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Non-self-sustained low-pressure glow discharge for nitriding steels and alloys

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Abstract. Structural steel 40Cr and die steels 8Cr4W2MoVSi2 and Cr12Mo were nitrided in the plasma of a non-self-sustained hollow-cathode glow discharge at a pressure of 1 Pa, negative bias of 300 V, and temperature of 520 °C and 400 °C. The method has been shown promising for low-temperature nitriding of steels to a depth of several tens of micrometers with minimum tempering.

1. Introduction

The most efficient and ecologically clean methods of increasing the wear resistance of metal products are those based on electron-ion plasma surface treatment [1]. In a hollow-cathode glow discharge at a pressure of ≈1 Pa, the electron free path measures several to tens of centimeters, allowing efficient gas ionization by electrons oscillating in the hollow cathode. In self-sustained mode, the discharge operates due to electrons produced through secondary ion-electron emission at the cathode surface and accelerated in the region of near-cathode potential fall. Such self-sustained operation is disadvantageous in the necessity of increasing the voltage to provide discharge currents acceptable for practical use, and this causes substantial etching of the cathode walls and contamination of the plasma by erosion products. In non-self-sustained mode, additional electrons are injected into the plasma, making it possible to decrease the operating pressure several times and the discharge voltage to several tens of volts, and to greatly increase the glow discharge current [2]. Thus, one can produce a plasma of density higher than 10^{18} m^{-3} in vacuum volumes larger than 0.1 m^3 [3]. In such a discharge, the average ion current density to a target reaches 15 mA/cm^2, which is promising for practical applications. The high ion current density in a non-self-sustained glow discharge greatly improves the efficiency of ion cleaning and shortens the nitriding time compared to anomalous glow discharges operating at 50–1000 Pa. Another important factor is that the positive ions produced in the nitrogen-containing discharge plasma and delivered to the surface of a target under negative bias are a source of atomic nitrogen which is absorbed by the surface and diffused deep into the target. Thus, it is valid to say that the plasma of a non-self-sustained glow discharge provides a high saturation efficiency, and this should assist in decreasing the nitriding time and temperature. The decrease in temperature is particularly urgent for hardened steels which are tempered during long-term nitriding at about 520 °C.

The aim of our study was to assess the possibility to obtain hardened layers several μm thick in steels through their low-temperature nitriding in the plasma of a non-self-sustained hollow-cathode glow discharge.

2. Material and research techniques

Figure 1 shows a schematic of the experimental test bench used in the study and described in detail elsewhere [3]. The glow discharge was ignited in its vacuum chamber of dimensions 650×650×650 mm^3 between hollow cathode *1* (inner chamber surface) and plane anode *2*. The glow discharge was powered by a stabilized source with an output voltage of up to U_d = 300 V and average output current of up to I_d = 200 A. For providing stable low-pressure operation of the glow discharge in high-purity nitrogen (99.999 %), we used an electron source based on an arc discharge with an integrally cold hollow cathode[4]. The test materials were specimens of structural steel 40Cr and die steels 8Cr4W2MoVSi2 and Cr12Mo with a diameter of 20 mm and thickness of 4–6 mm. The specimens were fixed on a holder kept at the hollow cathode potential in the center of the chamber and were nitrided for 3 h at a temperature of 520 and 400 °C, pressure of 1 Pa, discharge voltage of 100 V,negative bias of 300 V, and pulse duty factor of 50 % (table 1). Their heating and cleaning were provided via bombardment by nitrogen ions accelerated in the cathode layer of the glow discharge.

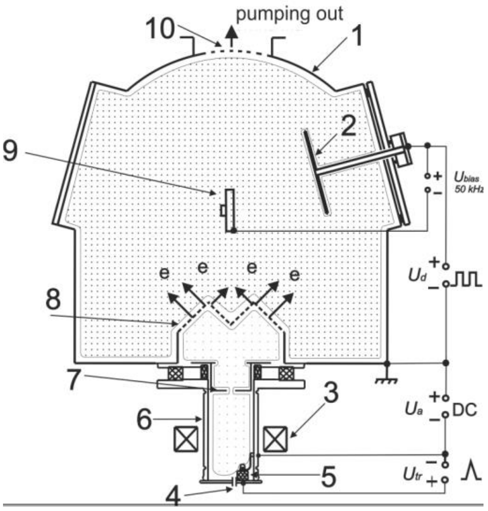

Figure 1. Schematic of experimental arrangement: *1* – cathode of glow discharge, *2* – anode of glow discharge, *3* – magnetic coil, *4* – gas inlet, *5* – igniter electrode, *6* – hollow cathode of auxiliary arc discharge, *7* – arc arrester, *8* – conical grid anode of auxiliary arc discharge, *9* – specimen holder, *10* – grid.

The nitriding efficiency was assessed by measuring the Vickers microhardness of lateral specimen sections in depth from the surface with a step of 10 µm at an indenter load of 0.5 N. Before nitriding, the surface microhardness of steels40Cr, 8Cr4W2MoVSi2, and Cr12Mo was 5.4, 7.1 and 7.3 GPa, respectively.

Table 1. Nitriding modes and surface microhardness of steels.

