Multi-cycle of AISI 5135 steel modification by irradiation of the "film (Si (0.2 μm) + Nb (0.2 μm))/(AISI 5135 steel) substrate" system with an intense pulsed electron beam

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Abstract. Steel AISI 5135 surface layer modification carried out by high-cycle high-speed melting of the “film (Si + Nb)/(steel AISI 5135) substrate” system with an intense pulsed electron beam with an impact area of several square centimeters, have been implemented in a single vacuum cycle on the “COMPLEX” setup. The regime of the system "film (Si (0.2 μm) + Nb (0.2 μm))/(steel AISI 5135) substrate" irradiation with an intense pulsed electron beam (20 J/cm², 200 μs, 3 pulses, 3 cycles) which makes it possible to form a surface layer with high thermal stability have been revealed. This layer is characterized by high hardness, more than 3 times higher than the hardness of AISI 5135 steel in the original (ferrite-pearlite structure) and wear resistance, more than 90 times higher than the wear resistance of the initial AISI 5135 steel. It is shown that the high strength and tribological properties of steel are due to the formation of the hardening phase particles (niobium silicide of Nb₅Si₃ composition).

1. Introduction
The development of new materials and technologies for their production is an indispensable condition for the successful technical and social development of society [1]. Binary systems “metal – silicon”, containing a relatively large amount of metal silicides, occupy a special place among the various types of modern materials [2, 3]. Currently silicides are successfully used for the manufacture of electrical contacts, connections, diffusion barriers of integrated circuits [4, 5], as catalysts for the growth of silicon and carbon nanostructures [6, 7]. The silicide’s application area is such an important industry as nuclear power [8, 9]. Metal silicides, possessing unique physical properties [10, 11], have found application in optoelectronic light-emitting devices, infrared detectors, and solar energy conversion devices [12]. Irradiation of the "film/substrate" system with an intense pulsed electron beam is one of the promising directions for the formation of silicides in the products surface layer made of metals and alloys, cermet and ceramic material [13-18].

The establishment and analysis the regularities of the phase composition and defect substructure of the surface alloy formed as a result of high-cycle irradiation with a low-energy pulsed electron beam of the
"film (Si + Nb)/(AISI 5135 steel) substrate" system formation in a single vacuum cycle is the aim of this work.

2. Material and methods
The material under study was a surface alloy of Fe-C-Cr-Si-Nb elemental composition, formed as a result of high-cycle high-speed melting of the "film (Si + Nb)/(AISI 5135 steel) substrate" system in a single vacuum cycle. The formation of the surface alloy was carried out on the "COMPLEX" setup, developed in the plasma emission electronics laboratory of the HCEI SB RAS, included in the list of unique installations of the Russian Federation (the "UNIKUUM" complex http://ckp-rf.ru/usu/434216/) [18]. We used pre-polished specimens of AISI 5135 steel ((0.31 - 0.44) C - (0.17 - 0.37) Si - (0.5 - 0.8) Mn - 0.3 Ni - 0.035 S - 0.035 P - (0.8 - 1.1) Cr - 0.3 Cu, the rest is Fe, wt.%) as a substrate. Specimens have the shape of a cylinder 10 mm in diameter and 10 mm thick. Thin (0.2 μm each) films of silicon and niobium were successively deposited to the polished surface of AISI 5135 steel specimens. The deposition of a silicon film was carried out by the magnetron method, and niobium films - by an electric arc method with plasma assistance. Then the resulting "film/substrate" system was irradiated with a pulsed electron beam in the selected mode. A similar procedure was repeated up to five times (1, 2, 3, 4 and 5 cycles of the steel surface layer modification). The following four modes of irradiation of the "film/substrate" system with a pulsed electron beam were chosen: (1) the energy density of the electron beam \( E_{S} = 20 \text{ J/cm}^2 \), pulse duration of the electron beam exposure \( \tau = 200 \mu \text{s} \), the number of exposure pulses \( n = 3 \); (2) \( E_{S} = 20 \text{ J/cm}^2 \), \( \tau = 200 \mu \text{s} \), \( n = 30 \); (3) \( E_{S} = 50 \text{ J/cm}^2 \), \( \tau = 200 \mu \text{s} \), \( n = 3 \); (4) \( E_{S} = 50 \text{ J/cm}^2 \), \( \tau = 200 \mu \text{s} \), \( n = 30 \). The accelerated electrons energy is 18 keV and the pulse repetition rate is 0.3 s\(^{-1}\) for all four modes of irradiation. Mathematical modeling of the temperature field formed in steel indicates that the selected irradiation regimes correspond to different degrees of the steel surface layer high-speed melting [19].

