Effect of ion nitriding by glow discharge on the physical and mechanical properties of the plastically deformed tool steel R6M5

R K Vafin, A V Asylbaev, D V Mamontov, I D Sklizkov, G I Raab, E F Khairetdinov and R S Esipov
Ufa State Aviation Technical University, 12 K. Marksa St., Ufa, 450000, Russia
Email: vafintrk@mail.ru

Abstract. This work is devoted to the study of the effect of the duration of ion nitriding by glow discharge on the physical and mechanical properties of tool steel with different initial structure. We used specimens of R6M5 tool steel with a coarse-grained structure obtained after annealing at a temperature of 850°C and with a fine-grained structure obtained after severe plastic deformation by torsion discharge. With an increase in the duration of ion nitriding, the thickness of the hardened layer and wear resistance increase. The combination of plastic deformation with ion nitriding by glow discharge increases the adsorption and diffusion rate of the saturating element due to the creation of a highly fragmented and disoriented fine-grained structure and contributed to reduction in processing time.

1. Introduction
With the development of engineering and technology, higher and higher demands are placed on the physical and mechanical properties of the materials used. Today, methods of ion-plasma treatment of parts and tools are becoming widespread in machine-building enterprises.

Among similar methods of chemical heat treatment, ion nitriding occupies a special place. This is due to the advantages it offers, such as high values of strength characteristics, corrosion resistance, wear resistance, heat resistance and shorter processing times [1, 2].

It is known that the diffusion of atoms proceeds better in metals with various structural defects. As these defects increase, the rate of diffusion in the metal increases. In addition to this effect on diffusion also affects the grain size of the metal: the smaller the grain, the higher the diffusion rate [3]. Therefore, methods of severe plastic deformation (SPD) have recently become increasingly widespread to increase the diffusion rate in metals. One of these methods is the method of severe plastic deformation by torsion (SPDT). This method of processing consists in mechanical deformation of metal by two simultaneously acting forces, compression and torsion, as a result of which a highly refined grain structure is formed – ultrafine grain structure (UFG) [4, 5].

The purpose of this work was to study the effect of the duration of ion nitriding by glow discharge on the physical and mechanical properties of P6M5 tool steel with different initial structure.

2. Methods of study
The material used in this research was R6M5 tool steel (0.9 C, 0.5 Si, 0.5 Mn, 0.4 Ni, 4.4 Cr, 5.3 Mo, 6.5 W, 2.1 V, 0.5 Co, 0.025 S, 0.03 P; all in wt.%).
SPDT treatment was conducted on the scientific installation "SKRUI-200" ("Nanotech", UGATU). Samples were subjected to 43% cold heading and 1.5 turns of torsion at a hydrostatic pressure of 4 GPa. The treatment scheme is illustrated in figure 1(a).

Figure 1. A schematic illustration of the processes: a-severe plastic deformation by torsion [1], b-ion nitriding in a glow-discharge.

For the experiments we used the modernized installation ELU-5M (laboratory "VIPT", UGATU). Deformed samples were placed on a table inside a vacuum chamber (figure 1(b)).

In the beginning of the experiments, ion cleaning of the sample surfaces was performed in a vacuum chamber for 20 min in an argon atmosphere at a pressure of 10 Pa. The ion nitriding were carried out in a gas mixture from argon, nitrogen and hydrogen (50% Ar + 35% N₂ + 15% H₂) at a gas pressure \( p = 200 \) Pa and a temperature \( T = 450^\circ\text{C} \).

The microhardness of the samples after ion nitriding was investigated on an automatic hardness tester EMCO-Test DuraScan 50. Measurement of microhardness by depth was carried out on oblique sections of the samples (angle 7°) by the Vickers method, at a load of 980.7 mN and the duration of exposure of the diamond indenter for 10 seconds. Tribological tests were carried out on a Nanovea TRB50N high-temperature tribometer, using the "ball-on-disc" method. Tungsten carbide balls with a diameter of 3 mm were used as a counterbody. The tests were performed in air in the dry friction mode with a load of 10 N on the counterbody holder and a rotation speed of 0.1 m/s for 2000 s.

3. Results and discussion
To study the effect of the duration of ion nitriding on the physical and mechanical properties of R6M5 tool steel, two batches of samples were prepared, treated for two and six hours. In each batch, one half of the samples had the original structure, and the other half had the SPDT structure.

Figures 2 show the results of measuring the microhardness of the samples in depth. They demonstrate that with increasing duration of ion nitriding the surface hardness and thickness of the hardened layer of the original sample increases by ~ 1.3 and 2 times, respectively, and that of the SPDT sample practically does not change. At the same time, the surface hardness and thickness of the hardened layer of the SPDT sample ion-nitrided for 2 hours are 1.5 and 2 times higher, respectively, than those of the original sample treated for the same time.

Such results are due to the fact that the SPDT samples after plastic deformation by torsion formed a highly fragmented and disoriented UFG structure [4]. This structure leads to an increase in surface free energy [6], which contributes to an increase in the adsorption of the saturating element and the formation of nitrides in the near-surface layer of the treated material, resulting in an increase in surface hardness. Also, due to an increase in dislocation density and the formation of microdefects and grain
refinement, the rate of diffusion of the saturating element deep into the material increased [3-5], which contributes to an increase in the thickness of the hardened layer. But as the duration of ion nitriding increases, the effect of the UFG structure decreases (figure 2(b)). This is due to the fact that in the first 2 hours of ion nitriding, the surface layer of the material is maximally saturated with nitrogen, and the nitrides formed during subsequent processing prevent further diffusion of nitrogen deep into the material. The values of surface hardness and thickness of the hardened layer in the SPDT sample ion-nitrided for 2 hours coincide with the values in the original sample ion-nitrided for 6 hours. It follows that the use of SPDT treatment before ion nitriding reduces the duration of treatment by a factor of 3 times, which is the fact of increasing the efficiency of ion nitriding. This result is confirmed by the results of tribological tests (figure 3, table 1).

![Figure 2](image2.png)

**Figure 2.** Microhardness distribution by depth of samples from R6M5 after ion nitriding during 2 and 6 h, with different structures: a-R6M5 original, b-R6M5 SPDT.

| Weight loss, µg | R6M5 (original) | R6M5 (SPDT) |
|----------------|-----------------|-------------|
| Samples        | Counterbody     | Samples     | Counterbody |
| 28             | 8               | 30          | 3           |

**Table 1.** Weight loss of samples and counterbodies after tribological tests.

![Figure 3](image3.png)

**Figure 3.** Friction coefficient of P6M5 samples with different structures ion-nitrided at 2 and 6 h.
Figure 3 clearly shows that the friction coefficient curves are almost identical. But at the same time, the wear of the counterbody used in the test of the original sample is much (2.5 times) greater than that of the counterbody for the SPDT sample (table 1). This is due to the fact that to the abrasive wear mechanism of the original sample observed in both samples, an adhesion component is added, due to which the counterbody material is transferred to the surface of the sample [7, 8].

4. Conclusions
The study established the following:

- The use of severe plastic deformation before ion nitriding contributes to an increase in surface hardness by 1.5 times and in the thickness of the hardened layer by 2 times, due to the formed highly fragmented and disoriented ultrafine grain structure.
- The use of severe plastic deformation before ion nitriding contributes to reducing the processing time by 3 times, due to an increase in the rate of diffusion of the saturating element deep into the material.

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