The Effect of Nitriding at Low Temperatures on Tribological and Magnetic Properties of Austenitic Stainless Steel

**M. Yu. Smolyakova[a], D. S. Vershinin[a], Yu. R. Kolobov[a], S. V. Chernikov[a],
O. V. Stognei[b], and I. M. Tregubov[b]**

[a] *Research and Education and Innovation Center Nanostructured Materials and Nanotechnology,
Belgorod State University, Belgorod, Russia*
[b] *Voronezh State Technical University, Voronezh, Russia*
*e-mail: SmolaykovaMarina@mail.ru, Vershinin@bsu.edu.ru, Kolobov@bsu.edu.ru,
Chernikov@bsu.edu.ru, sto@sci.vrn.ru, ilia.tregybov@mail.ru*

Abstract—The change of the structural and phase composition of the austenitic steel 12Cr18Ni10Ti is studied after nitriding in the plasma of a non-self-sustained low-pressure arc discharge at different temperatures. It is shown that, as a result of ion nitriding, it is possible to increase the microhardness and wear resistance of the investigated steel. The basic parameters of the nitriding process (temperature and composition of the gas mixture) that make it possible to improve the mechanical properties without significant changes in magnetic characteristics are selected.

Keywords: low-temperature nitriding, stainless steel, wear resistance, magnetic properties.

INTRODUCTION

It is known that nonmagnetic stainless instrument steel 12Cr18Ni10T has high corrosion resistance, ductility, and impact hardness and good fusability. This steel, in particular, has been widely used in medicine and the food industry for the manufacture of parts of structures operating in contact with aggressive media (solutions of acids, alkalis, and salts) [1, 2]. Such properties have been achieved through the optimal ratio of alloying components. However, for effective use in friction joints, this steel has insufficient wear resistance.

One of the ways to improve the properties of surface and subsurface layers of materials is the low-temperature plasma nitriding of a non-self-sustained low-pressure arc discharge. This method of nitriding has several advantages over gas nitriding and nitriding in a glow discharge [3]. Nitriding technologies based on the use of a low-pressure arc discharge and, therefore, characterized by a low content of residual oxygen in the chamber are implemented without the use of ammonia or mixtures of nitrogen and hydrogen; i.e., the process is carried out in pure nitrogen. Along with high current density characteristic of an arc discharge, it provides sufficient sputtering and high-quality cleaning of the processed surface, intensification of diffusion processes, reduction of the time of nitriding, and ecological cleanness of technologies.

It is known that there are always small amounts of nitrogen in steels. Its content depends on the method of melting and chemical composition. At the same time, the special introduction of nitrogen into austenitic steel leads to several problems such as the formation of nitrides of alloying elements (chromium, titanium, etc.) owing to the high affinity of nitrogen to these elements. For example, it is known that an increase in hardness and wear resistance occurs as a result of the formation of chromium nitrides [4–6]. However, there is depletion of the solid solution with respect to chromium, which leads to a decrease in the corrosion resistance and a change in magnetic properties owing to the appearance of the ferrite phase.

This paper aims to study of the effect of formation of nitrides of alloying elements, particularly chromium nitride, at low-temperature nitriding in the plasma of a non-self-sustained low pressure arc discharge on the structure and properties of surface and subsurface modified layers of the austenitic steel 12Cr18Ni10Ti.

EXPERIMENTAL

The study was conducted on the austenitic tool steel 12Cr18Ni10Ti (the chemical composition is presented in Table 1). Samples with a height 6 mm were cut from a rod with a diameter of 20 mm and then sub-

Table 1. Chemical composition of steel 12Cr18Ni10Ti, wt % (GOST 5632-72)

C	Si	Mn	Cr	Ni	Ti	Fe	S	P	Cu
≤0.12	≤0.8	≤2.0	17.0–19.0	9.0–11.0	5C–0.8	base	≤0.020	≤0.035	≤0.30

jected to grinding and polishing with diamond pastes to a mirror shine.

Nitriding was performed with a modernized ion-plasma installation NNV-6.6-I1 in the plasma of a non-self-sustained low pressure arc discharge at different temperatures (460, 480, 500, and 530°C) for 60 min. The vacuum chamber was evacuated by an N-250 oil-diffusion pump to the limiting residual pressure (3–5) × 10^{-5} Torr ((4–6) × 10^{-3} Pa). Then the vacuum chamber was filled with a nitrogen–argon gas mixture using PINK plasma generators [7]. The percentage of gases varied depending on the desired phase composition of the modified layer and, consequently, its properties.

