Plasma Production in a Low-Pressure Hollow-Cathode Non-Self-Sustained Discharge[1]

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Abstract – Research on a hollow-cathode non-self-sustained glow discharge demonstrate the feasibility of the discharge operation at pressures from 0.1 Pa. The discharge operating voltage, depending on experimental conditions, can reach ~ 50 V, which is much lower than that of a self-sustained glow discharge in the same electrode system. The discharge current can range to 30 A. The working gas is Ar, N_2, and N-based gas mixtures. Probe measurements of the discharge plasma show that the electron temperature is variable between 1.5 and 7 eV, depending on the gas pressure and kind, and the plasma density can reach 10^{11} cm^{-3} at 15-A currents. Nitriding in the discharge plasma at 600 °C for 2 h made it possible to increase the hardness of Ti (VT-1-0) 3.5 times and that of 12Cr18Ni10Ti three times, with a modified layer depth of 35 μm.

1. Introduction

Recently, diffusion saturation of materials and products has found wide application for increasing their hardness, wear and corrosion resistances in different industries. Among the most widespread modifications of the type are carbonization (carbon ion saturation) [1] and nitriding (nitrogen ion saturation) [1]. Much used nitriding methods are nitriding in a glow discharge in dissociated ammonia [2], in an arc discharge [3], and in a hollow-cathode non-self-sustained glow discharge [4]. The latter work demonstrated the advantages of nitriding in a hollow-cathode glow discharge such as the shorter nitriding time and the feasibility of nitriding of Ti alloys at comparatively low temperatures. However, the electrode system used in the work was inapplicable for treatment of several objects and large-size objects. Using the results of the foregoing work, a test bench has been made for studying the low-pressure hollow-cathode glow discharge and the applicability of this circuit to nitriding various steels alloys, including Ti alloys, under near-industrial conditions.

2. Experimental facility

The experimental test bench is based on a commercial NNV-6.6-I1 facility equipped with a turbomolecular pump TMN-500 with a pumping rate of 500 l/s. The dimensions of the vacuum chamber are 650×650×650 mm. The facility is also equipped with an electron source based on a plasma source with an integrally cold hollow cathode [5].

The glow discharge is ignited between a hollow cathode of surface area $1.7 \cdot 10^4$ cm^2 (the vacuum chamber) and a water-cooled anode of area 200 cm^2. For easy ignition of the glow discharge and its stable operation at low pressures, electrons are emitted to the hollow cathode through a grid. The electrons are emitted from the plasma produced by the plasma source with a cold hollow cathode and a hollow anode whose face is covered with a fine metal grid. Efficient confinement of the arc discharge plasma to preclude its ingress into the vacuum chamber, which is the hollow cathode of the main discharge, a system with two grids and an equipotential gap between them is employed.

A photo of the electron source is shown in Fig. 1.

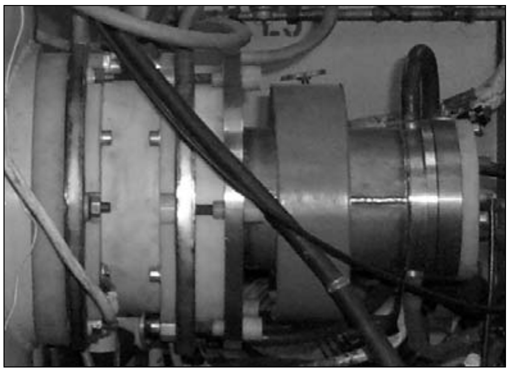

Fig. 1. Photo of the electron source with a cold hollow cathode

Schematic of the experimental test-bench is shown in Fig. 2.

3. Results and discussion

We studied the characteristics of the self- and non-self-sustained glow discharges in the electrode system in question and nitrided 12Cr18Ni10Ti stainless steel and VT-1-0 alloy.

Figure 3 shows a family of the current-voltage characteristics of the non-self-sustained glow discharge at different N_2 pressures for an arc (auxiliary) discharge current I_a = 25 A.

