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## COMPLEX ELECTRON-ION-PLASMA SURFACE MODIFICATION OF HIGH-ALLOY STAINLESS STEEL

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**Abstract.** The work is devoted to identification and analysis of patterns of change in the elemental and phase composition, defective substructure, mechanical (microhardness) and tribological (wear resistance and friction coefficient) properties of stainless high-chromium steel subjected to complex processing, combining vacuum irradiation of the samples surface layer with an intense pulsed electron beam of submillisecond exposure duration and subsequent nitriding under electron-ionic heating conditions. High-chromium steel AISI 310S, which in the initial state is a polycrystalline aggregate based on  $\gamma$ -iron, was used as the research material. Pulsed electron beam treatment of steel was carried out on a “SOLO” installation equipped with an electron source with a plasma cathode based on a low-pressure pulsed arc discharge with grid stabilization of the cathode plasma boundary and an open anode plasma boundary. Steel nitriding was carried out on a “TRIO” installation with a chamber size of 600×600×600 mm, equipped with a switching unit to implement the electron-ionic processing mode. Nitriding was carried out at 723, 793, and 873 K temperatures for 1, 3 and 5 h. It was found that electron-ionic nitriding of the samples pre-irradiated with an electron beam (10 J/cm<sup>2</sup>, 200  $\mu$ s, 3 pulses at 723 and 793 K for 3 h) is accompanied by the formation of a ceramic layer containing only iron and chromium nitrides. The highest values of steel wear resistance after electron-ionic nitriding, exceeding the wear resistance of the initial steel by more than 700 times, are observed at nitriding parameters of 793 K, 3 h.

**Keywords:** high-chromium steel, complex processing, pulsed electron beam treatment, nitriding, structure, phase composition, hardness, wear resistance, friction coefficient

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## КОМПЛЕКСНАЯ ЭЛЕКТРОННО-ИОННО-ПЛАЗМЕННАЯ МОДИФИКАЦИЯ ПОВЕРХНОСТИ НЕРЖАВЕЮЩЕЙ ВЫСОКОЛЕГИРОВАННОЙ СТАЛИ

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**Аннотация.** Работа посвящена выявлению и анализу закономерностей изменения элементного и фазового составов, дефектной субструктуры, механических (микротвердость) и трибологических (износостойкость и коэффициент трения) свойств нержавеющей высокохромистой стали, подвергнутой комплексной обработке, которая сочетает облучение в вакууме поверхностного слоя образцов интенсивным импульсным электронным пучком субмиллисекундной длительности воздействия и последующее азотирование в условиях элионного нагрева образцов. В качестве материала исследования используется высокохромистая сталь 20Х23Н18, являющаяся в исходном состоянии поликристаллическим агрегатом на основе  $\gamma$ -железа. Облучение стали импульсным электронным пучком авторы проводили на установке «СОЛО», оснащенной электронным источником с плазменным катодом на основе импульсного дугового разряда низкого давления с сеточной стабилизацией границы катодной плазмы и открытой границей анодной плазмы. Азотирование стали осуществлялось на

установке «ТРИО» с размерами камеры 600×600×600 мм, дооснащенной блоком коммутации для реализации элионного (электронного и ионного) режима обработки. Азотирование проводили при температурах 723, 793 и 873 К в течение 1, 3 и 5 ч. Элионное азотирование при температурах 723 и 793 К в течение 3 ч образцов, предварительно облученных электронным пучком (при режиме 10 Дж/см<sup>2</sup>, 200 мкс, 3 имп.), сопровождается формированием керамического слоя, содержащего только нитриды железа и хрома. Наиболее высокие значения износостойкости стали после элионного азотирования, превышающие износостойкость исходной стали более чем в 700 раз, наблюдаются при параметрах азотирования 793 К, 3 ч.