The study of changes in the structure of surface and subsurface layers after saturation was carried out on cross sections. In order to identify the structure of nitrided layers, cross sections were subjected to electrochemical etching in a saturated solution of oxalic acid. The change in surface morphology and structure of the modified layers of the 12Cr18Ni10Ti steel after the ion-plasma treatment were investigated using an Olympus GX 71 optical microscope and a Quanta 600 FEG scanning electron microscope. Measurements of microhardness on the surface and on the cross section of samples were performed using a DM-8B automatic microhardness tester (Affri) with a diamond pyramid under a load on the indenter of, respectively, 0.49 and 0.01 N in accordance with GOST State Standard 9450-76. The measurement error did not exceed 3%.

In order to determine the phase composition of modified layers, an ARLX'TRA X-ray diffractometer (Switzerland) using CuK_α radiation was used. The X-ray survey of the steel sample in its initial condition was performed in Bragg-Brentano geometry, and after nitriding, in the mode of moving beam with a step of 0.02°.

The magnetic characteristics of the austenitic steel before and after nitriding were determined by a vibrating magnetometer. It is known that the values of parameters of remagnetization of magnetics depend on many factors, including the shape and weight of the sample. Therefore, for magnetic measurements, samples of the same shape and weight were prepared, while the employed configuration of measurements minimized the demagnetization factor.

In order to investigate tribological characteristics, an automated friction and wear machine was used (high-temperature tribometer, CSM Instruments, Switzerland) for the "ball-on-disc" testing scheme [8]. A corundum ball with a diameter of 6 mm and microhardness of 19 GPa was used as a static friction partner. Tests were carried out in air in the mode of dry friction under a load of 4 N on the counterface holder and the speed of rotation of the sample of 10 cm/s. In the process, the sliding distance was 2000 m. In order to ensure equal conditions of testing of samples, a constant temperature of 30°C was maintained. Tests were in compliance with international standards ASTM G99-959 and DIN50324. The evaluation of wear resistance of samples and the static friction partner was carried out by the wear factor and tear which was determined after the tests.

The cross section of the wear track of the sample after tribological testing in five areas at three points each was measured using a SURTRONIC precision contact profilometer. On the basis of these data, the average value of the cross-sectional area was determined. The analysis of the spot of wear on the static partner (the ball) was performed using the Olympus GX 71 optical microscope.

RESULTS AND DISCUSSION

When selecting the optimal nitriding mode of the metal, the influence of such process parameters as temperature, duration of treatment, and gas mixture composition should be taken into account. In the present study, the effect of temperature of the treatment on the structure and properties of 12Cr18Ni10Ti steel was studied. Preliminary experiments on nitriding of austenitic steel showed that the optimum percentage of nitrogen/argon gases is 50%/50%. Hence, further research was conducted in the gas mixture of 50% Ar + 50% N_2, but at different nitriding temperatures, i.e., 460, 480, 500, and 530°C.

As a result of the low-temperature nitriding of steel, it was possible to significantly increase the surface microhardness at all studied temperatures of the treatment process (Fig. 1). The nitriding at 460°C leads to an increase in the microhardness by a factor of two and the formation of a modified layer with a thickness of 5 μm (Fig. 2a). Raising the temperature by only 20°C makes it possible to change the surface microhardness by a factor of four (from 3 to 12 GPa) and form a nitrided layer with the depth of 8 μm (Fig. 2b).

