Effect of applying a magnetic field in ion nitriding on probe characteristics of glow discharge, microhardness and R6M5 steel structure

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Abstract. Ion nitriding in a magnetic field has been studied. The influence of the magnetic field on glow discharge characteristics, structural-phase composition of the modified layer and surface microhardness of steels R6M5 has been analyzed. The probe current-voltage characteristic of the glow discharge in the magnetic field has been obtained.

1. Introduction
Efficiency, durability and reliability of tools in mechanical engineering are largely determined by surface and not bulk properties of materials. Therefore, a change in surface structural and phase composition can significantly improve the material at a minimum cost [1].

Various methods are used to modify the surface, most of which are based on the use of liquid media and thermal processes. Ion-plasma nitriding holds a special place in surface modification due to its favorable controllability and environmental safety [2].

Ion nitriding is carried out in a metastable glow discharge wherespecimens are heated to nitriding temperatures with simultaneous diffusion of nitrogen ions into the treated surface due to the energy of gas ions, which bombard the surface. All processes in the glow discharge plasma, i.e. excitation, ionization, dissociation and recombination occur in the so-called cathode voltage drop region [3].

Characteristics of glow discharge in a magnetic field determine its use in various gas-discharge systems [4–6]. In this study, glow discharge is used in ion nitriding to increase plasma density and gas ionization efficiency [2].

The purpose of this work was to study the effect of applying a magnetic field on ion nitriding and the structure, phase composition and microhardness of the resulting surface layer of R6M5 tool steel.

2. Methodology
An experiment was carried out on an upgraded electron beam installation ELU-5M (figure 1). A pulsed power supply ApEl M 5PDC was used to power the discharge. Discharge current and potential difference were monitored from the display of the power supply unit.

Ion nitriding in a glow discharge was preceded by ion cleaning of the sample surface during 15 min in Ar at the pressure in the vacuum chamber \( P = 5 \) Pa. Ion nitriding was carried out in a gas mixture of \( \text{N}_2 \) 50–80%, \( \text{Ar} \) 25–10%, \( \text{C}_2\text{H}_2 \) 25–10%. The gas flow rate was controlled by an RRG BUlP 3 control unit. The working gas pressure was constant \( P = 50 \) Pa. Temperature stability was controlled with a Thermixoptical thermometer.
Figure 1. Scheme of the experiment on the installation ELU-5M: 1 – power supply; 2 – cathode; 3 – neodymium magnets; 4 – anode; 5 – vacuum chamber; 6 – toroidal domain of bright glow; 7 – magnetic field lines; 8 – substrate; 9, 10 – samples.

The surface microhardness was measured with a Micromet-5101 microindentation hardness tester under the applied load of 0.49 N. The sample phase composition was studied with a DRON-4-07 diffractometer. The microstructure of the samples after etching in a 4% solution of nitric acid in ethyl alcohol was examined by a GSM 6390 (JEOL) scanning electron microscope.

Langmuir probe current-voltage characteristics [7, 8] were used to determine the main parameters of the glow discharge plasma. A single flat probe was used to measure the I-V characteristics (figure 2). The area of the probe was $S_p = 122.72 \times 10^{-6}$ m$^2$; the probe was energized with a GW INSTEK GPR-25H30D power supply; the I–V characteristics were recorded using a Hantek365F digital multimeter.

The measurement scheme is shown in figure 3.

Figure 2. Scheme of the single flat probe.

Figure 3. Scheme of probe measurements in a magnetic field: PS1 – discharge power supply, PS2 – adjustable power supply of a moving probe, M – magnetic system, A – anode, C – cathode (substrate), I – insulators, P – single flat probe.

3. Results and discussion

Surface microhardness analysis of the specimens made from R6M5 steel has shown that ion nitriding in a magnetic field results in an increase in the microhardness from 4150 to 19500 MPa. Hardening of the steel surface after nitriding appears due to doping a solid solution with nitrogen and the release of highly dispersed nitride particles.
Formation of a modified layer structure in high-speed steels during ion nitriding is specific to the chemical composition of the steels, i.e. high content of carbon and strong nitride-forming alloying elements (Cr, Mo, W, V); it is also influenced by the structure after heat hardening [2]. The initial structure of R6M5 steel after improvement consists of distributed carbides (20–22%) in sorbitol-like pearlite (doped ferrite and carbides M₇C₃) [9]. It is known [2] that a hardened layer with a thickness of not more than 25 μm is formed during gas nitriding of R6M5 steel for 3 hours. The thickness of the hardened layer was 200 μm when a magnetic field was applied during nitriding the steel in glow discharge for 4 h (figure 4), which indicates a high kinetic efficiency of the process.

Figure 4 demonstrates the microstructure of R6M5 steel after ion nitriding in a magnetic field. The microstructure analysis showed that the nitrided layer consisted of two zones. The total thickness of the nitratred layer was considered as a combination of the two zones observed under the microscope – I nitride zone and II diffusion zone (figure 4). The nitride layer appears almost structureless in the microstructure images and includes nitrides of iron and alloying elements. The diffusion sublayer consists of nitrogenous ferrite with carbide and fine nitride inclusions. It is known [2] that the nitride-forming elements V, Mo and Cr impede nitrogen diffusion and reduce the thickness of the nitrided layer while increasing surface microhardness.