**Ключевые слова:** высокохромистая сталь, комплексная обработка, облучение импульсным электронным пучком, азотирование, структура, фазовый состав, твердость, износостойкость, коэффициент трения

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## INTRODUCTION

The recent studies have highlighted the increasing demand for surface modification of metals and alloys through complex processing. These processing combines various methods such as saturating the surface layer with gas atoms, depositing thin films of different metals followed by mixing under high-energy pulsed electron beams, applying hard and superhard wear-resistant coatings, and more [1; 2]. These processes result in a gradient structure in the near-surface layer, with a gradual change in the concentration of alloying elements with depth, significantly enhancing the surface's hardness, wear resistance, corrosion resistance, and electrical conductivity [3 – 5]. The most commonly used ion-plasma nitriding method in the industry is nitriding in an abnormal glow discharge [6 – 8]. However, this method has the main disadvantage of relatively high operating pressure, which hinders effective ion cleaning of the surface during nitriding. To overcome this drawback, the Institute of High Current Electronics of the Siberian Branch of the Russian Academy of Sciences developed the plasma generator “PINK,” which has been successfully used for a quarter of a century [9 – 11]. The necessary temperature for the nitriding process using the “PINK” plasma generator is maintained by a flow of ions from the discharge plasma, accelerated to an energy determined by the electric bias on the samples. This often leads to intensive ion etching of the treated surface and a significant increase in its roughness [12; 13]. To mitigate the impact of intense ion bombardment on the formation of the modified layer, studies [14; 15] proposed using the electronic component of the plasma for heating the samples, implementing an elion process. This process allows for adjusting the processing temperature without significantly altering the intensity of ion bombardment.

The aim of this work is to establish the patterns of evolution in the structure, mechanical, and tribological properties of high-chromium steel subjected to a comprehensive treatment that combines pulsed electron

beam irradiation and subsequent elion nitriding in a low-pressure gas discharge plasma.

## MATERIALS AND METHODS

The material used for the study was high-chromium steel AISI 310S with the following composition (wt. %): C 0.2; Si 1.0; Mn 2.0; Ni 17 – 20; Cr 22 – 25; S 0.02; P 0.035; and the remainder Fe. The samples were in the form of plates measuring 10×10×5 mm. The steel was irradiated with a pulsed electron beam using the “SOLO” installation, equipped with an electron source featuring a plasma cathode based on a low-pressure pulsed arc discharge with grid stabilization of the cathode plasma boundary and an open anode plasma boundary [17; 18]. Based on thermal calculations, electron beam energy densities ( $E_s$ ) of 10 and 30 J/cm<sup>2</sup> were selected (pulse duration 200 μs; number of pulses 3; frequency 0.3 s<sup>-1</sup>). At an electron beam energy density of 10 J/cm<sup>2</sup> (200 μs, 3 pulses), a solid-phase mode is achieved, meaning transformations in the surface layer of AISI 310S steel occur within the temperature range where the surface layer remains in the solid state. At an electron beam energy density of 30 J/cm<sup>2</sup> (200 μs, 3 pulses), a liquid-phase mode is realized, meaning transformations in the surface layer of AISI 310S steel occur within the temperature range where the surface layer is in a molten state. The nitriding of the steel was performed using the “TRIO” installation, which has a chamber size of 600×600×600 mm and is equipped with a switching unit to implement the elion (electronic and ionic) processing mode [15]. This process was carried out at temperatures ranging from 723 to 873 K for 1 – 5 h. The temperature of the samples was regulated by the filling factor of the electronic phase. The samples were fixed on a stationary holder in the center of the chamber along the axis of the plasma sources, with the holder positioned at a 60° angle to each source and the samples on the front side of the holder. The process temperature was measured using a chromelalumel thermocouple fixed in the sample holder through a quartz cup.

The structure, elemental, and phase composition of the modified steel were investigated using *X*-ray diffraction analysis, optical microscopy, scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The mechanical properties of the steel were characterized by microhardness (indenter load 0.5 N), and the tribological properties were characterized by wear resistance and the coefficient of friction. The parameters for the tribological tests under dry friction conditions at room temperature were as follows: counter body – a 6 mm diameter silicon carbide (SiC) ball, wear track diameter 4 mm, load 5 N, and friction path length 2000 m.

