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Structure and properties of coatings based on refractory elements obtained by non-vacuum electron beam surfacing

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ABSTRACT

Introduction. The development of modern industry requires materials capable of withstanding high temperatures and loads while maintaining functionality and performance. Traditional materials, such as *0.4 C-Cr* structural steel, are widely used in mechanical engineering and are inexpensive. However, ordinary and low-alloy steels are subject to intense oxidation when exposed to temperatures above 400°C. To improve the performance of structural steels under high-temperature conditions, the development of effective methods for modifying their surfaces is an an urgent task. **The purpose of this work** is to develop a technology for creating high-temperature oxidation resistant surface layers on *0.4 C-Cr* structural steel. For this purpose, the non-vacuum electron beam surfacing method was used, employing powder materials based on refractory elements: niobium, molybdenum, and boron. **Materials and methods.** In this study, modified layers were formed on *0.4 C-Cr* steel using non-vacuum electron beam surfacing of *Nb-Mo-B* powder composites. The following research methods were used: optical microscopy, scanning electron microscopy, X-ray diffraction analysis, microhardness testing, high-temperature oxidation testing, and oxidation reaction kinetics determination. **Results and discussion.** The modified layers, which were 2.0–2.3 mm thick, exhibited a gradient structure consisting of molybdenum-doped niobium carbide present as dendrites and irregularly shaped crystals, as well as eutectic colonies based on the same carbide and α-*Fe* and α-(*Mo*,*Fe*) solid solutions. X-ray phase analysis identified the following phases in the modified layers: (*Nb*,*Mo*)*C* carbide and α-*Fe* and α-(*Mo*,*Fe*)-based solid solutions. The surfacing with *Nb*, *Mo*, and *B* resulted in the formation of layers on the surface of *0.4 C-Cr* carbon steel that are 2.9 times harder and 3.9 times more temperature oxidation resistant than those of the unmodified steel.

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Introduction

Industries such as power engineering, chemical engineering, aircraft manufacturing, and mechanical engineering require products capable of operating under high loads, at elevated temperatures, and in aggressive corrosive environments. To ensure these performance characteristics, components are manufactured from structural materials with enhanced strength and corrosion-resistant properties. However, the widespread use of such materials is limited by their high cost. Surface modification and protective coating methods,

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including surfacing, are considered to be cost-effective alternatives [1, 2]. Surface treatment methods include plasma, laser, electron beam, and other technologies [3, 4]. These technologies make it possible to form protective layers on the surface of components made from inexpensive and widely used materials. These methods can also be used to restore a damaged component surface instead of replacing it, which reduces the cost of expensive materials and shortens the repair time. A promising area in materials science is the forming coatings based on refractory elements (niobium, molybdenum, vanadium, tungsten, etc.) [5–7], since, depending on the chemical composition and production technology, they are capable of providing strength at elevated temperatures, high hardness and wear resistance [8], as well as increased high temperature oxidation resistance above 500 °C and under normal conditions [9]. Recently, it has been proposed to use compounds of refractory elements with non-metals, such as borides and carbides [10, 11, 12]. In particular, works [11, 12] consider the prospects of using compounds of niobium and molybdenum with boron and carbon [13-15]. The presence of these compounds in the structure of the material increases the heat resistance, hardness and wear resistance of the coating [16]. It should be noted that niobium, molybdenum and boron are used as alloying components individually or sometimes in combination of only two components, for example, boron-niobium [17, 18, 19]. However, the production of coatings by surfacing on structural medium-carbon steel with simultaneous alloying with niobium, molybdenum and boron has not been considered in the literature. An important point is that the creation of coatings based on refractory elements requires high energy costs [20]. The use of a beam of relativistic electrons released into the air atmosphere, due to its high efficiency, makes it possible to easily remelt powder mixtures of refractory elements and their compounds. Thus, it is possible to form a modified layer up to 3 mm thick, based on refractory compounds, possessing a set of properties: wear resistance, corrosion resistance, heat resistance, and high hardness [21]. Coatings obtained by this technology have high adhesion to steel and low defectivity. Part of the electron beam energy is dissipated in the powder layer, causing its heating and melting, and part of the energy is used to heat the substrate. This method allows for the melting of even refractory elements and compounds while exerting minimal thermal impact on the substrate [22]. In the present study, modified layers based on *Nb*, *Mo*, and *B*, their microhardness, and high temperature oxidation resistance were investigated for the first time. The layers were obtained by non-vacuum electron beam surfacing of *Nb-Mo-B* powders, with the simultaneous introduction of these three components into the melt pool.