![Microstructure of the nitrided layer in R6M5 steel (×270).](image)

There is no clear boundary between the nitride layer and the diffusion sublayer in the microstructure images; the transition from the nitrided layer to the underlying layers is smooth, which is one of the main requirements to a nitrided layer [10, 11].

X-ray structural studies were performed to determine the phase composition of the surface. Reflections of the ε-phase Fe₃N, as well as phases of nitrides of alloying elements (WN, WN₂, CrN, Cr₂N), were found in the diffractogram (figure 5) of the R6M5 steel sample surface after ion nitriding in the applied magnetic field. It is known [3] that iron nitrides have a higher heat capacity than iron, i.e. favorable conditions occur that prevent thermal flashes on the tool surface.

After processing in a mixture of nitrogen, argon and acetylene (N₂ 50–80%, Ar 25–10%, C₂H₂ 25–10%), reflections of iron carbides (Fe₃C) were detected on the surface of the samples. The use of the gas mixture in ion nitriding makes it possible to deactivate residual oxygen and intensify diffusion saturation.

A single flat probe was used to measure current-voltage characteristics at different heights (h) from the magnetic system to determine the influence of the magnetic field on the main characteristics of the glow discharge plasma.
Based on the measurement results, the probe current was expressed as a function of voltage – $I_p = f(U_p)$ (figure 6a); then a logarithm was taken of this function to plot the I–V characteristics on a semilogarithmic scale – $\ln(I_p) = f(U_p)$ (figure 6b).

Floating potentials ($U_f$) and currents ($I_0$) were determined graphically on the obtained I–V characteristics of the glow discharge in the magnetic field (figure 6b). Figures 7–9 demonstrate $U_f$ and $I_0$ for $h = 10–30$ mm.
The main plasma characteristics were calculated using formulas (1–3) [7, 8]; the results are shown in table 1 and figure 10.

Electron temperature:

\[
T_e = \left[ \frac{|e| \cdot \Delta U_p}{k \cdot \Delta \ln I_p} \right]^{1/2},
\]  

(1)

where \(\Delta U_p\) – probe potential difference; \(\Delta \ln(I_p)\) – the difference of logarithms of the probe current.

Electron thermal velocity:

\[
v_e = \frac{8 \cdot k \cdot T_e}{\pi \cdot m}^{1/2},
\]  

(2)

Electron concentration:

\[
n_e = \frac{4 \cdot I_0}{e \cdot v_e \cdot S_p},
\]  

(3)

where \(I_0\) – current of the floating potential \(U_f\).
Table 1. Main characteristics of the magnetized glow discharge plasma.

| Height from the magnetic system $h$ (mm) | Electron temperature $T_e$ (K) | Electron thermal velocity in plasma $v_e$ (m·s$^{-1}$) | Electron concentration $n_e$ (m$^{-3}$) | $I_0$ (µA) |
|----------------------------------------|--------------------------------|-------------------------------------------------|---------------------------------|-------------|
| 10                                      | $17.53 \times 10^3$            | $8.23 \times 10^5$                               | $4.58 \times 10^{16}$           | 0.185       |
| 20                                      | $5.13 \times 10^3$             | $4.45 \times 10^5$                               | $1.42 \times 10^{16}$           | 0.031       |
| 30                                      | $27.55 \times 10^3$            | $10.32 \times 10^5$                              | $0.302 \times 10^{16}$          | 0.015       |

It can be seen from the results that electron concentrations $n_e$ and currents $I_0$ decrease with the increase in the height $h$ from the magnetic system. This is because the magnetic field captures and retains electrons and forms a concentration gradient of charged particles, which leads to an increase in the concentration of electrons in the cathode region.

Besides, it has been established that electron temperature $T_e$ and thermal velocity in plasma $v_e$ decrease and then sharply increase with the increase in the height $h$ from the magnetic system. This is due to the phenomenon known as a cathode voltage drop, where electrons knocked out from the cathode under ion bombardment are accelerated towards the anode when passing through the voltage drop region. In this case, the electrons acquire high energy values.

4. Conclusions

- A mixture of nitrogen, argon and acetylene impedes the formation of an oxide film during nitriding due to deactivation of residual oxygen, which contributes to efficient saturation of the surface with nitrogen.
- Ion nitriding of R6M5 tool steel in a magnetic field results in a significant increase in the surface microhardness by ~ 4.5 times due to the formation of a hardened zone that consists of the nitrides of iron and alloying elements.
- It has been established that electron concentration $n_e$ and currents $I_0$ decrease with the increase in height $h$ from the magnetic system due to the formation of the concentration gradient of charged particles by the magnetic field. Besides, it has been established that electron temperature $T_e$ and thermal velocity $v_e$ decrease and then sharply increase with the increase in height $h$ from the magnetic system due to the cathode potential drop.

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