## RESULTS AND DISCUSSION

Surface treatment of AISI 310S stainless steel with a pulsed electron beam at an energy density ( $E_s$ ) of 10 J/cm<sup>2</sup> (200 μs, 3 pulses) results in the formation of slip traces on the irradiated surface (Fig. 1, *a*), indicating intense deformation of the surface layer due to the relaxation of elastic stresses formed in the surface layer of the samples during the rapid energy input and cooling process. The surface of the samples remains smooth, with no microcracks, microcraters, or micropores observed. This indicates that the irradiation did not lead to the melting of the surface layer of the samples.

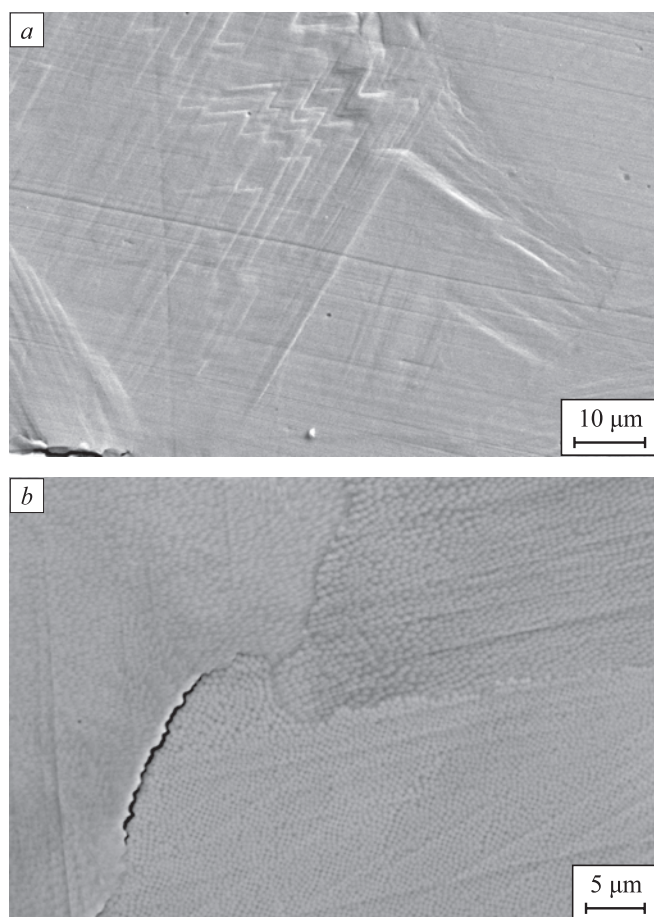
Surface treatment of AISI 310S stainless steel with a pulsed electron beam at an energy density of 30 J/cm<sup>2</sup> (200 μs, 3 pulses) results in the formation of a highly relief structure on the irradiated surface, characterized by a large number of microcraters. A cellular structure is observed within the grains, indicating melting and subsequent high-speed crystallization of the surface layer (Fig. 1, *b*). Therefore, under this irradiation mode, the high-speed melting of the surface layer occurs, which aligns with the results of the temperature field calculations. The crystallization cell sizes range from 330 to 500 nm. Microcracks are present on the surface of the steel, located along the grain boundaries, indicating a high level of residual stresses formed in the surface layer due to rapid cooling.

It has been established that increasing the electron beam energy density leads to a rise in the wear coefficient (a decrease in wear resistance) of the steel from  $1.9 \cdot 10^{-4}$  mm<sup>3</sup>/(N·m) at 10 J/cm<sup>2</sup> to  $5.2 \cdot 10^{-4}$  mm<sup>3</sup>/(N·m) at 30 J/cm<sup>2</sup>. The wear coefficient of the steel before pulsed electron beam irradiation is  $4.9 \cdot 10^{-4}$  mm<sup>3</sup>/(N·m). Additionally, it was shown that the microhardness of the samples increases with the electron beam energy density, from 1.7 GPa in the initial state to 2.4 GPa after irradiation at 30 J/cm<sup>2</sup>.