The purpose of this study was to modify the surface layers of *0.40% C-1% Cr* structural steel using non-vacuum electron beam surfacing of powder composites consisting of niobium, molybdenum, and boron to form layers with increased high temperature oxidation resistance and hardness. To achieve this goal, the following ***objectives*** were addressed:

– to form modified layers containing refractory elements such as niobium, molybdenum, and boron on *0.40% C-1% Cr* steel blanks using surfacing;

– to analyze the structure and phase composition of the modified layers;

– to evaluate the microhardness of the resulting materials;

– to analyze the high temperature oxidation resistance of the layers.

Materials and methods

Structural steel *0.40% C-1% Cr* was chosen as a model material for forming a heat-resistant coating [23]. This steel is widely used in the manufacturing products and mechanisms; however, it does not have high-temperature oxidation resistance. Therefore, this work considers the prospects of forming protective coatings based on refractory elements on inexpensive structural materials. In this regard, powder mixtures of niobium, molybdenum**,** and boron were surfaced onto the steel surface (Table 1). In order to minimize oxidation processes during electron beam processing, a fluxing additive – magnesium fluoride (MgF_2) – was added to the charge mixture to ensure the creation of a protective environment. An *ELV-6* industrial electron accelerator was used as a source of concentrated energy during surfacing; the work was carried out on an experimental setup of the *G.I. Budker Institute of Nuclear Physics SB RAS* (Novosibirsk). The surfacing modes are listed in Table 2.

Table 1

Powder mixtures compositions

Composition designation	Powder mixtures compositions, wt.%			
	Nb	*Mo*	*B*	MgF_2
Composition 1 (*Nb20-Mo10*)	20	10	10	60
Composition 2 (*Nb10-Mo20*)	10	20	10	60

Table 2

Non-vacuum electron beam surfacing modes

Parameter	Value
Electron beam current, ***I***	23 mA
Electron beam energy, ***E***	1.4 MeV
Powder mass per unit area, ***m***	0.45 g/cm²
Sample movement speed, ***V***	10 mm/s

The structure of the surfaced layers was analyzed using a *Carl Zeiss Axio Observer Alm* metallographic microscope and a *Carl Zeiss EVO50 XVP* scanning electron microscope in secondary electron mode. Particle sizes and their volume fraction in the surfaced layer were estimated using the ImageJ software package based on five images of the structure from different areas of the coating. Energy-dispersive spectroscopy was used to determine the elemental composition of the microstructure. Microhardness was measured using the Vickers method in accordance with *GOST 6507-1-2007* on a *Wolpert Group 402MVD* microhardness tester at a load of 0.98 N in the direction from the surface of the surfaced layer to the steel substrate [24]. For each surfacing mode, the hardness was estimated using five samples. X-ray phase analysis of the materials was performed on a *Thermo Scientific ARL X'TRA* diffractometer. The high-temperature oxidation resistance of 40Kh steel and the resulting layers was assessed in accordance with *GOST 6130-71* [25]. The high-temperature oxidation resistance test parameters were as follows: samples were maintained at 850°C for 48 hours in air. Every 4 hours, the samples were weighed on a Gosmetr-124 analytical balance with a measurement accuracy of 0.1 mg. The base material, *0.40% C-1% Cr* steel, was used as the reference for all measurements.