T, °C	U_d, V	I_d, A	j_i, mA/cm^2	W, kW	U_{bias}, V	$HV_{0.5}$, GPa 40Cr / 8Cr4W2MoVSi2
400	100	29	1	2.9	–300	7.39 / 9.85
520	100	66	1.9	6.6	–300	7.2 / 9.85

T – nitriding temperature, U_d – discharge voltage, I_d – discharge current, j_i – ion current density, W – discharge power, U_{bias} – bias, $HV_{0.5}$ – surface microhardness after nitriding

3. Results and discussion

The microhardness distributions in depth of steels 40Cr, 8Cr4W2MoVSi2, and Cr12Mo are presented in Figures 2(a), (b) and 3, respectively. As can be seen from figures 2, 3, all steels after nitriding reveal a substantial increase in their surface microhardness. The nitrided layer thickness depends on the chemical composition of the steels, and its maximum value of 300 μm is found in low-alloy steel 40Cr nitrided at 520 °C. Decreasing the nitriding temperature to 400 °C decreases the nitrided layer thickness to 140 μm. As it follows from figure 2(a), the bulk microhardness of steel 40Cr after nitriding at 400 °C is 40 % higher than that after nitriding at 520 °C. For steel 8Cr4W2MoVSi2, there is no need to decrease the nitriding temperature because the thickness of its nitrided layer at 400 °C is small, and its heating and aging at 520 °C fails to provide a substantial increase in the bulk microhardness. When nitrided both at 520 and 400 °C, die steel Cr12Mo and steel 40Cr are involved in tempering which decreases their bulk microhardness by 25 % and by 8−10 %, respectively, compared to the initial state. Decreasing their nitriding temperature decreases the nitrided layer thickness to 50 μm, i.e., almost two times.

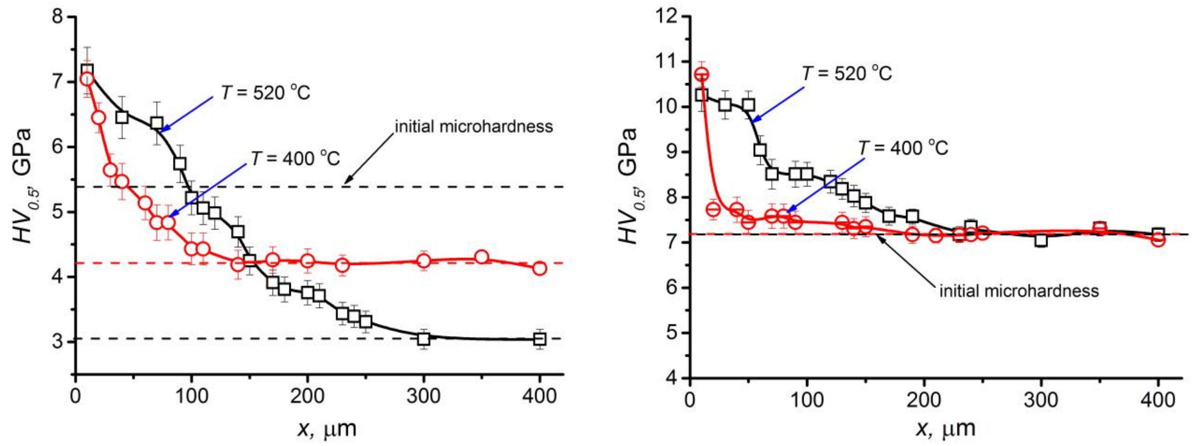

Figure 2. In-depth microhardness distributions in nitrided steels 40Cr (**a**) and 8Cr4W2MoVSi2 (**b**).

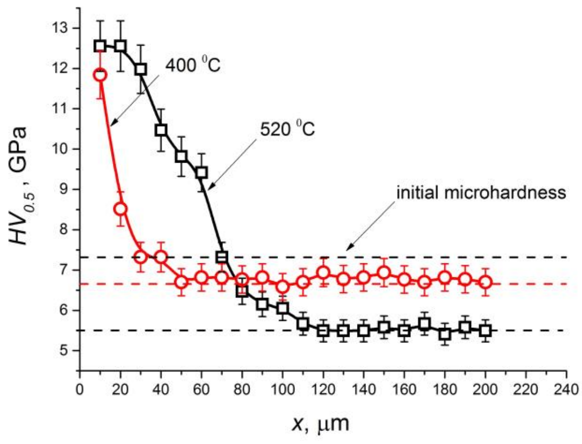

Figure 3. In-depth microhardness in nitrided steel Cr12Mo.

4. Conclusion

Thus, our study demonstrates the efficiency of using a non-self-sustained low-pressure glow discharge with a hollow cathode for nitriding structural and tool steels. The nitrided layer thickness after treatment for 3 h at 400 and 520 °C is respectively 140 and 300 μm in steel 40Cr,≈30 μm and 150 μm in steel 8Cr4W2MoVSi2, and ≈50 μm and 110 μm in steel Cr12Mo.Decreasing the nitriding temperature decreases the nitriding rate and the tempering, and this greatly increases the bulk hardness of steels 40Cr and Cr12Mo. From the data obtained, one can select appropriate temperatures and times of nitriding to provide a desired thickness of nitrided layers and desired bulk hardness of steels.

Acknowledgements
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