Subsequent nitriding of the steel resulted in a significant increase (4 to 9 times compared to the initial state) in

the hardness of the surface layer. The hardness of the steel decreases with increasing nitriding temperature and electron beam energy density. The maximum thickness of the hardened layer is 45 – 50 μm, achieved through a comprehensive treatment that combines irradiation at an electron beam energy density of 10 J/cm<sup>2</sup> and subsequent nitriding at a temperature of 793 K for 3 h. Nitriding at 793 K for 3 h yields the best results in tribological tests, with the wear resistance of steel samples irradiated with a pulsed electron beam at  $E_s = 10$  J/cm<sup>2</sup> reaching  $1.2 \cdot 10^{-6}$  mm<sup>3</sup>/(N·m), and at  $E_s = 30$  J/cm<sup>2</sup> reaching  $0.58 \cdot 10^{-6}$  mm<sup>3</sup>/(N·m), which is significantly higher than the wear resistance of the steel in both the initial and irradiated states.

*X*-ray phase analysis determined that the main phases of the modified samples are α-iron and γ-iron, as well as iron nitrides Fe<sub>4</sub>N, Fe<sub>2</sub>N, chromium nitrides CrN, and



**Fig. 1.** Electron microscopic image of the surface structure of AISI 310S steel samples irradiated with a pulsed electron beam:

*a* –  $E_s = 10$  J/cm<sup>2</sup> (200 μs, 3 pulses);

*b* –  $E_s = 30$  J/cm<sup>2</sup> (200 μs, 3 pulses)

**Рис. 1.** Электронно-микроскопическое изображение структуры поверхности образцов стали 20Х23Н18, облученной импульсным электронным пучком:

*a* –  $E_s = 10$  Дж/см<sup>2</sup> (200 мкс, 3 имп.);

*b* –  $E_s = 30$  Дж/см<sup>2</sup> (200 мкс, 3 имп.)

the complex nitride  $\text{Fe}_3\text{NiN}$ . The highest microhardness values (15.8 and 15.6 GPa) were demonstrated by samples subjected to comprehensive treatment, which includes preliminary pulsed electron beam treatment ( $10 \text{ J/cm}^2$ ,  $200 \mu\text{s}$ , 3 pulses) and subsequent nitriding at temperatures of 723 and 793 K for 3 h. The surface layer structure of these samples is characterized by the formation of a ceramic layer containing only iron and chromium nitrides.

Scanning electron microscopy first observed the phenomenon of blistering during electron-ion-plasma nitriding, resulting in the formation of bubbles on the material surface (Fig. 2, *a*).

It is noteworthy that the formation of bubbles is observed on the surface of metals and alloys, metal-ceramic, and ceramic materials subjected to intense corpuscular exposure (ions  $\text{H}^+$ ,  $\text{B}^+$ ,  $\text{He}^+$ , etc.), and is most prominently manifested in nuclear and thermonuclear reactor technologies, as well as in space [19 – 21].