To calculate the rate constant of the oxidation reaction, the following Equation was used:

$$(\Delta W)^n = k_p t\,, \tag{1}$$

where ΔW is the mass gain per unit area; n is the exponential constant; t is the oxidation time; k_p is the oxidation rate constant.

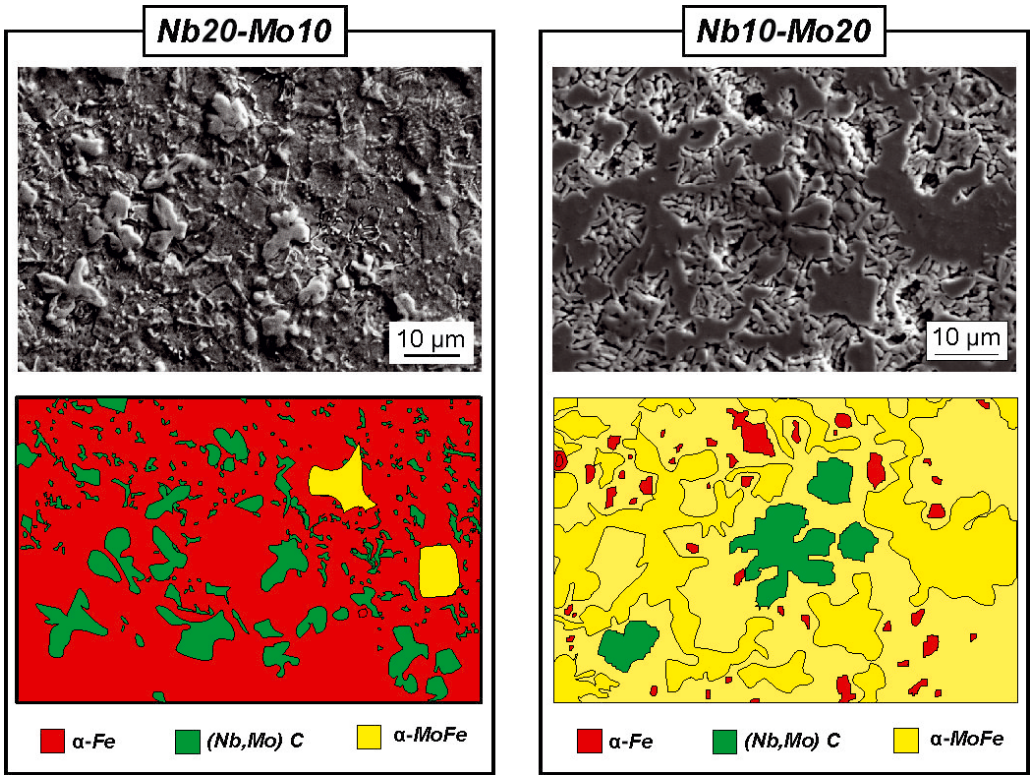
Equation (1) can be reduced to a linear equation using logarithms:

$$n\ln\Delta W = \ln k_p + \ln t\,. \tag{2}$$

A linear regression approximation of the graph in coordinates $\ln \Delta W - \ln t$ was carried out.

Results and Discussion

Fig. 1 shows the images of the microstructure and the schematic diagram of the structure of the formed layers. For materials with **composition 1** (*Nb20-Mo10*) and **composition 2** (*Nb10-Mo20*), a similar structure is formed during surfacing. The microstructure consists of finely dispersed particles (having an average size of 8 ± 2 μm and 18 ± 5 μm for the modified layers obtained by surfacing **compositions 1** and **2**, respectively), which are presumably molybdenum-alloyed niobium carbides, distributed uniformly throughout the volume of the surfaced layer. The morphology of the carbide particles varies from crystals with irregular



