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Non-self-sustained discharge with hollow anode for plasma-based surface treatment

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Abstract. The paper discusses plasma methods for surface modification using the non-self-sustained glow discharge with a hollow anode. This discharge is characterised by low voltage and high values of electron and ion currents. It can be easily excited in vacuum-arc installations that are widely used for coatings deposition. It is shown that such type of discharge may be effectively used for ion pumping, film deposition, ion etching, diffusion saturation of metallic materials, fusion and brazing of metals, and for combined application of above mentioned technologies in a single vacuum cycle.

Key words: diffusion saturation • film deposition • hollow anode • ion etching • ion pumping • non-self-sustained glow discharge

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Introduction

Non-self-sustained gas discharge, in which the additional charge carriers are produced by a vacuum-arc evaporator, is characterised by high values of current and degree of ionisation [1–3]. Such type of discharge may be easily excited in widely used vacuum--arc deposition set-ups. Power sources in such set--ups provide, as a rule, the arc current ranging from tens to hundreds of amperes at a voltage of a few tens of volts between metallic cathodes and anode. With easy switching on such equipment, one can obtain a discharge in a gas with almost the same values of current and voltage. Due to enhanced plasma density and degree of ionisation, the processes of surface treatment in such gas discharge are much more intense than they are in a self-sustained glow discharge. The application of hollow anode instead of a plane one provides an additional opportunity for ion flux focusing [3–5]. As a result, the ion current density is increased by one order of magnitude that in turn increases the rate of etching, film deposition or diffusion saturation.

In this paper, we present results for plasma parameters measurements and discuss some technological methods of plasma surface modification with using the non-self-sustained gas discharge with a hollow anode.

Experimental set-up

Figure 1 shows the scheme of Bulat-type deposition set-up, which is additionally equipped with switches and screens to produce a dense flux of gaseous ions. The plasma source 1 with anode 5 can work in two modes: (1) as a usual electric-arc evaporator with consumable cathode 7, that is, as a source of metal ions, and (2) as a source of gas ions, when it forms together with anode 5 the hollow anode. In the first case, the switch SW1 connects the negative terminal of power source PS-1 to cathode 7 and positive terminal to the anode 5 and chamber 10. In second case, the switch SW1 connects the negative terminal of PS-1 to the chamber 10, and positive one to the anode 5, to cathode 7, and to additional ring anode 8. Since the output voltage of power supply PS-1 does not exceed 110 V, the gas discharge between hollow anode and chamber walls cannot start. Its ignition at such a low voltage requires the presence of additional charge carriers.

These charge carriers are produced by the vacuum-arc evaporator 2. Its output aperture is overlapped by screen 3, which prevents the deposition of ions and neutrals on samples 9. At the same time, a small portion of electrons is scattered by the molecules of working gas, enters in the chamber and ignites the gas discharge between hollow anode (consisting of electrodes 5, 7, 8) and chamber 10. The connection of the ring electrode 8 to structure of hollow anode significantly reduces the minimal pressure at which the discharge is burning. The diaphragm 4 reduces the entrance hole of the hollow anode, thereby increasing the gradient of electric potential here. Sufficiently high electric field in this region, together with the magnetic field of focusing coil 6, enhances the degree of ionisation and forms the dense flux of gaseous ions emitted from the hollow anode into chamber 10. This flux may be used for intensive cleaning or surface etching of samples 9, depending on the value of the negative bias applied to them from power source PS-3. If the negative terminal of power source PS-3 is applied to the chamber 10 and positive one to the samples 9, another non-self-sustained glow discharge is exited between walls of chamber 10 and samples 9. In this case, the samples are heated by electrons of this discharge and may be subjected to diffusion saturation (without etching of its surface) in intensive ion flux, which is emitted from a hollow anode (see Fig. 2).