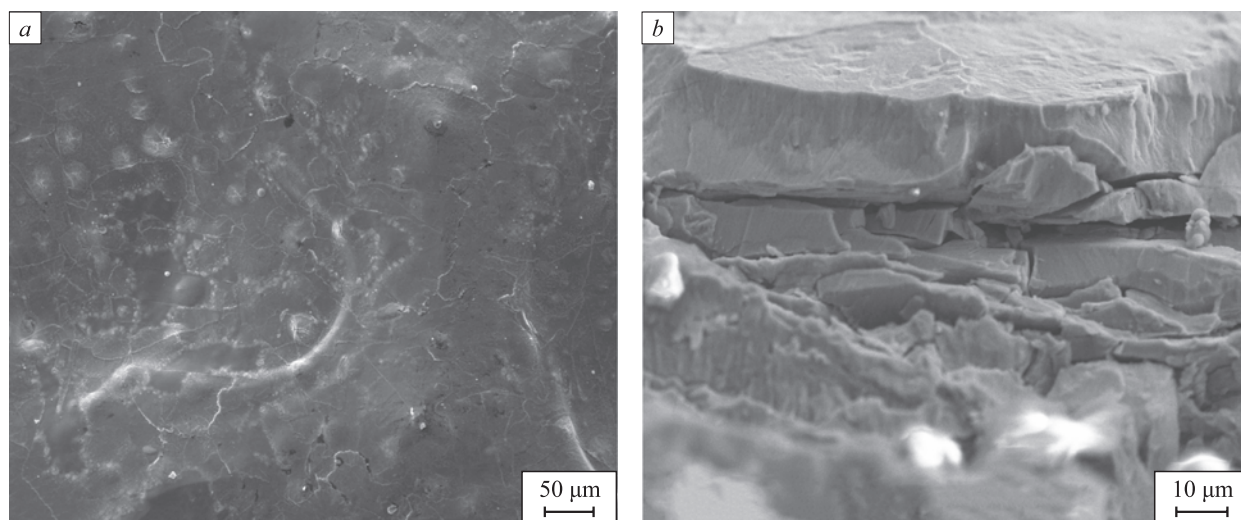
Studies of the fracture surface of samples pre-irradiated with a pulsed electron beam and subjected to elion nitriding revealed that the destruction of the surface layer of the steel predominantly follows a quasi-brittle mechanism (Fig. 2, *b*).

The defect structure of the modified layer was studied using transmission electron microscopy. It was found that nitriding is accompanied by the formation of a lamellar structure (Fig. 3).

Nitriding of samples subjected to preliminary treatment with a pulsed electron beam at an electron beam energy density of  $10 \text{ J/cm}^2$ , pulse duration of  $200 \mu\text{s}$ , and three pulses, leads to the formation of a structure with alternating iron nitride and chromium nitride plates and results in a structure characterized by alternating  $\gamma$ -iron plates and predominantly iron nitrides.