a *b*

Fig. 1. Microstructure of modified layers formed during surfacing of powder mixtures:
a – Nb20-Mo10; *b – Nb10-Mo20*

geometry to a petal-shaped form. According to the literature, irregularly shaped niobium carbide crystals are formed first. The primary formation of niobium carbides is due to their crystallization temperature and a stronger affinity for carbon in niobium than in molybdenum and iron [26]. Next, niobium carbide crystals grow, acquiring a petal-shaped form. Simultaneously with the preceding process, partial replacement of niobium atoms in the carbide with molybdenum atoms occurs. This is how molybdenum-alloyed niobium carbides are formed. Furthermore, it should be noted that a decrease in niobium concentration in the melt is a key factor influencing the morphology of the forming phases. A niobium deficiency limits the growth of petal-shaped carbide crystals, leading to the formation of finely dispersed, irregularly shaped particles. The final stage of the crystallization process is the precipitation of an iron-molybdenum matrix, which fills the spaces between the molybdenum-alloyed niobium carbides.

The volume fraction of hardening particles (carbides) in the coating of **composition 1** – *Nb20-Mo10* – is approximately 17.5 %. For the **composition 2** – *Nb10-Mo20* – a decrease in the volume fraction of hardening particles to 12.5% was recorded (Fig. 1).

The elemental mapping results presented in Fig. 2, *a*, *b* for coatings obtained by surfacing powder **compositions 1** and **2**, respectively, allow for a qualitative analysis of the element distribution among the structural components. The maps demonstrate that the petal-shaped and irregular crystals are enriched in niobium, molybdenum, and carbon, which supports their identification as the carbide phase *(Nb,Mo)C*. The intercrystalline space, in turn, is characterized by a predominant content of iron and molybdenum, indicating the formation of a ductile matrix based on an **α**-iron solid solution (**α**-*Fe*) and a molybdenum-based solution (**α**-(*Mo*,*Fe*)).

The analysis of the diffraction patterns (Fig. 3) showed that the phase composition of the surfaced layer includes a carbide phase of the (*Nb*,*Mo*)*C* type and two main metallic phases – solid solutions based on **α**-*Fe* and **α**-(*Mo*,*Fe*).

According to the literature, boron has low solubility in *Nb* (~0.15 wt.%), *Mo* (~0.1 wt.%), and **α**-*Fe* (~0.002 wt.%). However, it easily forms niobium borides [26]. Despite this, no chemical compounds with

