AISI 5140 steel nitriding in a plasma of a non-self-sustaining arc discharge with a thermionic cathode under the pulse action of ions

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Abstract. Steel AISI 5140 was exposed to pulsed (50 kHz) low-energy (up to 1 kV) nitriding in the plasma of a non-self-sustained arc discharge with a hot hollow cathode in a pure nitrogen atmosphere. The aim was to investigate the effect of the ion energy (pulse amplitude) and ion current density on the nitride layer thickness and on the depth of diffusion saturation with nitrogen. Experiments were also performed to study the effect of the ion energy and ion current density on the efficiency of nitriding at a constant specimen temperature and constant process time.

1. Introduction
Now, pulsed discharge sources are widely used in science and technology [1-3], and their advantage is the possibility to independently control their parameters such as the pulse duration, repetition frequency, duty factor, voltage, and current [4-6]. Among the applications of such sources is diffusion saturation of materials with dopants in gas discharge plasmas, e.g., plasma nitriding of steels and alloys [7]. Varying the parameters of pulsed discharge sources influences the parameters of nitrided layers, and this influence is not quite clear, requiring further research.

Here we analyze the features of AISI 5140 steel nitrided in the plasma of a non-self-sustained arc discharge at different pulse duty factors, bias amplitudes, and arc currents.

2. Material and research technique
All experiments were performed on the COMPLEX vacuum setup (figure 1) designed and manufactured at IHCE SB RAS [5]. For producing a bulk plasma, we used a PINK generator based on a non-self-sustained glow discharge with a hot hollow cathode [8]. In addition to the PINK generator, the setup is equipped with an original metal plasma source based on a vacuum arc (for depositing corrosion-resistant, wear-resistant, and superhard coatings) and a plasma-emitter electron gun (for ultrafast surface melting, quenching, and mixing).

The test material was AISI 5140 steel shaped as cylinders of dimensions Ø20×5 mm. Before treatment, the specimens were polished, washed with benzene in an ultrasonic bath, wiped dry, and fixed on a stainless steel holder of dimensions 120×100×6 mm with a galvanically isolated chromel-alumel thermocouple at its back. The holder was horizontally positioned in a vacuum chamber of dimensions 900×500 mm (figure 2) at 200 mm from the hollow cathode of the PINK generator so that the specimen with its polished side facing the generator was on the system axis. The vacuum chamber
was pumped by a turbomolecular pump to a pressure no greater than $1 \times 10^{-3}$ Pa at 1500 l/s, and upon supplying high-purity nitrogen to the chamber via a gas inlet, a discharge was ignited at 0.6 Pa.

![Image](image1.png)

**Figure 1.** COMPLEX setup.

After ignition of the discharge, a negative pulsed bias with an amplitude of 990 V and frequency of 50 kHz was applied to the holder. The pulse duty factor and the average bias current were set to provide heating of the specimen from room temperature to a required level in 15 min. During this time, the specimen surface was heated, cleaned, and activated. The source ensured a voltage fall time such that the fall time of the ion current was almost independent of its amplitude. As the holder temperature reached 500°C, predetermined discharge parameters were set for each specimen (table 1), and the specimens were kept at them for 1 h (isothermal aging). The operating nitrogen pressure for all specimens was 0.6 Pa. After nitriding, the source and the nitrogen supply were switched off, and the specimens were left in the chamber for cooling. Once their temperature went down to less than 50°C, the specimens were removed from the chamber for examination.

![Image](image2.png)

**Figure 2.** Simplified experimental arrangement.

The hardness on the surface and crosswise the specimens was measured using a PMT-3M device (load 500 mN). The surface morphology and the structure in depth of the nitrided steel were analyzed on a Hitachi S-3400N scanning electron microscope and a μVizo MET-221 metallographic microvisor.
3. Results and discussion

The required specimen temperature in this type of nitriding is determined by the energy and average current of ions arrived at the holder. However, changing the pulse duty factor changes the total time of their direct action because the specimen during pulse-to-pulse intervals is at anode (chamber) potential and the ion current to it is almost absent. Besides, the specimen temperature is decisive for diffusion and phase transformation in the steel, and it is expedient to analyze the efficiency of different modes at the same nitriding temperature.