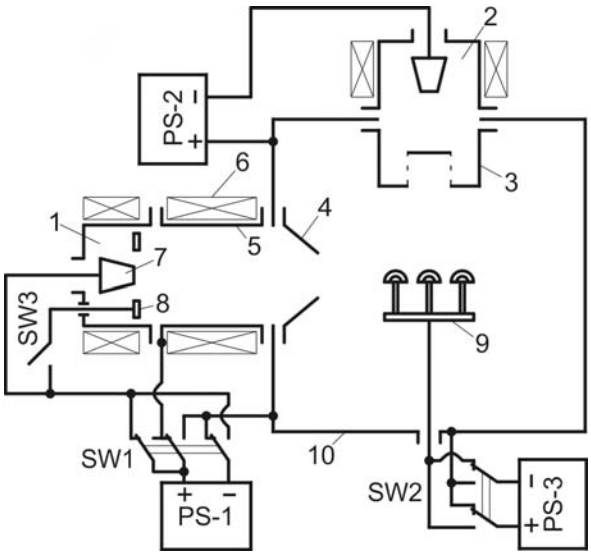

Fig. 1. Scheme of the experimental set-up. Notations: 1 & 2 – vacuum-arc evaporators; 3 – screen; 4 – diaphragm; 5 – anode; 6 – focusing coil; 7 – central anode (or cathode in deposition mode); 8 – additional ring anode; 9 – samples; 10 – vacuum chamber. Power sources PS-1 and PS-2 supply arc and gaseous discharges; PS-3 is a bias voltage source.

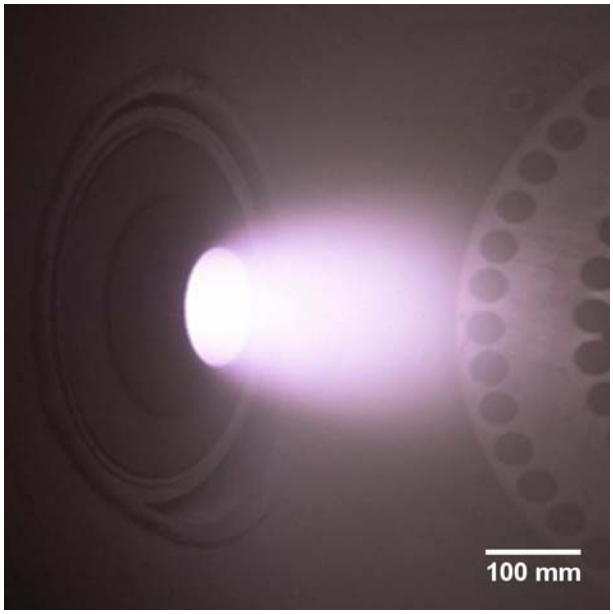

Fig. 2. The nitrogen ions beam emitted from the hollow anode.

Results and discussion

Plasma parameters

Plasma parameters of discharge were measured with a Langmuir probe and two-grid retarding field analyser. The distribution of a floating potential V_f along the axis of a hollow anode is presented in Fig. 3.

Due to the voltage drop near the diaphragm 4 (see Fig. 1), the electrons are accelerated into

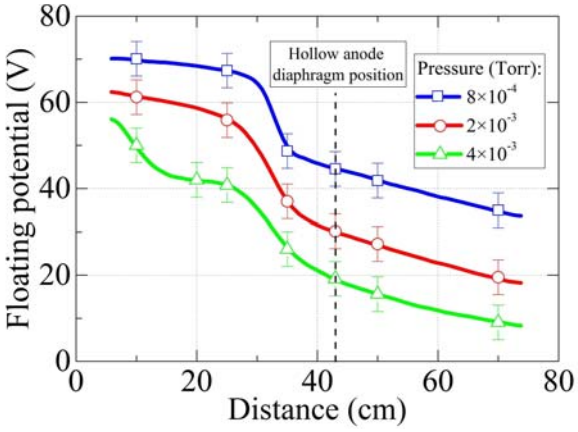

Fig. 3. Distribution of floating potential along the axis of the hollow anode.