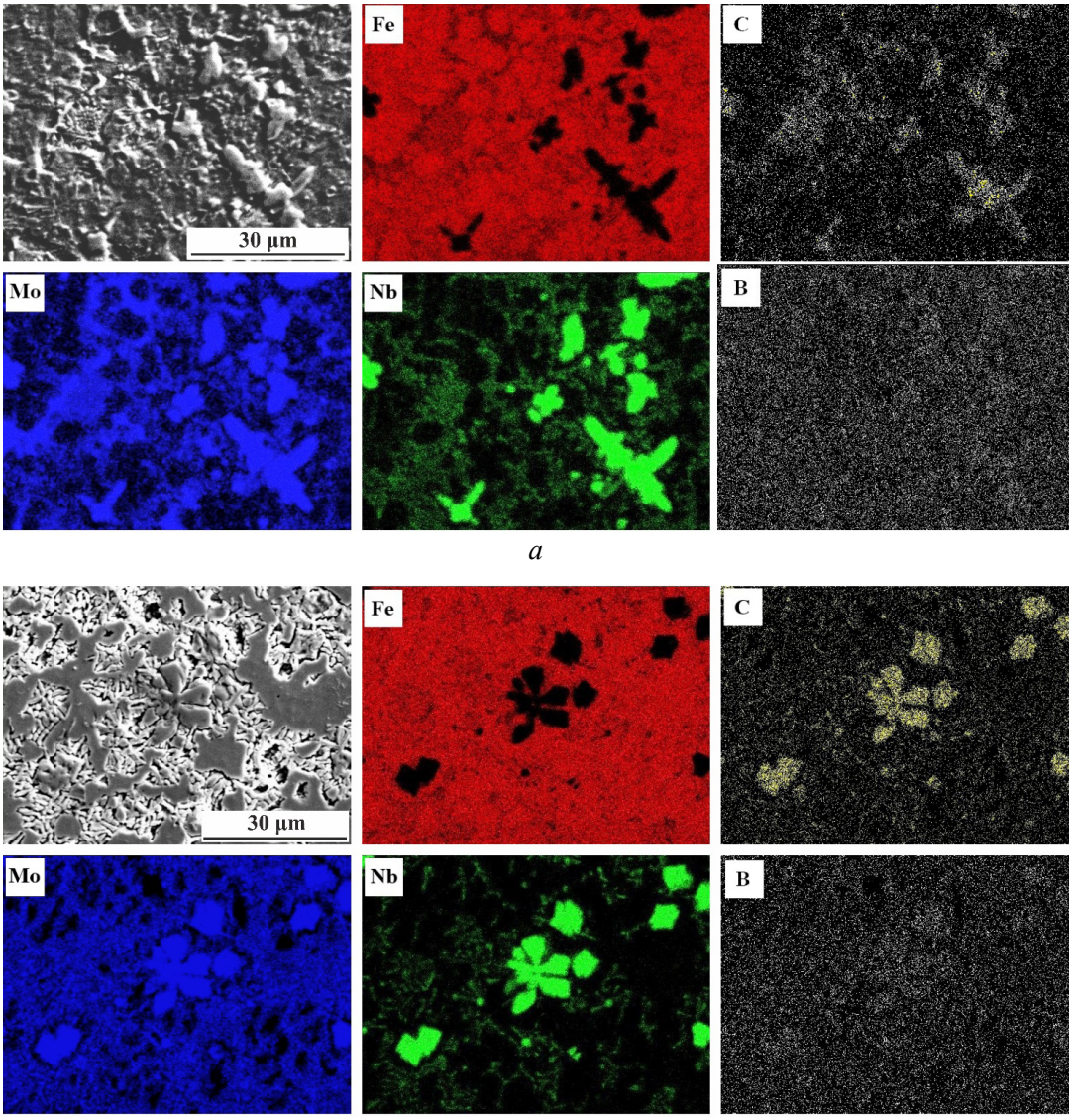


Fig. 2. Energy-dispersive spectroscopy of modified layers: composition of the powder mixture:

a – Nb20-Mo10; *b – Nb10-Mo20*

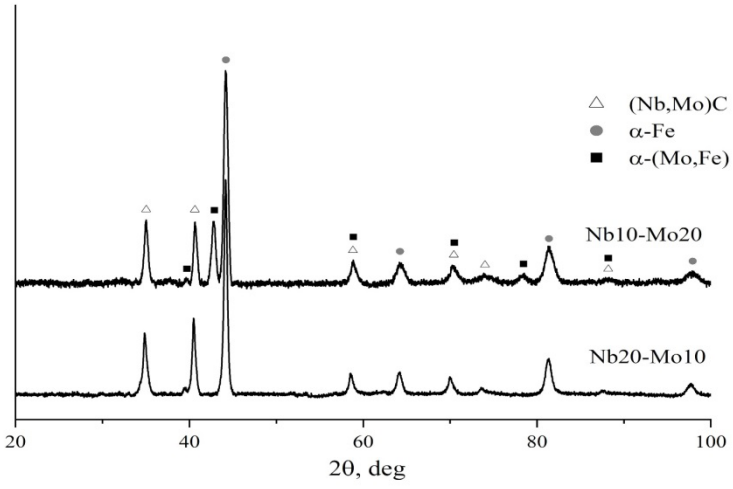


Fig. 3. X-ray diffraction patterns of modified layers

boron were recorded. This is explained by the oxidation of most of the boron during the surfacing process with the formation of boron anhydride B_2O_3. This oxide melts at 450 °C and mixes with slag [27]. The remaining small portion of boron dissolved in the plastic matrix based on **α**-phase solutions of molybdenum and iron and did not have time to separate as individual compounds due to the suppression of diffusion processes caused by high cooling rates. The results of energy-dispersive spectroscopy confirm the distribution of boron throughout the volume of the material (Fig. 2).

