Ion nitriding of martensitic and austenitic steels after SPD at different temperatures

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Abstract. The article investigates the influence of ultrafine-grained structure of martensitic and austenitic steels on diffusion processes at various temperature regimes of ion nitriding. Ion nitriding at various temperatures has been proven to lead to a change in hardness of the strengthened layer and main material. The wear tracks and dependences of a friction coefficient on wear duration of the samples have been analyzed. The dependence of loss in weight of samples on temperature of ion nitriding has been defined.

1. Introduction
Today a steady tendency to develop new constructional materials with high mechanical properties and technological parameters is being observed in machine engineering industry [1, 2]. One of the promising directions to increase strength of constructional materials is severe plastic deformation (SPD). SPD methods allow manufacturing materials with ultrafine-grained (UFG) structure with an average size of structural components of at least 1 µm and mainly high-angle grain boundaries [1, 3]. Parts of machine engineering made of materials with UFG structure are characterized by high fatigue strength, strength-to-weight ratio at operating temperatures (to 450 ºC) [3].

Despite high physical and mechanical properties of constructional steels with UFG structures, the surfaces of machine parts and mechanical devices operating in conditions of contact loads are subject to intensive wear. Thermo-chemical treatment, in particular nitriding in glow discharge (ion nitriding), is widely used to increase operating characteristics of surface layers of machine parts [4-6]. However, traditional ion nitriding of constructional steels is conducted at high temperatures (550-650 ºC) at long-term holding (to 36 hours).

It is known [6] that prolonged exposure to high temperatures leads to degradation of UFG structure and, therefore, decreases physical and mechanical properties of the material base. Consequently, ion nitriding should be performed at low temperatures (350-450 ºC) to prevent growth of structural components and to maintain operating characteristics of steel.

This article aims at investigation of mechanical and operating properties of austenitic and martensitic steels with UFG structures after ion nitriding in glow discharge.

2. Experiment
Martensitic steel [0.10-0.16 C; 10.5-12.0 Cr; 1.5-1.8 Ni; 0.18-0.30 V; 0.35-0.50 Mo; 1.60-2.0 W] after quenching from 1050 ºC and tempering at 800 ºC and austenitic steel [0.12 C; 17-19 Cr; 9-11 Ni; 0.8 Ti] after quenching from 1050 ºC were subjected to ion nitriding.
The UFG structure of the material was achieved by high-pressure torsion (HPT) at a temperature of 300 °C under a pressure of \( P = 6 \) GPa, with a number of turns \( n = 10 \). Before HPT processing the samples were in the form of disks with a diameter of 20 mm and thickness of 0.5 mm.

Low-temperature ion nitriding was performed on the modified unit ELU-5M designed to conduct processes of vacuum heat and thermo-chemical treatment [4]. The treatment period was 6 hours at temperatures of 450, 500 and 550 °C in the nitrogen-containing mixture of gases of nitrogen, argon and hydrogen (\( N_2 30\% \), \( Ar 55\% \), \( H_2 15\% \)) under a pressure of 150 Pa in the working chamber.

The microhardness was measured by the standard procedure using the microhardness tester Struers Duramin 1. The value of static load applied to the diamond indenter was 980.7 mN (100 g) during 10 seconds.

The tribotechnical tests of the sample surface were conducted by the method of “Ball on flat” on the high-temperature tribometer Nanovea at the dry friction regime. A ball made of steel ShKh-15 with a diameter of Ø3 mm was used as a counterbody at a normal load of 4 N applied to the sample surface. The motion rate of the ball along the circular trajectory with a radius of 2.5 mm was 9450 mm/min.

3. Results and discussion
Figure 1 shows the graphs of microhardness distribution along the depth of austenitic steel (figure 1a) and EI961 steel (рис. 1b) samples after ion nitriding at various temperatures.

![Figure 1. Microhardness distribution along the depth of austenitic (a) and martensitic (b) steels samples after ion nitriding at various temperatures.](image)

The analysis of the results of microhardness measurement illustrated that when the distance from the surface increased there was a gradual decline of hardness to the value of hardness of the main material, which was caused by the decrease in the dissolved nitrogen concentration in \( \alpha \)-phase for martensitic steel and in \( \gamma \)-phase for austenitic steel [6]. The thickness of the strengthened layer of austenitic steel samples after nitriding at temperatures of 450, 500 and 550°C was ~40, 50 and 60 µm. When the temperature of nitriding increased, both the hardness values of the strengthened layer and the main material decreased as recrystallization processes started to take place in the steel [8]. The microhardness values of the base of austenitic steel samples after nitriding at temperatures of 450, 500 and 550°C were, correspondingly, 630, 520 and 510 HV. The thickness of the strengthened layer of the martensitic steel samples after nitriding at temperatures of 450, 500 and 550°C increased and was 155, 160 and 170 µm, respectively. An increase in the nitriding temperature led to the growth of both hardness of the strengthened layer and main material. This was due to precipitation of dispersed...
particles of carbides, which were a strengthening phase [9]. An increase in the treatment temperature led to the growth in the number of carbides. Therefore, the microhardness of the base of steel samples after nitriding at temperatures of 450, 500 and 550°C was 620, 635 and 650 HV, correspondingly.