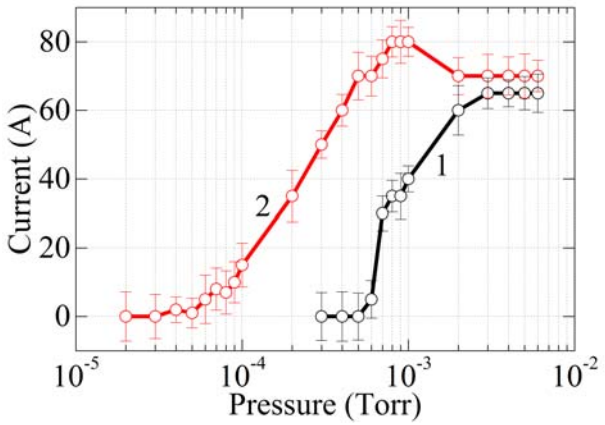

Fig. 4. Non-self-sustained discharge current as a function of nitrogen gas pressure: 1 – the ring electrode 8 (see Fig. 1) is not connected to the hollow anode; 2 – the ring electrode 8 is connected to the hollow anode.

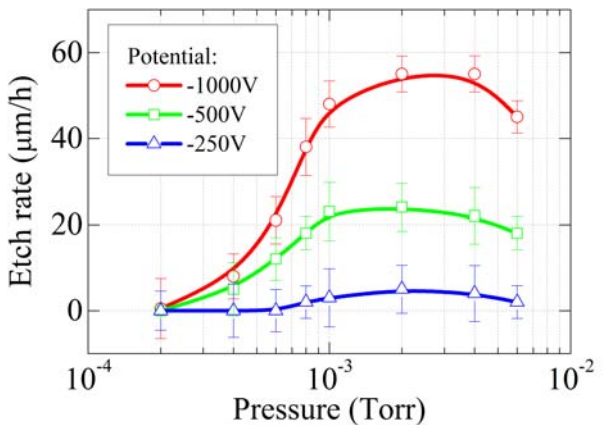

Fig. 6. The etch rates of stainless steel samples by argon ions ejected from hollow anode as functions of gas pressure. The samples are at a distance of 10 cm from the aperture of hollow anode.

a hollow anode, causing ionisation, and the ions are accelerated into the chamber. The ion current through the diaphragm 4 increases with a pressure and reaches its maximum value up to 3 A in a nitrogen discharge, and up to 2 A in a propane-butane discharge. This current is directly proportional to the total current of non-self-sustained glow discharge, which depends on pressure as it is shown in Fig. 4.

As it can be seen in Fig. 4, the total current is significantly greater (curve 2), if the ring electrode 8 (see Fig. 1) is connected to the hollow anode. The mean energy of ions emitted from the hollow anode depends on a gas pressure, diaphragm diameter, on a distance of the diaphragm and, as has been shown in [3], varies from 10 to 30 eV when the potential is distributed according to Fig. 3.

In nitrogen plasma, the electron temperature reaches up to 22 ± 3 eV at a pressure of (6÷20) × 10^{-5} Torr (see Fig. 5, curve 1). As the pressure increases, the electron temperature falls and reduces to 1.5 ± 0.5 eV under 8 × 10^{-3} Torr. Anomalously high temperature at low pressures may be related to the primary electrons, which are accelerated by voltage of 25 V between the cathode and anode of the vacuum-arc evaporator 2, and are scattered elastically on gas molecules inside the chamber. The