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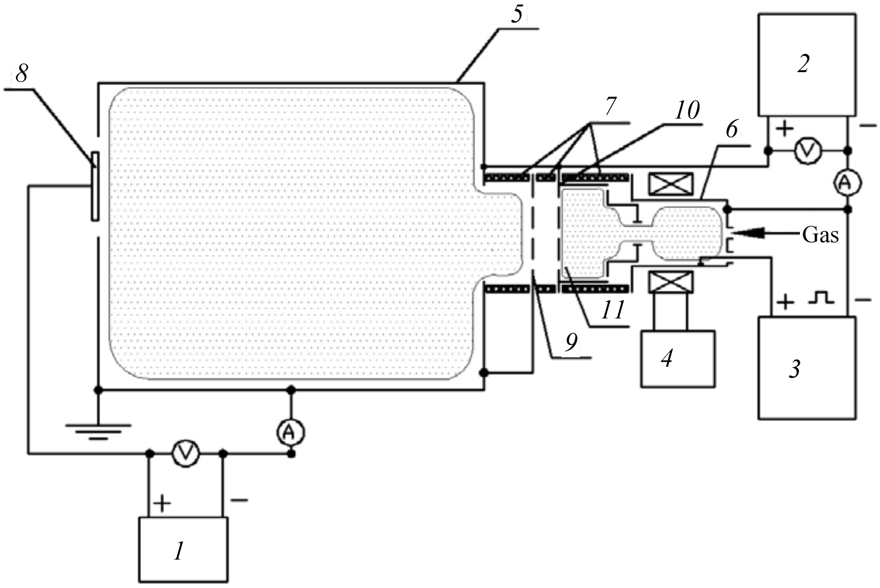

Fig. 2. Experimental bench test: *1* – power supply of the glow discharge; *2* – power supply of the arc discharge; *3* – trigger pulse source; *4* – power supply of the magnetic coil; *5* – vacuum chamber; *6* – integrally cold hollow cathode; *7* – insulators; *8* – glow discharge anode; *9* – grid; *10* – arc discharge anode; *11* – arc discharge plasma

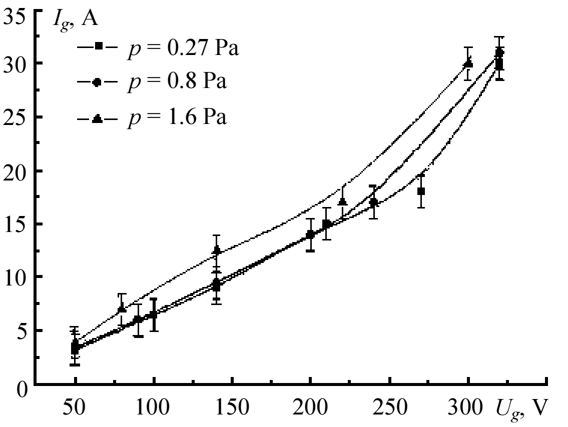

Fig. 3. Current-voltage characteristic of the non-self-sustained glow discharge

It is seen that stable operation of the non-self-sustained discharge is observed even at U_g = 50 V with a discharge current I_g ~ 3 A. As the voltage U_g is increased to 220–270 V (depending on pressure), the current I_g increases gradually to 17–18 A, whereupon its increase becomes more rapid. This behavior of the current-voltage characteristic can be explained as follows. The first portion of the characteristic corresponds to a non-self-sustained discharge whose existence is due to electrons extracted from the auxiliary discharge. The second portion of the curve, where the current grows more rapidly, corresponds to the γ-processes occurring on the surface of the hollow cathode. The inflection of the current-voltage characteristic corresponds to the initiating voltage of the self-sustained glow discharge for this electrode system. The current-voltage characteristic of the self-sustained glow discharge at an N_2 pressure p = 1.6 Pa is shown in Fig. 4.

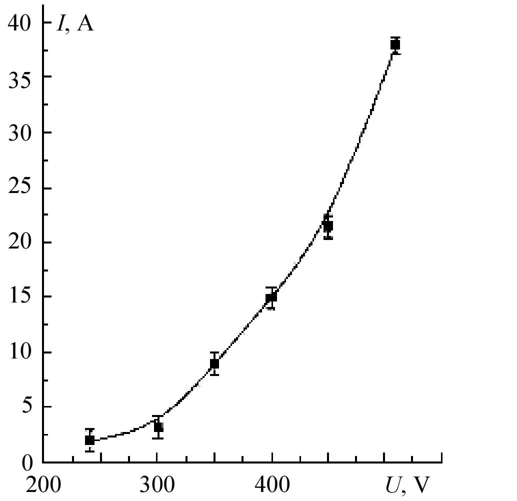

Fig. 4. Current-voltage characteristic of the self-sustained glow discharge. The pressure in the chamber p = 1.6 Pa

It is seen that at p = 1.6 Pa, the initiating voltage of the self-sustained discharge for this electrode configuration U_g = 240 V. It is in this voltage range that the inflection of the current-voltage characteristic of the non-self-sustained discharge is observed with increasing the discharge current risetime.

In this electrode system, we also studied the dependence of the initiating voltage of the self-sustained discharge on the anode area. For this purpose, an area-variable water-cooled anode was made. The results of experiments are shown in Fig. 5.

It is seen that increasing the anode area causes a considerable increase in discharge operating voltage.

For nitriding Ti alloys, it is appropriate to use nitrogen-argon and helium-neon gas mixtures [6]. We took probe measurements of the plasma parameters in these gas mixtures. For comparison of the plasma parameters in different working gases, the auxiliary discharge current $I_a = 15$ A and voltage $U_a = 36$ V, and the glow discharge current $I_g = 14$ A were kept constant. The operating voltages for each gas, in this case, were different. The measurements were taken at varied pressure.