The influence of the ion energy, pulse duty factor, and ion current density (to the holder) on the nitriding efficiency was studied in experiments at constant specimen temperatures and constant times of their isothermal aging. The chosen operating pressure provides collisionless ion acceleration to the treated surface, i.e., the ion energy is specified by the bias amplitude. The ion energy and the ion current were varied by varying the bias amplitude and discharge current, respectively. The main parameters of the process are presented in table 1.

| Specimen No. | Discharge current, A | Bias, V | Duty factor, % | Ion current, A |
|--------------|---------------------|---------|----------------|----------------|
| 1            | 53                  | 990     | 28             | 0.3            |
| 2            | 17                  | 990     | 85             | 0.3            |
| 3            | 53                  | 390     | 85             | 0.8            |
| 4            | 53                  | 990/390 | 28/85          | 0.3/0.8        |

Our analysis shows that the surface roughness of all specimens after nitriding (figure 3a) is higher, compared to their initial polished state, due ion etching during heating and nitriding. Their cross-sectional structure after nitriding is the same in all cases (figure 3b) and reveals a thin (13–20 µm) near-surface layer, which is likely Fe₄N, and an extended layer of nitrogen solid solution in iron, which is identified from a change in microhardness.

![Figure 3. Nitrided surface of steel specimen No. 1 (a) and its cross-sectional structure (b). Scanning electron microscopy.](image)

The surface microhardness of specimens No. 1 and No. 2 remains almost invariant after increasing the pulse duty factor from 28 to 85 % with a simultaneous decrease in the discharge current at a constant ion energy and constant average ion current density (nitride layer ≈10 µm). However, the zone with enhanced hardness (diffusion saturation with nitrogen) decreases in depth from 250 to 150 µm, which is likely due to the decrease in the non-self-sustained arc current from 53 to 17 A and hence in the maximum ion current density.

Thus, in systems with a pulsed bias, one should take into account that the diffusion rate is influenced mostly by the maximum ion current density rather than by its average value to targets. Reasoning from the above, we supposed that increasing the nitrogen concentration by increasing the
discharge current with a simultaneous decrease in the bias (and hence in the etching rate of near-surface Fe nitride layers) would increase the depth of diffusion saturation.

According to the assumption, experiments were performed in which we varied the ion energy to the specimens by decreasing the amplitude of their pulsed bias at a constant specimen temperature (500°C) and constant arc current (53 A). For specimen No. 3, the pulse duty factor was 85%, and the bias amplitude was 390 V. For specimen No. 4, the mode was mixed: 990 V, 28% within the first 30 min (like for specimen No. 1), and 390 V, 85% within the next 30 min (like for specimen No. 3).

It is seen from figure 4 that decreasing the bias from 990 to 390 V (specimen No. 3) fails to provide the expected increase in the depth of diffusion saturation but increases the surface hardness and the nitride layer thickness to 8.1 GPa and 20 µm, respectively (figure 5a).

![Figure 4. In-depth microhardness distribution (T=500°C).](image)

In this case, the nitride layer formed on the surface is a barrier to nitrogen diffusion deep into the specimen, and the low ion energy does not allow its efficient etching. The formation of an extended diffusion layer (specimen No. 4) with efficient etching at high bias and then a hard nitride layer also fails to increase the depth of diffusion saturation. In this case, the total depth of the nitride layer is about 200 µm, the surface hardness is 8 GPa, and the depth of the nitride zone is 13 µm (figure 5b).

![Figure 5. Typical cross-sections of nitrided steel specimens No. 3 (a) and No. 4 (b). Optical microscopy.](image)
4. Conclusion

Thus, our experiments on non-self-sustained arc plasma nitriding of AISI 5140 steel in a pure nitrogen atmosphere shows the following. Nitriding at 500°C increases the surface microhardness of the steel from 3 to 8 GPa (about three times) and provides the formation of a modified layer of depth up to 250 µm in 1 h.

The in-depth microhardness distribution depends on the ion energy to the treated surface and on the time of direct pulsed exposure.

Acknowledgments

The work was supported by the Russian Science Foundation (project No. 14-29-00091).

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