The results of structural and phase studies also indicate that the layers surfaced with powder mixtures of **compositions 1** and **2** have different proportions of iron-molybdenum-based solid solutions. *Nb10-Mo20* materials contain a higher amount of the **α**-(*Mo*,*Fe*) phase than *Nb20-Mo10*. This is confirmed by scanning electron microscopy images (Figs. 1 and 2) and X-ray phase analysis, as evidenced by the increased intensity of reflections from this phase (Fig. 3). This feature is associated with the increased molybdenum content in the powder coating.

The results of microhardness tests (Fig. 4) demonstrate the formation of modified layers with significantly higher hardness values compared to the original steel matrix. The layers formed by surfacing with mixture **composition 1** exhibit the greatest increase in microhardness values — 2.9 times. This is explained by the presence of a large amount of (*Nb*,*Mo*)*C* carbides in the structure of these layers, which have a high hardness of approximately 20 GPa [28].

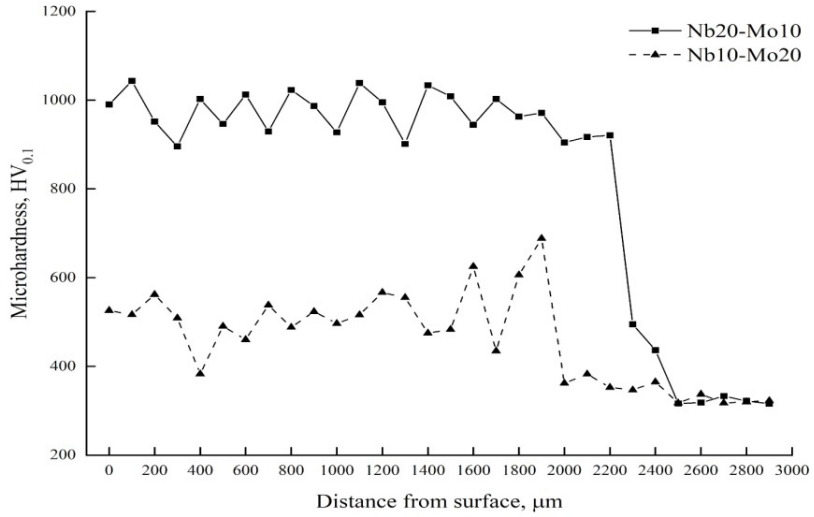


Fig. 4. Microhardness testing results

This study focuses on improving the high-temperature oxidation resistance of structural steel. To this end, high-temperature oxidation resistance tests were conducted on samples, followed by an analysis of the reaction kinetics and morphology of the oxidation products.

Fig. 5 shows the dependences of the change in sample weight on the holding time in the furnace, obtained from the results of high-temperature oxidation resistance tests. The plot shows that *0.40% C-1% Cr* steel is characterized by a linear relationship between the increase in weight and the holding time. This means that the oxidation rate does not change over time and the steel does not possess high-temperature oxidation resistance. A nonlinear relationship is characteristic of the modified *Nb20-Mo10* and *Nb10-Mo20* layers. It is also evident from the dependences that the increase in weight after oxidation does not reach the saturation level, which is typical for the cessation of the oxidation process and surface passivation. This is likely due to intense oxidation processes along the interfaces of the carbide phases and the solid solution [29]. Fig. 6 shows a diagram of the relative high-temperature oxidation resistance of the studied materials.