A further increase in the nitriding temperature to 500°C does not lead to significant changes in the surface microhardness compared with the value obtained for 480°C. The length of the modified layer in this case is 12 μm (Fig. 2c). Conducting the process at a tem-

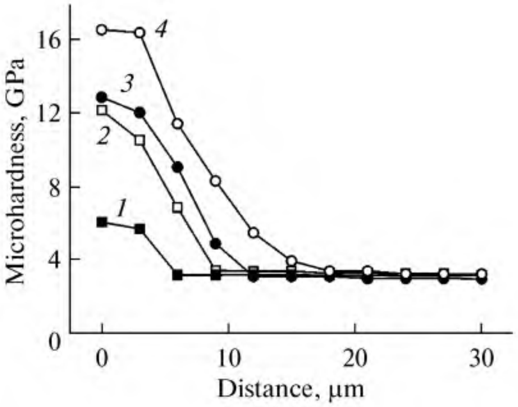

Fig. 1. Distribution of microhardness in cross sections from the surface to the base of the material of samples nitrided at temperatures of 460°C (*1*), 480°C (*2*), 500°C (*3*), and 530°C (*4*) (on the vertical axis at the point "0" values of microhardness measured on the sample surface are given).

perature of 530°C gives the maximum values of the surface microhardness of 16.4 GPa and the thickness of the nitrided layer of 15 μm. The data on the thickness of modified layers are consistent with the data of measurement of the distribution of microhardness in cross sections (Fig. 1).

The analysis of X-ray diffraction patterns showed that the low nitride phase γ'-Fe_4N is formed in the subsurface layers regardless of the temperature of the nitriding process. The modification of the surface at a temperature of 460°C does not lead to the formation of chromium nitride (Fig. 3a), while after treatment at 480°C a small amount of this phase can be observed (Fig. 3b). A further increase in the treatment temperature leads to an increase in the proportion of chromium nitride in the nitrided layer. The assumption on the phase transition of austenite to ferrite using X-ray diffraction analysis was not confirmed because of the small content of the ferrite phase.

In the next phase of research, tribological tests of the investigated material in the initial state were carried out when the load on the holder of the counterface was 4 N. They were stopped after 80 min and a friction path of 478 m due to a catastrophic wear of both the sample and the counterface. In this case, the wear factor of the sample was 6.60×10^{-4} $mm^3/(N\ m)$ and for the the static friction partner it was 3.17×10^{-5} $mm^3/(N\ m)$ (Fig. 4) The analysis of images of the friction track in this case (Fig. 5a) showed that the main mechanism of wear was the wear with traces of plastic flow of material. This is confirmed by the formation of characteristic grooves and sag on the friction surface, occurring in the plastic edging of the material owing to the impact of abrasives.

The nitriding process can significantly increase the wear resistance of the samples. In the presented histogram (Fig. 4), it can be seen that the smallest dimension factor of wear is observed after nitriding at temperatures of 500 and 530°C. This improvement in wear resistance is due not only to the formation of a nitride layer but also to the formation of chromium nitride in

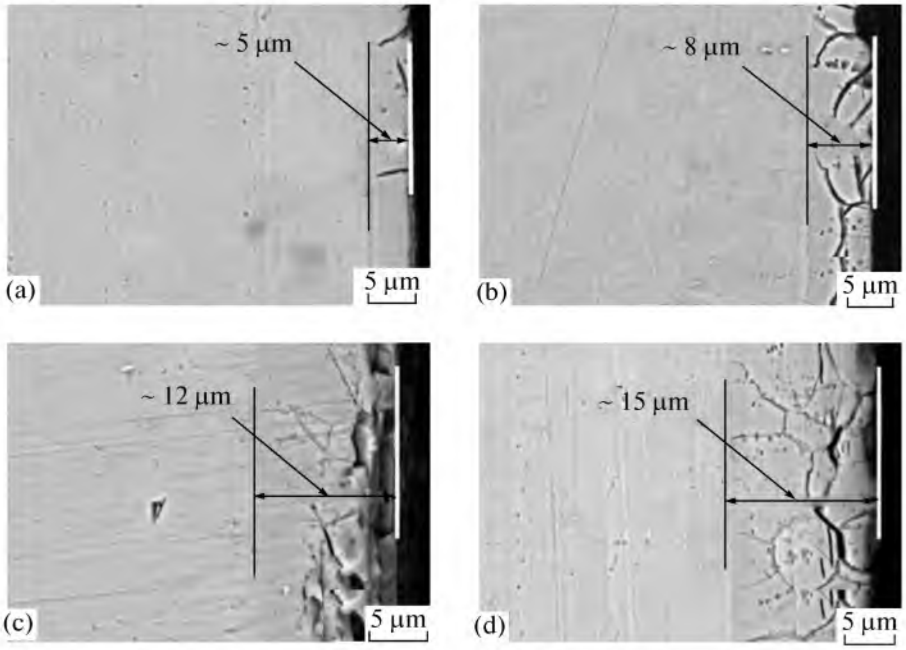

Fig. 2. Structure of the modified layers of the austenitic steel after nitriding at different temperatures, °C: 460 (a), 480 (b), 500 (c), and 530 (d).