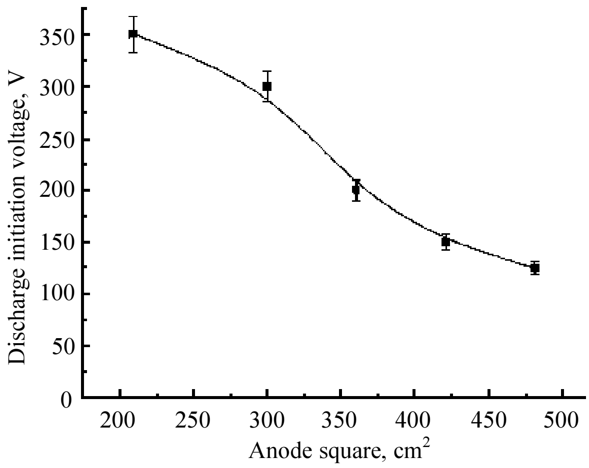

Fig. 5. Initiating voltage of the self-sustained glow discharge versus the anode area. The Ar, pressure 0.4 Pa

Figure 6 shows the operating voltage of the non-self-sustained glow discharge versus pressure for different gases.

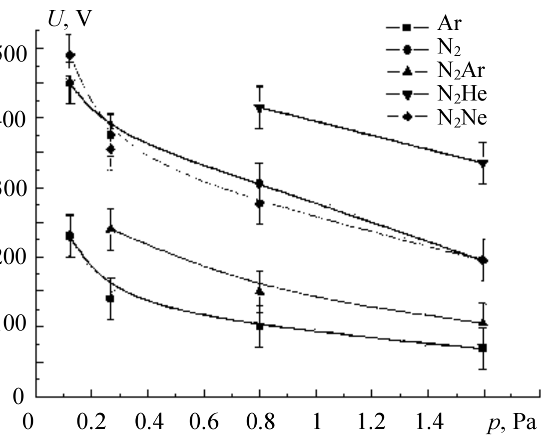

Fig. 6. Operating voltage of the non-self-sustained glow discharge versus pressure at a 14-A current for different gases

The gas-mixture ratios were specified by the particle pressure as 1/1.

It is seen that the nitrogen-helium mixture provides the highest operating voltage and the lowest operating voltage is found with argon.

The results of measurements of the plasma parameters by a double probe are shown in Table 1. It is seen that the lowest electron temperature is found with the nitrogen – argon mixture, and the highest one with the argon – helium and argon – neon mixtures. The highest ion density in the plasma is attained with argon, and the lowest one with the nitrogen – helium gas mixture. The pressure dependence of the ion density in the plasma is direct and that of the electron temperature in the plasma is inverse.

In addition to the studies described above, we investigated the non-self-sustained discharge in the case where the electrons extracted from the auxiliary discharge were accelerated in the space between the grids by an additional voltage source. Figure 7 shows the current of the non-self-sustained glow discharge and the emission grid current versus pressure. The working gas is nitrogen, the glow discharge voltage $U_g = 250$ V, the accelerating voltage $U_e = 60$ V, the auxiliary discharge current $I_a = 15$ A, and the auxiliary discharge operating voltage $U_a = 50$ V.

The character of the pressure dependence of the hollow-cathode glow discharge current is similar to that described in [7]. The pressure dependence of the emission grid current nearly replicates that of the glow discharge current. This may be associated with the fact that in this circuit, not only the electrons from the auxiliary discharge, but part of the ions from the glow discharge as well participate in the current transport. As this takes place, the auxiliary discharge, like the main discharge, is a hollow-cathode discharge, and hence the variation in the current with pressure in the auxiliary discharge features the same peculiarities as the main discharge.

Moreover, with the system under consideration, nitriding in the self-sustained discharge at a pressure of 0.8 Pa was realized. The specimens to be nitrided were 12Cr18Ni10Ti stainless steel and VT-1-0 titanium. The specimens were at potential of the chamber (cathode). The current of the glow discharge $I_g = 10$ A.