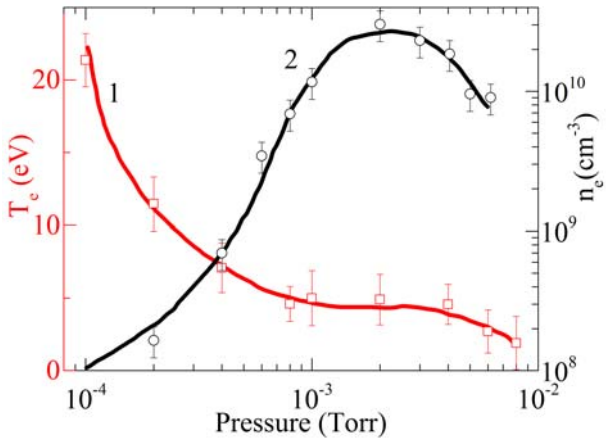

Fig. 5. Electron temperature – 1 and electron density – 2 in nitrogen plasma as function of pressure. The measurements were made in chamber on the axis of hollow anode at a distance of 7 cm from diaphragm.

temperature of electrons in hydrocarbon plasma has a similar dependence on the pressure, but is approximately two times lower than in nitrogen plasma. It should be noted that the electron temperature is practically the same in different parts of the vacuum chamber. In contrast, the electron density grows as one approaches the hollow anode. The maximum of electron density in nitrogen and hydrocarbon plasma is observed at pressures of (1÷4) × 10^{-3} Torr. In particular, the electron density has a maximum value of (2 ± 0.5) × 10^{10} cm^{-3} on the axis of the hollow anode, at a distance of 7 cm of diaphragm 4 in nitrogen plasma (see Fig. 5, curve 2).

Ion etching, diffusion saturation and film deposition

Sufficiently high ion current density (up to 50 mA/cm^2) in the vicinity of diaphragm 4 (Fig. 1) may be used for a high-speed etching of metallic samples. Figure 6 shows the etch rates vs. pressure of stainless steel plates placed in the vacuum chamber 10 at a distance of 10 cm from the diaphragm 4. The surface of plates was oriented at an angle of 60° to the direction of flux of argon ions. The curves were obtained for three values of bias voltage: –250, –500 and –1000 V. The etching was performed in the discontinuous mode so that the temperature of plates does not exceed 650°C. As seen in Fig. 6, in non-self-sustained glow discharge with the hollow anode the etch rate reaches several tens of microns per hour and over.

It is known that in a non-self-sustained discharge the rate of diffusion saturation process, such as nitriding, oxidation, carburisation, is much greater than, e.g., in the nitriding in glow discharge [6]. The excitation of such discharge with the hollow anode makes the saturation more intensive due to an increase in the ion current density.

Figure 7 shows the microhardness distribution on a depth in the VT-1.0 titanium-based alloy plate of dimensions of (20 × 10 × 2) mm^3 after 20 min exposition in a nitrogen-ion flux at 850°C. The surface of the plate was oriented normal to the ion flux. It should be noted that the back surface of the plate is nitrided to the same extent as the side facing the flow

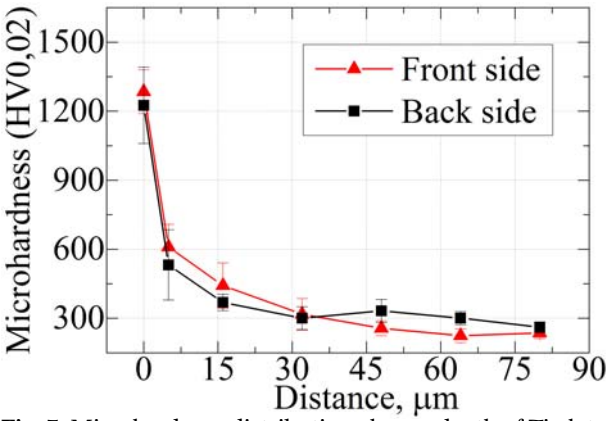

Fig. 7. Microhardness distribution along a depth of Ti plate after nitriding at 850°C. The ion flux is normal to the surface of plate. $P = 1 \times 10^{-3}$ Torr. Front side is the side turned to ion flux, and back side is in the shadow of ion flux. The nitriding time was 20 min.

of ions. The distribution of hardness vs. depth does not depend on whether the samples were heated by ions (when were negatively biased) or by electrons (when the samples had a positive potential). The second variant is advantageous since the saturation of products is not accompanied by their etching. More detailed data on the diffusion saturation of titanium and stainless steel in non-self-maintained gaseous discharge are presented in [4] and [5].