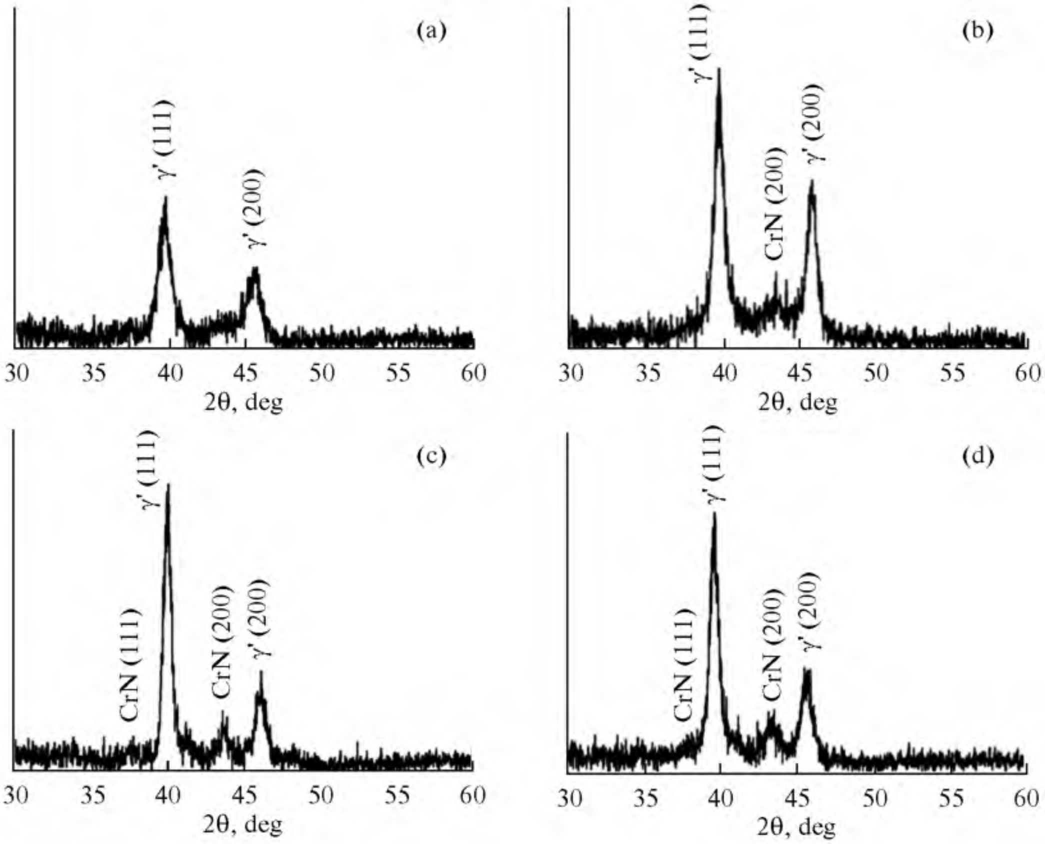

Fig. 3. X-ray diffractograms of 12Cr18Ni10Ti steel after low-temperature nitriding at different temperatures, °C: 460 (a), 480 (b), 500 (c), and 530 (d).

it, which, as is known, in addition to a significant increase in hardness also contributes to the increased wear resistance [5, 6]. It should also be noted that nitriding at temperatures below 500°C does not lead to a significant increase in wear compared with samples at ≥500°C (Fig. 4). The investigation of the track after friction tests showed that adhesive wear is added to the abrasive component of the wear mechanism observed for untreated samples. This is evident from scratches on the surface of the friction track and the transfer of material of the sample on the static friction partner (Fig. 5b). As is commonly known, there are three stages of the friction process [9]. The initial period of wear, which is short, is characterized by a nonstationary state of the tribosystem. This stage is called the breaking in of the tribosystem, during which an equilibrium roughness and constant coefficient of friction are established, while the wear is reduced. On the graph of the dependence of the coefficient of friction on the friction path, it can be seen that the breaking-in phase ends at a distance of 500 m (Fig. 6). Then comes the second stage of wear, characterized by a stable friction coefficient and relatively low wear.