The elemental and phase composition, the state of the defective substructure of the surface alloy were studied by scanning electron microscopy (Philips SEM-515 microscope with EDAX ECON IV microanalyzer) and X-ray diffraction analysis (Shimadzu XRD 6000 diffractometer). The properties of the material were characterized by microhardness (PMT 3 device, load on the indenter 0.5 N). Tribological studies (determination of wear resistance and friction coefficient) were carried out on the Oscillating TRIBOtester (TRIBotechnic, France, Pin on Disc) with the following parameters: a ball made of 100Cr6 steel with a diameter of 6 mm, a track radius of 2 mm, an indenter load of 5 N, a track length of 800 m.

3. Results and discussion
The modification of the surface layer of AISI 5135 steel was carried out in a single vacuum space, carried out by high-cycle high-speed melting of the "film (Si + Nb)/(AISI 5135 steel) substrate" system with an intense pulsed electron beam with an impact area of several square centimeters. The high-cycle modification of AISI 5135 steel mode by irradiation of the "film (Si (0.2 μm) + Nb (0.2 μm))/(AISI 5135 steel) substrate" system with an intense pulsed electron beam (20 J/cm\(^2\), 200 μs, 3 pulses, 3 cycles) was revealed. It makes it possible to form a surface layer characterized by a high hardness equal to 9300 MPa (3.2 times higher than the hardness of AISI 5135 steel in the initial (ferrite-pearlite structure) state) and wear resistance more than 90 times higher than that of the initial steel. It was found that an increase in the energy density of the electron beam up to 50 J/cm\(^2\) (200 μs, 3 and 30 pulses) leads (regardless of the number of irradiation cycles of the "film (Si + Nb)/(AISI 5135 steel) substrate" system) to the formation of a high-relief surface layer with low strength and tribological properties.

It has been shown by scanning electron microscopy that the highest level of hardening of the steel surface layer, achieved after 3 irradiation pulses at 3 and 5 irradiation cycles (20 J/cm\(^2\), 200 μs), takes place under processing modes leading to high-speed hardening of the steel subsurface layer with the
formation of a martensitic structure (figure 1(a)). An increase in the number of irradiation pulses to 30 leads to an increase in the average size of grains and subgrains, martensite crystals (figure 1(b)), which is accompanied by a decrease in the strength and tribological properties of the material.

The studies of the phase composition of the steel AISI 5135 surface layer, subjected to high-cycle processing by X-ray phase analysis methods have been carried out. It was found that the main phases formed upon irradiation of the system "film (Si (0.2 μm) + Nb (0.2 μm))/(steel AISI 5135) substrate" by an intense pulsed electron beam (regardless of the modification cycles number and the pulsed electron beam irradiation mode) are solid solutions based on chromium (Cr 0.93 - Si 0.07) and (Cr 0.96 - Nb 0.04), iron (Fe 0.94 - Si 0.06) and (Fe 0.83 - Si 0.135 - Nb 0.03). In the sample that showed the highest values of microhardness and wear resistance (20 J/cm², 200 μs, 3 pulses, 3 cycles), along with solid solutions of Fe 0.83 - Si 0.135 - Nb 0.03 and Cr 0.96 - Nb 0.04 composition, there is a niobium silicide with the composition Nb₅Si₃ (up to 9 wt.%).

**Figure 1.** Electron microscopic image of the steel AISI 5135 specimens surface structure, formed as a result of irradiation of the "(Si (0.2 μm) + Nb (0.2 μm))/(steel AISI 5135) substrate" system by a pulsed electron beam with parameters of 20 J/cm², 3 (a) and 30 (b) pulses. Three-cycle processing.

4. Conclusion
The modification of the AISI 5135 steel surface layer was carried out in a single vacuum space, carried out by high-cycle high-speed melting of the "film (Si + Nb)/(AISI 5135 steel) substrate" system with an intense pulsed electron beam with an impact area of the several square centimeters. The high-cycle of AISI 5135 steel modification mode by irradiation of the "film (Si (0.2 μm) + Nb (0.2 μm))/(AISI 5135 steel) substrate" system with an intense pulsed electron beam (20 J/cm², 200 μs, 3 pulses 3 cycles) was detected. It makes possible to form a surface layer characterized by a high hardness equal to 9300 MPa (3.2 times higher than the hardness of AISI 5135 steel in the initial (ferrite-pearlite structure) state) and wear resistance more than 90 times higher than that of the initial AISI 5135 steel. It has been shown that the high strength and tribological properties of the modified steel are due to the formation of a martensitic structure strengthened by particles of niobium silicide with the Nb₅Si₃ composition during high-speed quenching.

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