Structure and properties of high-chromium steel irradiated with a pulsed electron beam and nitrided in a low-pressure gas discharge plasma

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Abstract. Ion-plasma saturation of the surface of machine parts and mechanisms with gas elements (nitrogen, oxygen, carbon) is currently one of the most effective and widely used methods of surface hardening of metal products for various purposes in the industry of developed countries. The aim of this research is to develop a complex method for modifying the surface layer of AISI 310 steel, combining irradiation with an intense pulsed electron beam and subsequent nitriding in the plasma of a low-pressure gas discharge. As a result of the studies performed, the optimal parameters of modification were revealed, which make it possible to increase the hardness of the surface layer of steel by more than 11 times, relative to the hardness of the initial material, and 8 times, relative to the hardness of steel irradiated with a pulsed electron beam. In this case, the wear resistance of the steel exceeds the wear resistance of the original and irradiated material by more than 100 times. It has been established that the high strength and tribological properties of the modified steel are due to the formation of a two-phase (iron nitride and chromium nitride) layered nanoscale structure in the surface layer.

1. Introduction

Chemical heat treatment (CHT) is widely used in the industry of the overwhelming majority of developed countries to modify the structure and properties of the surface of metals and alloys [1]. One of the most effective and widespread methods of surface hardening of metal products by CHT methods is ion-plasma saturation of the surface of machine parts and mechanisms with gaseous elements (nitrogen, oxygen, carbon, as well as their various combinations) [2]. The processes of complex modification of the surface of solids are less studied. A number of works have shown that the combination in various sequences of the action of concentrated energy flows on the surface of the metal and alloys (laser beams, plasma flows, pulsed and continuous electron beams, etc.) and ion-plasma saturation with gas elements leads to a significant increase in the service characteristics of material [3-6]. The aim of this research is to develop a complex method for modifying the surface layer of AISI 310 steel, combining irradiation with an intense pulsed electron beam and subsequent nitriding in the plasma of a low-pressure gas discharge.
2. Material and methods

High-chromium AISI 310 steel was used as the material for the study. The surface of the steel specimens was irradiated on a SOLO electron-beam installation with an electron source based on a low-pressure pulsed arc discharge with grid stabilization of the cathode plasma boundary and an open anode plasma boundary [7]. Irradiation was carried out with the following parameters: the energy of accelerated electrons \( eV = 18 \text{ keV} \); electron beam energy density \( E_s (\text{J/cm}^2) = 10, 20 \) and \( 30 \); pulse duration \( \tau (\mu s) = 50 \) and \( 200 \); number of pulses \( N = 3 \); pulse repetition rate \( f = 0.3 \text{s}^{-1} \); gas pressure (argon) in the working chamber of the installation \( \approx 0.02 \text{ Pa} \). Steel nitriding was carried out on a modernized installation of NNV-6.6-II type, equipped with a PINK plasma generator [8]. Nitriding temperature (K) was \( 723, 793 \) and \( 873 \), nitriding time \( -1, 3, 5 \) hours. The structure and phase composition of the material was studied by scanning and transmission diffraction electron microscopy and X-ray diffraction analysis. The properties of the modified layer were characterized by microhardness and wear resistance.

3. Results and discussion

It has been established that AISI 310 steel in the initial state is a polycrystalline material. Globular particles of the second phase (chromium and iron carbides) of submicron sizes are located in the bulk and along the grain boundaries. It is shown that the electron-beam treatment of steel leads to the dissolution of globular particles of the carbide phase and the formation of a cellular crystallization structure [9]. Along the boundaries of the cells, nanosized (\( \approx 25 \text{ nm} \)) particles of chromium and iron carbides, which stabilize the defective substructure of the material, are located. Subsequent nitriding of steel is accompanied by the formation of a surface layer, the hardness and wear resistance of which substantially depend on the parameters of combined processing. It was found that the steel specimens possessing the highest tribological properties were complex treatment at the following parameters: irradiation with a pulsed electron beam of \( 30 \text{ J/cm}^2, 200 \mu s, 3 \) pulses; subsequent nitriding at a temperature of \( 793 \text{ K} \) for \( 3 \) hours. In this case, the wear resistance of the steel exceeds the wear resistance of the original and irradiated material by more than \( 100 \) times. The hardness of the surface layer of steel modified with these parameters exceeds the hardness of the initial material by more than \( 11 \) times and \( 8 \) times the hardness of steel irradiated with a pulsed electron beam. The thickness of the hardened layer reaches \( 40 \mu m \).