In the course of its development, the wear gradually increases and leads to significant damage and catastrophic wear. It should be noted that all the nitrided samples under these test conditions did not reach the stage of destruction, and the coefficient of friction remained stable. The increase in temperature of the process did not result in a significant change in the

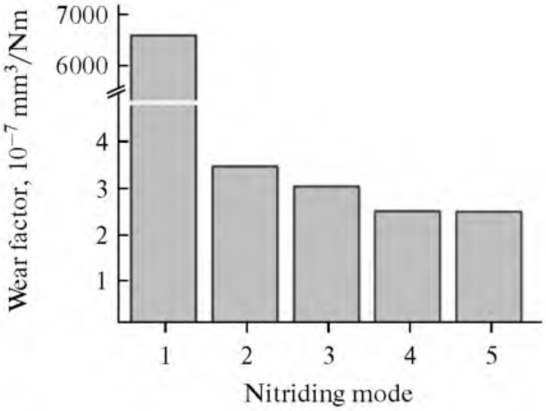

Fig. 4. Distribution of values of the wear factor of 12Cr18Ni10Ti steel before and after nitriding at different temperatures, °C: untreated (*1*), 460 (*2*), 480 (*3*), 500 (*4*), and 530 (*5*).

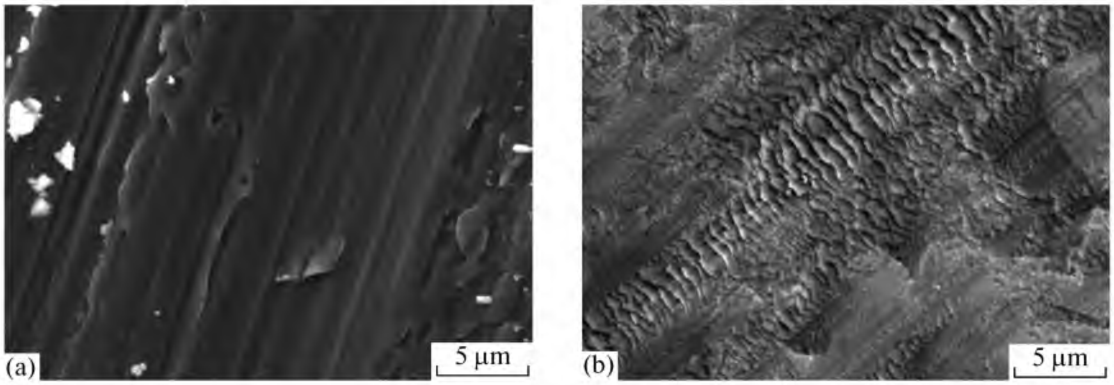

Fig. 5. Image of the friction track after the test on wear resistance in the friction pair steel–corundum: without treatment (a), after nitriding at 480°C (b).

coefficient of friction (Table 2); however, in comparison with the value for the untreated steel, it decreased.

As a result of the processing of samples of the austenitic steel in the surface modified layers, a change in the magnetic properties can occur. In this regard, measurements of the magnetic characteristics of the samples in the initial state and subjected to nitriding at different temperatures were carried out. The analysis showed that the magnetization curve of the untreated sample is typical for a ferromagnetic material, namely, there is a magnetic hysteresis, and the curve goes to the area of technical saturation in fields of 4 kOe (Fig. 7). After the treatment of samples, the parameters of the magnetization curves change. The degree of increase in saturation magnetization with respect to the value obtained for the initial sample increases with increasing nitriding temperature and reaches a maximum value after treatment at 500°C. Apparently, the observed changes in magnetic properties after nitriding are associated with the change in the phase composition in the modified layer, namely, with the appearance of the ferrite phase. It should also be noted that conducting the process of nitriding at temperatures of 460 and 480°C makes it possible not only to preserve low values of saturation magnetization but also to reduce the coercive force by about a factor of two relative to the state prior to treatment.