As a result of the calculation, the oxidation reaction rate constant $\boldsymbol{k_p}$ for the *Nb20-Mo10* coating was 2.3×10^{-3} mg^2/(cm$^4\cdot$h) for the *Nb10-Mo20* coating it was 0.84×10^{-3} mg^2/(cm$^4\cdot$h). For *0.40% C-1% Cr* steel, the reaction rate constant was 16.1×10^{-3} mg^2/(cm$^4\cdot$h). A lower constant value indicates better high-temperature oxidation resistance. The exponent for the **surfaced** compositions was approximately equal to

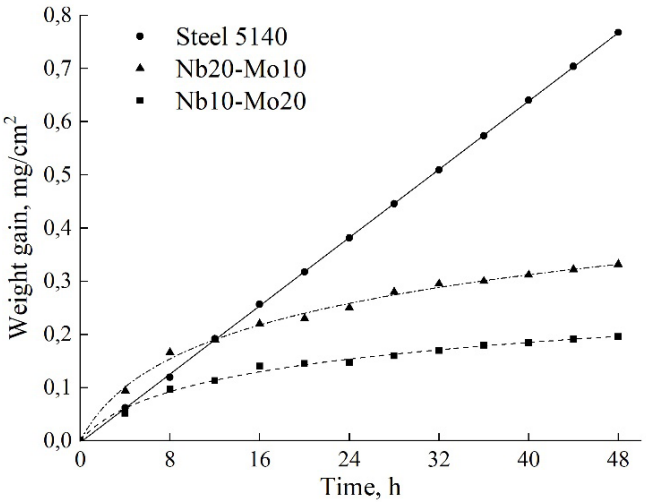


Fig. 5. Dependence of the mass change on the test time

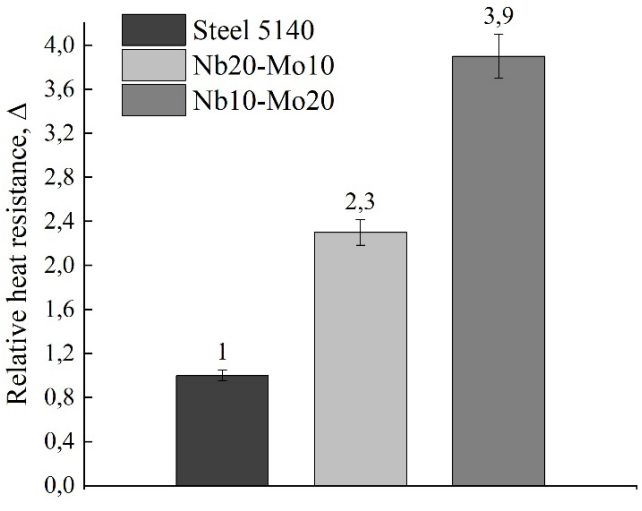


Fig. 6. Relative high-temperature oxidation resistance of surfaced layers

2, which indicates a parabolic law of oxide film growth. The exponent for *0.40% C-1% Cr* steel is 1, which confirms the linear law of film growth. The linear law corresponds to a steady-state oxidation mode; in this case, the oxidation process is determined by the rate of oxygen diffusion through the formed oxide layer.

Fig.7 shows the morphology of the oxide films formed on the samples after high-temperature oxidation resistance testing. The film structure on the *Nb20-Mo10* samples developed in the form of columnar structures (Fig. 7, *a*, *b*). They consist of plate-shaped crystals with rounded edges, quite tightly bonded to