The discharge with the hollow anode may be used also to produce thin films from a gas phase. For example, for the a-C:H films deposition the non-self--sustained discharge may be exited in propane--butane mixture [3].

Ion pumping, quenching, brazing and melting

At the middle stage of evacuation of the vacuum chamber, the excitation of non-self-sustained discharge in residual vacuum markedly increases the rate of pumping. In addition, the vacuum degasation of the chamber walls and heating of the samples occur. Applying a negative bias to the samples allows its cleaning yet during pumping. The connection of the ring electrode 8 (see Fig. 1) to the hollow anode can significantly expand the range of pressures under which the non-self-sustained discharge may exist (see Fig. 4). The main part of discharge current falls on the central electrode 7 (see Fig. 1), whereas on the ring electrode 8 current does not exceed 6 A. Thus, the usage of non-self-sustained discharge allows significant reduction of the cycle time of plasma treatment.

For specimens heating in vacuum furnaces with the aim of the subsequent quenching, two methods are mainly used: induction heating and indirect--resistance heating [7]. The induction heating heats up primarily the outer layer of specimen. As a result, the temperature is often overshot and non-uniform heating occurs. On the other hand, the indirect--resistance elements are expensive and oxidise easily. The heating in non-self-sustained gas discharge is free from these disadvantages. Power for heating is released directly on the specimen. For example,

Fig. 8. A melted tungsten rod.

to heat the samples 9 in Fig. 1, it is necessary to connect the negative terminal of power source PS-3 to the chamber 10 and positive one to the holder of samples 9. The electron streams may be distributed by shading screens in a suitable manner over the surface of samples for uniform heating. The rapid cooling of quenched specimens is achieved by quick sending the inert gas into vacuum chamber.

Any heat-resistant material may be bonded securely in non-self-sustained gas discharge by melting a thin layer of filler metal in the space between them. For example, the cathodes of graphite and tungsten for PVD coating equipment may be reliably bonded with the titanium or stainless steel holder through the copper foil. Ceramics and glass can be bonded with any metal in the same manner. When the temperature of products to be joined is above the melting point of copper, the copper wets and spreads over the surface of products, providing the good adhesive bonding after cooling. In some cases, copper and metals to be joined form a eutectoids.

Any metal may be melted by concentrating the flux of electrons in non-self-sustained gas discharge on specimen. In particular, the melting point of tungsten is easily achievable, as shown in Fig. 8.

Conclusions

The non-self-sustained gaseous discharge with a hollow anode may find a wide variety of applications. Among them are surface saturation, thin film deposition, high speed etching of metals and vacuum heat treatment such as quenching, brazing and melting of metals, including refractory ones. Sufficiently high ion current density in vicinity of aperture of hollow anode allows to increase the etch rate to more then 100 μm/h. The thickness of layer saturated with nitrogen in titanium or stainless steel is growing at a rate of 0.5–1 μm/min. It was found also that the nitriding, oxidation or carburisation occurs on surfaces that have no contact with plasma, that is, in hollows or cavities. The heating of samples by electrons of non-self-sustained gas discharge avoids the ion etching and does not degrade the quality of the treated surface during the saturation process. All of the above-mentioned processing methods, together with the vacuum arc coating technology, may be implemented in a single vacuum cycle. For example, parts of tools to be joined may be first brazed by heating in non-self-sustained argon discharge initiated between these parts 9 (anode) and chamber 10 (cathode) (Fig. 1). Then these parts may be etched in argon ion flux emitted from the hollow anode. The next steps may be nitriding and deposition of TiN or MoN coating depending on the

material of cathode 7 (Fig. 1). Thus, the application of non-self-sustained gas discharge with the hollow anode may significantly extend the technological capabilities of vacuum-arc deposition set-ups.

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