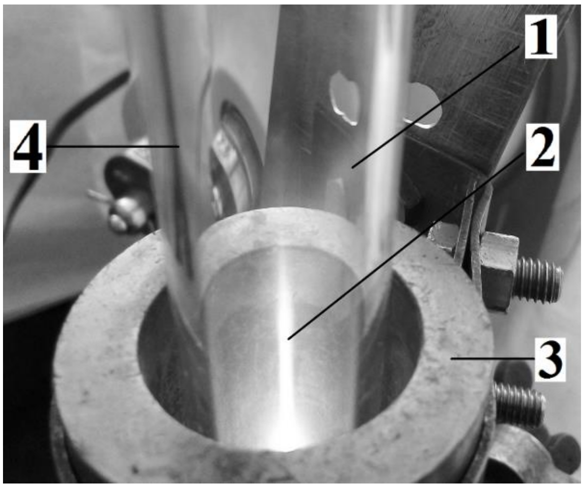

Fig. 3. Plasma expansion at the end of the channel of high-frequency capacitive discharge:
1 – One of the parts of the plasma that glows as acuity, («plasma tip»); 2 – High-frequency capacitive discharge channel (cord); 3 – The upper grounded electrode; 4 – Quartz tube

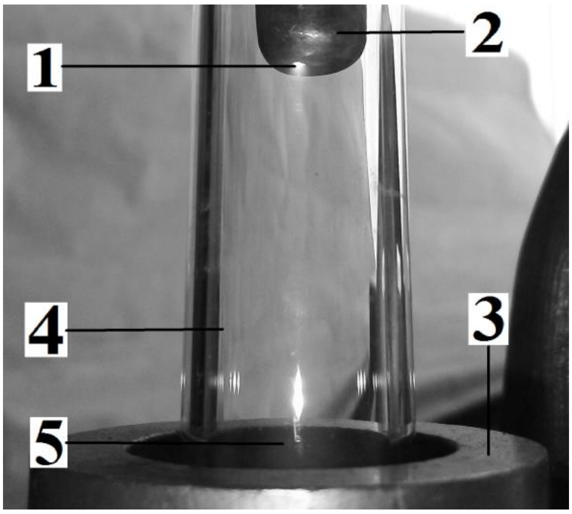

Fig. 4. Formation of a torch on a metal electrode:
1 – A near-electrode spot of the torch; 2 – Metal cap of thermoelectric converter; 3 – The upper grounded electrode; 4 – Quartz tube; 5 – The basis of the diffuse shell of the torch

The torch, which is formed on a separate electrode, stabilizes the plasma. This is confirmed by the fact that in its absence the «tips» of plasma 1 (Fig. 3) at the top of the electrode 3 move to the inner surface of the quartz tube 4, moving in a circle centered on the axis of the tube or perform oscillatory movements along the arc of the circle. In this case, changes in the shape and size of the «tips» of the plasma are also possible. And after the formation of the torch, no movements are noticed. Stabilization of the torch in general and a near-electrode spot in particular is manifested in fixing the location of the spot relative to the electrode and the shape of the diffuse shell. This makes it possible to form a coating on the end part of the cap-electrode, which serves as a substrate in our case. An important effect that was found in our studies was the presence of a sharp boundary of this film coating, and the coating area corresponded to the size of the near-electrode plasma.

An aerosol of a water solution of aluminum nitrate was used to produce the aluminum oxide film, and the zinc oxide film was precipitated by an aerosol of a water solution of zinc acetate. According to R_f power = 80 W, high-frequency capacitive discharge voltage U_c = 2000 V and the feed rate of plasma-forming argon gas 0.01 l/s, the deposition rate of aluminum oxide films was 50 nm/s, and zinc oxide-40 nm/sec.

Experiments on the process of pyrolysis of zinc acetate to its oxide in high-frequency discharge plasma were carried out more clearly. The confirmation of the completed pyrolysis of zinc acetate in a capacitive discharge was a change in the glow of argon plasma. The lower part of the capacitive discharge had a predominant purple hue, characteristic of argon radiation without impurities of other gases. At the level of this part of the discharge, it was not possible to obtain a coating of zinc oxide on the inner surface of the quartz tube with the formation of turbulence.

There was the transition region above, the length of which significantly depended on the active capacity of the capacitive discharge and the flow rate of argon transport gas. These parameters influenced the placement of the transition region along the discharge cord, the shift of which was also carried out by moving the external electrodes. Above this area, to the upper end of the cord, the glow was uniform, indicating a constant composition of the plasma. In the visible emission spectrum of plasma with the pyrolysis products, intense blue lines were dominant. From this plasma, at any level, a coating of zinc oxide could be obtained on the inner surface of the quartz tube.

The presence of clearly defined zones on the cord of high-frequency capacitive discharge makes it possible to control plasma-chemical reactions and effectively place the substrate from the point of view of energy costs and materials, that is, to organize the technological process of synthesis and deposition of coatings.

For high-frequency flare discharge, ionization processes predominate in the gas volume, and secondary processes at the electrodes and on the walls of the discharge chamber are less important [12]. And for this reason, the presence of a thin dielectric substrate on the end surface of the thermocouple unit 2 (Fig. 1) did not change the conditions of formation of films of titanium nitride or zinc oxide.

Conclusions and prospects for further research.

The effect of transfer of working substances by a narrow channel (cord) of high-frequency capacitive discharge is experimentally confirmed. This makes it possible to carry out chemical reactions of the preparation of a pair of working substances for the synthesis of the film on the substrate. The reactions were carried out on the example of pyrolysis, which resulted in a working oxide and gaseous substances that are easily removed and do not affect the synthesis of the film. For theoretical justification of the described effect it is necessary to conduct probe studies for visualization of temperature fields in plasma.

The possibility of forming a plasma high-frequency capacitive discharge of the torch, which concentrated the working substances on the substrate and created stable conditions for the synthesis of thin films was experimentally revealed. We need probe studies in the diffuse shell of the torch of additional single-electrode high-frequency discharge, which will confirm the stabilization of the temperature fields of the plasma after the formation of the torch.

Oxide films that can be used in nanotechnology have been obtained. Fixing the boundary of the film formed from the flare discharge is not enough to detect the discharge conditions and create a model of the film deposition process. First of all, it is necessary to profile the surface of the film in different directions, as well as the use of other methods of studying the morphology of the surface. Since for films with small size, profiling is quite a difficult task, it is planned to upgrade the plasma torch in order to obtain a coating on a larger area by moving the end surface of the electrode, which serves as a lining, relatively near the electrode spot of the torch.

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