Ion nitriding effect on the structure and mechanical properties of R6M5 high-speed steel after SPD

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Abstract. The paper studies the effect of ion nitriding in a glow discharge on the structure and mechanical properties of R6M5 steel obtained by the method of traditional hot thermomechanical treatment and subsequent severe plastic deformation (SPD). It is found out that the surface microhardness of material increases significantly (~ 1.7 times) as a result of ionic nitriding after SPD and for the sample without SPD an increase is twice as much times in the line region of carbide-nitride inclusions. It is proved that the microhardness distribution over the diffusion zone depth in the sample after the SPD is smoother, and the thickness of the diffusion layer exceeds almost 2 times.

1. Introduction
Due to the advent of new difficult-to-machine materials, the problem of hardening both cutting and die tools is becoming more and more urgent. One of the main drawbacks of R6M5 steel is the presence of line pattern along the axis roll and an uneven distribution of carbides over the section of a roll when delivered in the form of hot-rolled bars [1].

In order to improve the performance properties of metallic materials and their structural homogeneity, the methods of severe plastic deformation (SPD) have been intensively used [2, 3]. The problem of increasing the hardness and wear resistance of the surface layer of the tool today is still being solved by various methods of chemical-thermal treatment [4, 5].

Ion nitriding in a glow discharge plasma is among the variety of methods for surface hardening, which has many advantages over the traditional ones [4].

The purpose of this work is to study the process of ion nitriding in a glow discharge on the structure and mechanical properties of the surface layer of R6M5 steel after SPD.

2. Research methods
The experiments on the effect of ion nitriding in a glow discharge on the structural-phase composition and mechanical properties of R6M5 high-speed steel after SPD were performed on an upgraded installation ELU-5M designed for thermal and chemical-thermal treatment in vacuum (figure 1). A sample of P6M5 steel in the form of a disk (diameter $D = 20$ mm and thickness $h = 2.2$ mm) was subjected to upsetting by 43 % in a cold state and torsion – 1.5 turns with a hydrostatic pressure of 4 GPa. Subsequently quenching was used (from 1200 °C) and one-time high-temperature tempering (at 560 °C).
The samples were subject to ion nitriding in a gas mixture: nitrogen N₂, argon Ar, and hydrogen H₂, which was added to depassivate oxygen on the surface of material. The soaking temperature was 550±10°C for 6 hours at a gas pressure of 150±5 Pa.

3. Results and discussion
Figure 2 shows the initial microstructure of P6M5 steel obtained by the method of hot thermomechanical treatment and subsequent severe plastic deformation.

The microstructure analysis of the obtained samples showed that with the torsion of 1.5 turns on the periphery (figure 2a) the orientation of carbide inclusions in the tangential direction is observed, which is caused by shear deformations during sample rotation. In the central zone (figure 2b) at a distance of 0.1R, more uniform distribution of carbides is observed. Neither banding nor stripiness was spotted. The hardness from the surface of the sample amounted to 67 HRC.
A detailed analysis of the microstructure of steel after SPD in the central zone of the sample (figure 3) showed that the carbide inclusions in material were uniformly distributed and located at the grain boundaries. Moreover, there was a partial crushing of copper carbides the size of which was about 1.5 µm.

The method of scanning electron microscopy was applied to study the microstructure of modified layer. The analysis of the sample structure in the initial state and after ion nitriding showed the presence of a dark (strongly etching) diffusion zone, representing the α phase – nitrogenous ferrite with a body-centered cubic lattice – the period of which varies depending on the nitrogen content [4]. Nitride emissions, as well as the nitride layer were absent. The transition from the nitried layer to the material base (matrix) was smooth, which is one of the main requirements for the microstructure of nitrided steel.

Figure 4 shows images of the microstructure of a sample of R6M5 steel after SPD and subsequent ion nitriding for 6 hours. The analysis of the images obtained showed that the sample of P6M5 steel after SPD had more developed dark diffusion zone (figure 4b) compared to the initial state (figure 4a). At the same time, the transition from the nitried layer to the base of the material was smooth (figure 4b).

It is known that in the process of ion nitriding the diffusion of nitrogen atoms occurs deep into the surface layers of the material. The dissolution of nitrogen in the solid solution causes elastic distortions of its lattice, which leads to an increased hardness of the hardened layer. In this case, the hardness will
increase if the nitrogen content in the solid solution increases [6]. Microhardness measurements over the depth of the diffusion zone were carried out on the transverse section of the samples. The measurement results are shown in figure 5.

![Microhardness distribution across the depth of the diffusion zone of R6M5 steel samples in the initial state and after SPD.](image)

**Figure 5.** Distribution of microhardness across the depth of the diffusion zone of R6M5 steel samples in the initial state and after SPD.

The analysis of microhardness measurement results showed that the surface microhardness in the sample after SPD and subsequent ion nitriding has increased ~ 1.7 times, and in the sample with no SPD it has increased ~ 2 times. Such an increase in the microhardness was due to the formation of nitride phases with a high nitrogen content in the surface layer [4], which was due to the low diffusion coefficient of nitrogen in steel without SPD. The microhardness distribution across the depth of the diffusion zone of the sample after SPD was smoother (figure 5), which is associated with the appearance of crystal lattice defects in the material structure as a result of plastic deformation and, as a consequence, more favorable conditions for diffusion of nitrogen into the material. The thickness of the diffusion layer in the sample after SPD was ~ 2 times greater than the layer on the sample without prior SPD. The graphs show that ion nitriding at a given temperature does not lead to a decrease in the mechanical properties of base material.

To determine the phase composition formed on the surface of the studied R6M5 steel after nitriding, an X-ray phase study of the sample surfaces was carried out. Table 1 shows the phase composition of the sample surface – R6M5 steel – after nitriding in different states.

| State of material | Surface phase composition |
|-------------------|---------------------------|
| Initial           | α-Fe, W2C, Cr2N, F2N, F3N |
| After SPD         | α-Fe, W2C, Cr2N, F2N      |

The studies of the phase composition of the samples that undergone ion nitriding have shown that the formation of nitride phases of different composition (Fe2N, Cr2N) occurs on the surface of R6M5 steel. The intensity of peaks of the nitride phases is practically independent of the structural state of the
steel. However, there are no phase peaks of tungsten carbide W2C and iron nitride Fe2N in the region of 70º on R6M5 steel after SPD. The absence of W2C reflections is apparently associated with the grinding of carbides after SPD, which is confirmed by the data obtained from the scanning electron microscope (figure 4). The absence of Fe2N is due to the low concentration of nitrogen in the surface layer of steel, because an increased diffusion coefficient leads to the outflow of nitrogen from the surface into the sample material.

4. Conclusion
The purpose of this work is to study the process of ion nitriding in a glow discharge on the structure and mechanical properties of the surface layer of R6M5 steel after SPD.

Based on the results of the study of the influence of ion nitriding process on the structure and mechanical properties of R6M5 steel the following was established:

- after ion nitriding in a glow discharge, a more developed dark diffusion zone is observed in the sample after preliminary SPD;
- as a result of ion nitriding in a glow discharge, a significant increase in the surface microhardness of material is observed: after SPD there was an increase by ~ 1.7, and for the sample with no SPD the increase was ~ 2 as much;
- the microhardness distribution over the depth of the diffusion zone in the sample after SPD is smoother, and the thickness of the diffusion layer in the sample after SPD is ~ 2 times greater than the thickness of the diffusion layer of the sample without SPD;
- ionic nitriding leads to the formation of nitride phases of different composition (Fe2N, Cr2N). The intensity of the peaks of the nitride phases is independent of the structural state of the steel.

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