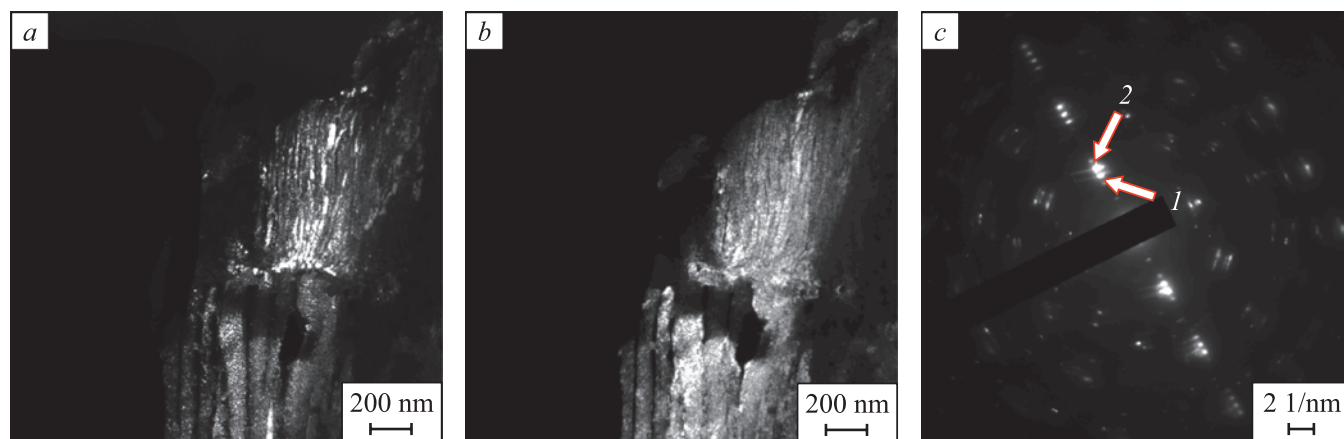
## CONCLUSIONS

Nitriding process was carried out on samples of AISI 310S steel, which had been pre-irradiated with a pulsed electron beam, in a low-pressure gas discharge plasma with sample heating by plasma electrons (elion nitriding method). The formation of blisters on the material surface was observed during elion nitriding. Nitriding resulted in the formation of a plate-like structure with alternating iron nitride and chromium nitride plates (for samples pre-irradiated with a pulsed electron beam at an energy density of  $10 \text{ J/cm}^2$ , pulse duration of  $200 \mu\text{s}$ , and three pulses) or a structure with alternating  $\gamma$ -iron plates and predominantly iron nitrides (for samples pre-irradiated with a pulsed electron beam at an energy density of  $30 \text{ J/cm}^2$ , pulse duration of  $200 \mu\text{s}$ , and three pulses). The highest microhardness values (15.8 and 15.6 GPa) were demonstrated by samples subjected to combined treatment, which included preliminary treatment with a pulsed electron beam ( $10 \text{ J/cm}^2$ ,  $200 \mu\text{s}$ , three pulses) and subsequent nitriding at temperatures of 723 and



**Fig. 2.** Electron microscopic image of the AISI 310S steel structure subjected to complex modification, combining irradiation with a pulsed electron beam at  $10 \text{ J/cm}^2$ ,  $200 \mu\text{s}$ , 3 pulses (*a*) and  $30 \text{ J/cm}^2$ ,  $200 \mu\text{s}$ , 3 pulses (*b*) and subsequent nitriding at 793 K for 3 h:  
*a* – modification surface; *b* – fracture surface

**Рис. 2.** Электронно-микроскопическое изображение структуры стали 20Х23Н18, подвергнутой комплексному модифицированию, сочетающему облучение импульсным электронным пучком при  $10 \text{ Дж/см}^2$ ,  $200 \text{ мкс}$ , 3 имп. (*a*) и при  $30 \text{ Дж/см}^2$ ,  $200 \text{ мкс}$ , 3 имп. (*b*) и последующее азотирование при 793 К, 3 ч:  
*a* – поверхность модифицирования; *b* – поверхность излома



**Fig. 3.** Electron microscopic images of the AISI 310S steel surface layer structure subjected to complex modification (irradiation with a pulsed electron beam at 30 J/cm<sup>2</sup>, 200 μs, 3 pulses and subsequent nitriding at 793 K for 3 h):  
*a* and *b* – dark fields obtained in [111]γ-Fe and [002]γ-Fe + [002]Fe<sub>4</sub>N reflections;  
*c* – microelectron diffraction pattern (arrows indicate reflections in which dark fields 1 (*a*), 2 (*b*) were obtained)

**Рис. 3.** Электронно-микроскопическое изображение структуры поверхностного слоя стали 20Х23Н18, подвергнутой комплексной модификации (облучение импульсным электронным пучком при 30 Дж/см<sup>2</sup>, 200 мкс, 3 имп. и последующее азотирование при 793 К, 3 ч.):  
*a* и *b* – темное поле, полученное в рефlekсах [111]γ-Fe и [002]γ-Fe + [002]Fe<sub>4</sub>N;  
*c* – микроэлектроннограмма (стрелками указаны рефlekсы, в которых получены темные поля 1 (*a*), 2 (*b*))

793 K for 3 h. The highest wear resistance, significantly surpassing that of the steel in both its initial and irradiated states, was observed in samples pre-irradiated with a pulsed electron beam at  $E_s = 30$  J/cm<sup>2</sup> and nitrided at a temperature of 793 K for 3 h.

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