Table I. Plasma parameters measured with a double probe

	$p = 0.12$ Pa		$p = 0.27$ Pa		$p = 0.8$ Pa		$p = 1.6$ Pa	
	T_e, eV	n_i, cm^{-3}	T_e, eV	n_i, cm^{-3}	T_e, eV	n_i, cm^{-3}	T_e, eV	n_i, cm^{-3}
Ar	4.3	$1.4 \cdot 10^{11}$	3.14	$2 \cdot 10^{11}$	2.36	$2.5 \cdot 10^{11}$	2.1	$2 \cdot 10^{11}$
Ar + N_2	–	–	2.57	$1.7 \cdot 10^{11}$	2	$2 \cdot 10^{11}$	1.6	$1.3 \cdot 10^{11}$
N_2	5.6	$7.7 \cdot 10^{10}$	4	$1.1 \cdot 10^{11}$	2.57	$1.3 \cdot 10^{11}$	2.5	$1.8 \cdot 10^{11}$
N_2 + Ne	7.19	$7.5 \cdot 10^{10}$	4.75	$7.3 \cdot 10^{10}$	3.17	$1.5 \cdot 10^{11}$	2.5	$1.8 \cdot 10^{11}$
N_2 + He	–	–	–	–	3.1	$1 \cdot 10^{11}$	2.5	$1.1 \cdot 10^{11}$

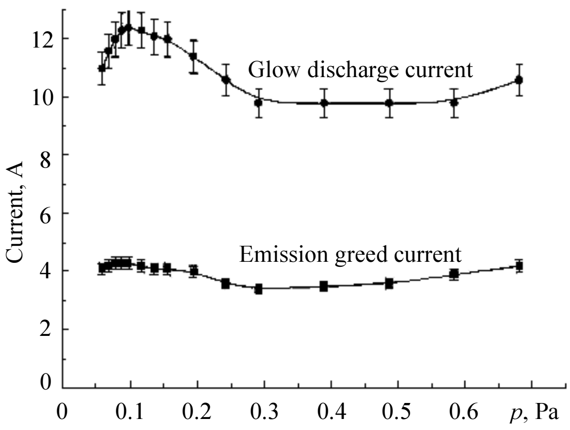

Fig. 7. Non-self-sustained glow discharge current and emission grid current versus pressure

The ion current density to the specimens $J_i = 1$ mA/cm^2. The specimen temperature was 450 °C. After two-hour treatment, the microhardness of the 12Cr18Ni10Ti specimen was increased near 1.5 times and was 4.8 GPa, and that of the VT-1-0 specimen was increased by 10% and was 2.2 GPa. After four-hour treatment under the same conditions, the microhardness of the specimens was 12 and 2.3 GPa, respectively. Figure 8 shows the microhardness distribution in depth for the 12Cr18Ni10Ti specimen after nitriding at a temperature of 450 °C for 4 h.

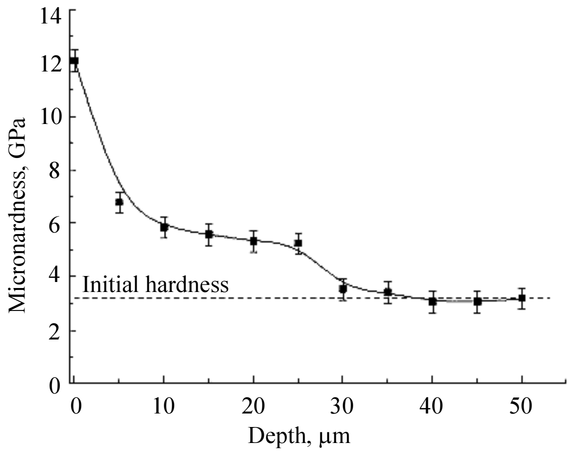

Fig. 8. Microhardness distribution in depth for the 12Cr18Ni10Ti specimen

It is seen that the modified layer with increased microhardness is about 30 μm.

The VT-1-0 specimen was also nitrided in the non-self-sustained glow discharge plasma. In the process, an additional negative bias of 400 V was applied to the specimen. The ion current density $J_i = 2.2$ μA/cm^2. The specimen temperature was 600 °C. The nitriding time was 1 h. The process was realized in the nitrogen – helium mixture (in the ratio 1/1). The surface microhardness was 11 GPa.

4. Conclusion

The experimental bench-test was designed for studying the low-pressure high-current hollow-cathode glow discharge. Investigation of the hollow-cathode non-self-sustained glow discharge show that this discharge can operate at pressures ranging from 0.1 Pa. The discharge operating voltage, depending on experimental conditions, can reach ~50 V, which is six times lower than the operating voltage of the self-sustained discharge in the same electrode system. The discharge current can reach 30 A.

The working gas can be Ar, N_2, and N-based gas mixtures, and this allows the use of the discharge for intense nitriding of Ti alloys.

The measurements of the plasma parameters of the non-self-sustained glow discharge show that the electron temperature, depending on the gas pressure and kind, can vary between 1.5 and 7 eV and the plasma density at currents of 15 A can reach 10^{11} cm^{-3}.

Experiments on nitriding in the non-self-sustained glow discharge were realized. The experiments show that the discharge can be used for nitriding. The surface microhardness of the 12Cr18Ni10Ti specimen after nitriding at a temperature of 450 °C for 4 h increases more than three times and reaches 11 GPa.

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