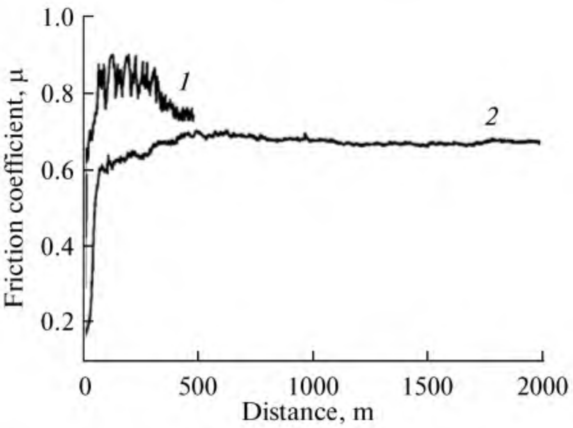

Fig. 6. Dependence of the coefficient of friction on the distance for steel before (*1*) and after (*2*) nitriding at 480°C.

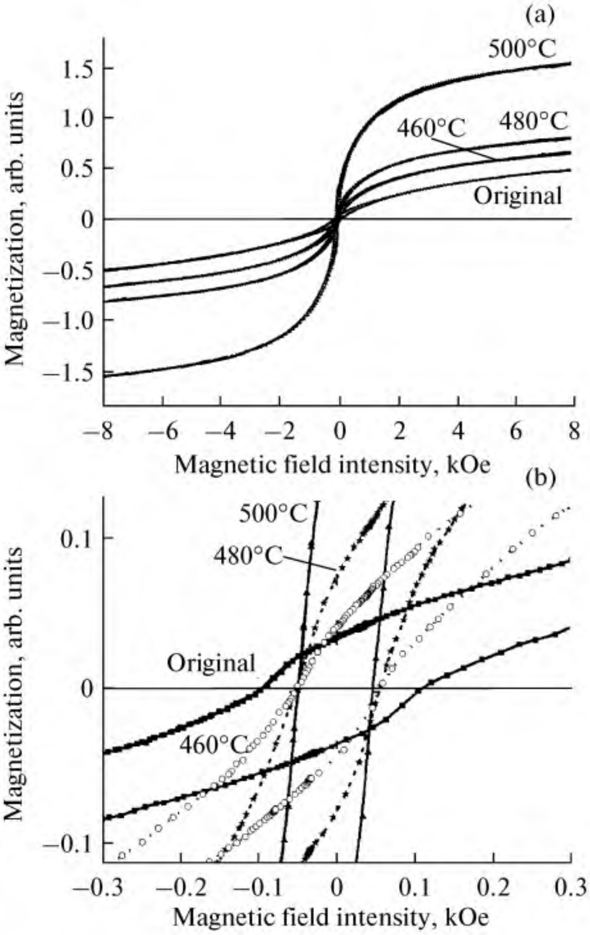

Fig. 7. Switching curve of the 12Cr18Ni10Ti steel in the initial state and after nitriding at different temperatures (a). The hysteresis loops of the samples (b).

Table 2. Dependence of the coefficient of friction on the temperature of the nitriding process

	Nitriding temperature, °C				
	without treatment	460	480	500	530
Friction coefficient	0.79	0.67	0.66	0.66	0.68

CONCLUSIONS

The structural-phase composition and tribological and magnetic properties of austenitic steel 12Cr18Ni10Ti were studied after low temperature nitriding in the plasma of a non-self-sustaining low pressure arc discharge. It was shown that the low-temperature nitriding makes it possible to increase the surface microhardness by more than a factor of five (from 3 to 16.4 GPa). It was found that increasing the nitriding temperature from 460 to 530°C leads to the increase in the level of chromium nitride in the modified layer with a simultaneous increase in the value of the magnetization of the material. The change in the magnetic characteristics can occur owing to the formation of the ferrite phase. On the basis of these data, it was revealed that, in order to improve the properties of austenitic steel 12Cr18Ni10Ti, it is necessary to carry out the process of nitriding in the plasma of a non-self-sustaining arc discharge of low pressure at a temperature of 480°C in a gas mixture of 50% N_2 + 50% Ar. This mode leads to the optimal combination of physical and mechanical properties of the investigated steel, as a result of which the surface microhardness increases to 12.1 GPa and the thickness of the nitrided layer is 8 mm. The wear rate decreases by three orders of magnitude from 6.60×10^{-4} to 3.05×10^{-7} and the magnetic properties vary slightly.

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