The wear tests of the samples were conducted with the aim to study the impact of the ion nitriding temperature on the operating properties. Figures 2-7 show the optical images of wear tracks and dependence of the friction coefficient change in the austenitic and martensitic steels samples with UFG structures after ion nitriding at various temperatures.

**Figure 2.** Optical image of the wear track (a) and the dependence of the wear coefficient on the test duration (b) of the austenitic steel sample with UFG structure after ion nitriding at $T=450°C$.

The analysis of the images of the wear tracks in the austenitic steel samples with UFG structures after the tests demonstrated that the main mechanism of surface wear was adhesive wear with traces of micro-cutting of friction surfaces on the sample subjected to nitriding at a temperature of 450°C (figure 2a). At the same time there was plastic flow of metal as lapping.

**Figure 3.** Optical image of the wear track (a) and the dependence of the wear coefficient on the test duration (b) of the austenitic steel sample with UFG structure after ion nitriding at $T=500°C$.

The results of the analysis of the graph of the friction coefficient dependence on the wear duration of the samples (figure 2b) illustrated that the duration of the running-in stage $t$ was at least 1 min.
stage of normal wear was the longest one. At this stage of testing from ~1 min to 10 min a gradual
increase in the friction coefficient \( K_F \) from 0.4 to 0.7 was observed. After the expiry of 10 min of wear
the friction coefficient \( K_F \) destabilized gradually and set at the value of ~0.8 during the remaining
period of the test.

The images of wear tracks of the austenitic steel sample subjected to ion nitriding at a temperature
of \( T=500^\circ C \) (figure 3a) show that the main mechanism of wear is adhesive wear. Smooth micro
cuttings on the wear track prove this fact [10]. The analysis of the graph of the friction coefficient
dependence on the wear duration (figure 3b) demonstrated that the duration of the running-in stage
was at least 1 min. At the stage of normal wear during the period of test from 1 to 10 min there was a
gradual increase of the friction coefficient from 0.55 to 0.75. During the period from 10 to 30 min a
gradual growth of the friction coefficient from 0.75 to 0.8 was observed. After 30 minutes of the test
there was a destabilization of the friction coefficient characterized by its value varying in the range
from 0.6 to 1.0.

**Figure 4.** Optical image of the wear track (a) and the dependence of the wear coefficient on the test
duration (b) of the austenitic steel sample with UFG structure after ion nitriding at \( T=550^\circ C \).

**Figure 5.** Optical image of the wear track (a) and the dependence of the wear coefficient on the
test duration (b) of the martensitic steel sample with UFG structure after ion nitriding at \( T=450^\circ C \).
The images of the wear tracks of the austenitic steel sample subjected to nitriding at $T=550^\circ C$ (figure 4a) illustrated that the main mechanism of wear was adhesive and abrasive wear [11, 12]. Tear-outs of the material, plastic deformation and smooth micro-cuttings on the wear track proved this fact. The analysis of the graph of the friction coefficient dependence on the wear duration (figure 4b) showed that the duration of the running-in stage was at least 1 min. The friction coefficient was 0.45 up to 5 min of testing. After 5 min of testing a gradual growth of the friction coefficient to its defined value of 0.65 was observed within 10 min of testing. Then there was a gradual increase in instability of the friction coefficient.

Figure 5 demonstrates the optical image of the wear tracks and the dependence of the friction coefficient on the wear duration of the surface of austenitic martensitic steel.

The analysis of the wear tracks images of the martensitic steel samples with UFG structures after testing showed that the main mechanism of wear of the surface was abrasive wear on the sample subjected to nitriding at $T=450^\circ C$ (figure 5a). At the same time there was no plastic flow of metal as lapping. The results of the analysis of the graph of the friction coefficient dependence on the duration of wear of the samples (figure 5b) demonstrated that the duration of the running-in stage was at least 1 min. At the stage of normal wear there was practically a constant average value of the friction coefficient $K_F=0.9$ during the whole testing period. It is seen that the friction coefficient varied in the range of 0.6–1.2.

Figure 6. Optical image of the wear track (a) and the dependence of the wear coefficient on the test duration (b) of the martensitic steel sample with UFG structure after ion nitriding at $T=500^\circ C$.