It is obvious that such significant differences in the strength and tribological properties of steel in the initial state and after modification by the combined method (irradiation with \( 30 \text{ J/cm}^2, 200 \mu s, 3 \) pulses + nitriding at \( 793 \text{ K}, 3 \) hours) are due to the transformation of the structural-phase state of the surface layer. Studies of the phase composition of the modified steel, performed by X-ray diffraction analysis methods, showed that a multiphase state is formed in the surface layer, represented by a solid solution based on \( \alpha \)-Fe, iron nitride of \( \text{Fe}_4\text{N} \) composition, and chromium nitride of \( \text{CrN} \) composition (figure 1). The relative content of these phases is as follows: \( \alpha \)-Fe - 9.4 % vol.; \( \text{Fe}_4\text{N} \) - 64.2 % vol.; \( \text{CrN} \) - 26.4 % vol.

Investigation of the defective substructure of the modified steel layer, performed by transmission electron microscopy, revealed the formation of a layered structure in the surface layer of \( (3.5-4.0) \mu m \) thick (figure 2), which similar to the structure of lamellar pearlite of carbon steels [10]. The layers are oriented perpendicular to the modification surface and differ in transverse dimensions and contrast. The transverse dimensions of the dark contrast layer vary from \( 25 \text{ nm} \) to \( 36 \text{ nm} \). The transverse dimensions of the light contrast layer vary from \( 10 \text{ nm} \) to \( 15 \text{ nm} \). By the relative content of iron and chromium nitrides in the surface layer, revealed by X-ray diffraction analysis, it can be assumed that the dark contrast layers are iron nitrides of \( \text{Fe}_4\text{N} \) composition, the layers of light contrast are chromium nitrides \( \text{CrN} \).

Studies of the phase composition of the surface layer of steel, carried out by diffraction electron microscopy using the dark-field technique and subsequent indexing of microelectron diffraction patterns, confirmed the above assumption. It was found that the layered structure forming the surface layer is two-phase and is formed by iron nitrides and chromium nitrides (figure 3). The layers formed by iron nitrides...
have large transverse dimensions (figure 3, c), in comparison with the layers formed by chromium nitrides (figure 3, d).

![X-ray diffraction pattern](image)

**Figure 1.** Fragment of an X-ray diffraction pattern of the surface layer of AISI 310 steel subjected to complex processing, combining irradiation with a pulsed electron beam (30 J/cm², 200 μs, 3 pulses) and subsequent nitriding in the plasma of a low-pressure gas discharge (793 K, 3 hours).

![Surface layer structure](image)

**Figure 2.** The structure of the surface layer of AISI 310 steel subjected to complex processing, combining irradiation with a pulsed electron beam (30 J/cm², 200 μs, 3 pulses) and subsequent nitriding in the plasma of a low-pressure gas discharge (793 K, 3 hours). The arrow on (a) indicates the modification surface.
Figure 3. Electron microscopic image of the surface layer structure of AISI 310 steel subjected to complex processing, combining irradiation with a pulsed electron beam (30 J/cm², 200 μs, 3 pulses) and subsequent nitriding in the plasma of a low-pressure gas discharge (793 K, 3 hours); a - bright field, b - microelectron diffraction pattern, c, d - dark fields obtained in the reflections [311]Fe₄N and [311]CrN, respectively. In (b), arrows indicate reflexes in which dark fields were obtained: 1 - for (c), 2 - for (d).

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Thus, using the methods of X-ray phase analysis and transmission electron diffraction microscopy, it was found that in the surface layer of AISI 310 steel subjected to complex processing, combining irradiation with a pulsed electron beam (30 J/cm², 200 μs, 3 pulses) and subsequent nitriding in the plasma of a low-pressure gas discharge (793 K, 3 hours), the layered two-phase structure of the nanoscale range, represented by iron nitrides and chromium nitrides is formed.

4. Conclusion
A complex processing of AISI 310 steel, combining irradiation with a pulsed electron beam and subsequent nitriding in the plasma of a low-pressure gas discharge was carried out. Steel specimens were found to have the highest hardness and wear resistance, the complex treatment of which was carried out at the following parameters: irradiation with a pulsed electron beam of 30 J/cm², 200 μs, 3 pulses; subsequent nitriding at a temperature of 793 K for 3 hours. The wear resistance of steel specimens processed in this way exceeds the wear resistance of the original and irradiated material by more than 100 times. The hardness of the surface layer of the steel modified with these parameters exceeds the hardness of the initial material by more than 11 times and 8 times the hardness of the steel irradiated with a pulsed electron beam. The thickness of the hardened layer reaches 40 μm. It is shown that high strength and tribological properties of the steel are due to the formation of a layered two-phase structure in the nanoscale range, represented by iron nitrides and chromium nitrides.

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