The images of wear tracks of the martensitic steel sample with UFG structure subjected to nitriding at $T=500^\circ C$ (figure 6a) showed that the main mechanism of wear was abrasive wear. This was proved by long micro-cuttings on the wear track. The analysis of the graph of the friction coefficient dependence on the wear duration (figure 6b) demonstrated that the duration of the running-in stage was at least 1 min. At the stage of normal wear the average value of wear coefficient of 0.8 was practically constant during the whole test. At the same time the range of values of wear coefficient from minimum to maximum was 0.7–0.9.

The image of the wear track of the martensitic steel sample with UFG structure subjected to nitriding at $T=550^\circ C$ (figure 7a) shows that the main mechanism of wear was abrasive wear. Deep long and minor micro-cuttings were defined on the wear track. The analysis of the graph of the friction coefficient dependence on the wear duration (figure 7b) demonstrated that the duration of running-in stage was at least 1 min. At the stage of normal wear a practically constant value of wear coefficient of ~0.9 was observed during the whole time of the test. The range of the wear coefficient from the minimum to maximum values was 0.7-1.1.
Figure 7. Optical image of the wear track (a) and the dependence of the wear coefficient on the test duration (b) of the martensitic steel sample with UFG structure after ion nitriding at $T=550^\circ$C.

Figure 8 shows the diagrams of loss in weight of the samples after wear resistance tests depending on the temperature of ion nitriding. The analysis of the obtained data has defined that loss in weight of martensitic steel with UFG structure after nitriding at temperatures of 450, 500 and 550$^\circ$C was 11.983, 12.792 and 12.274 $\mu$g, correspondingly. The loss in weight of martensitic steel with UFG structure after nitriding at temperatures of 450, 500 and 550$^\circ$C was 12.748, 11.863 and 10.137 $\mu$g, correspondingly. An increase in the weight loss of the austenitic steel sample with UFG structure after nitriding at 500$^\circ$C is connected with the beginning of recrystallization process and decrease in hardness of the base of the material, as illustrated by the results of microhardness measurements by the depth (figure 1a). The diagram (figure 8b) shows that an increase in temperature of ion nitriding of the martensitic steel samples leads to a growth of wear resistance due to the increase in the hardness of both the main material and the strengthened layer.

Figure 8. Loss in weight of the samples after wear resistance tests depending on the temperature of ion nitriding: (a) austenitic steel, (b) martensitic steel.

4. Conclusion
The investigation of mechanical and operating properties of the surface of martensitic and austenitic steels with UFG structure after ion nitriding at various temperatures has defined that
– when the holding temperature increases from 450 to 550°C, the thickness of the strengthened layer on the martensitic steel rises from 155 to 170 µm, and decreases from 60 to 40 µm on the austenitic steel. The decrease in the thickness of the modified layer is linked with degradation of the UFG structure of the austenitic steel at nitriding over ~450°C.

– when the holding temperature rises from 450 to 550°C, the hardness of the base of the martensitic steel increases from 800 to 850 HV, and the hardness of the base increases from 620 to 650 HV, precipitation of strengthening finely dispersed carbide phases occurs. The surface microhardness of the austenitic steel decreases from 770 to 580 HV, as there is a process of structure recrystallization.

References
[1] Akhmadeev N A, Valiev R Z, Kopylov V I and Mulyukov R R 1992 Metals No. 5 96
[2] Gromov V E, Gorbunov S V, Ivanov, Y F, Vorobiev S V and Konovalov S V 2011 Journal of Surface Investigation 5 974
[3] Ivanisenko Y, et al. 2003 Acta Mater. 51 5555
[4] Budilov V V, Ramazanov K N, Khusainov Yu G, Zolotov I V and Babenko N S 2015 Journal of Physics: Conference Series 652 012052
[5] Arzamasov B N, Vinogradov A V, Mulyakaev L M, Burdonskiy S I 1978 Bulletin of mechanical engineering No. 7 67
[6] Arzamasov B N 1979 Chemical and thermal treatment of metals in activated gas environments (M.: Mashinostroenie ) p 230
[7] Fang Y, Chen X, Madigan B, Cao H and Konovalov S 2016 Fusion Engineering and Design 103 21
[8] Yamashita A, Yamaguchi D, Horita Z and Langdon T G 2000 Mater Sci Eng A 287 100
[9] Valiev R Z, Korznikov A V and Mulyukov R R 1992 PhMM No. 4 70
[10] Budilov V V, Ramazanov K N, Khusainov Y G and Zolotov I V 2015 Vestnik UGATU No. 2 1
[11] Ivanov Y F, Koval N N, Gorbunov S V, Vorobyov S V, Konovalov S V and Gromov V E 2011 Russian Physics Journal 54 575
[12] Gerasimov S A, Kuksenova L I, Lapteva V G 2012 Structure and wear resistance of nitrided constructive steels and alloys (M.: MSTU by N.E. Bauman) 518 p