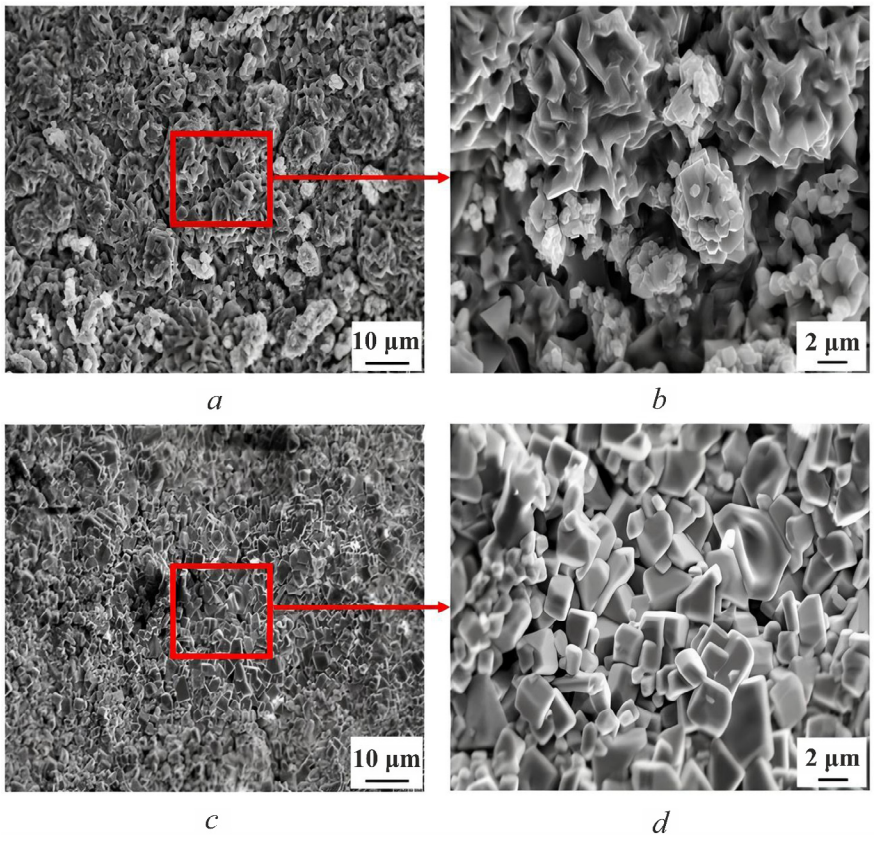


Fig. 7. Oxide layers formed on the surface of samples after high-temperature exposure:

a, b – Nb20-Mo10; *c, d – Nb10-Mo20.* The red square and arrow highlight the areas shown at a higher resolution on the right

one another. The oxide layer on the surface of the *Nb10-Mo20* samples is dense, without cracks or pores (Fig. 7, *c*, *d*). The oxidation products are uniformly distributed polyhedral oxide crystals with sizes of 1–5 μm.

According to experimental data, the coating obtained by surfacing the powder mixture of **composition 2** has better high-temperature oxidation resistance compared to the *Nb20-Mo10* coating. This difference is due to the fact that *Nb10-Mo20* coatings form a denser, continuous**,** and uniform oxide film, presumably of the composition MoO_2 [30]. The proportion of molybdenum introduced into the powder mixture of **composition 2** (*Nb10* + *Mo20*) is greater than in **composition 1** (*Nb20* + *Mo10*). This led to a greater enrichment of the modified layer with molybdenum and increased its high-temperature oxidation resistance, which is consistent with the literature data [31].

Conclusion

1. The study demonstrated that non-vacuum electron beam surfacing of powder mixtures based on niobium (*Nb*), molybdenum (*Mo*), and boron (*B*) enables the creation of modified layers on the surface of *0.40% C-1% Cr* medium-carbon structural steel up to 2,400 μm thick. The structure of these layers consists of molybdenum-alloyed niobium carbides distributed in a ductile matrix consisting of solid solutions of iron and molybdenum.

2. The analysis revealed that, at any of the studied niobium and molybdenum ratios (20% *Nb* + 10% *Mo* or 10% *Nb* + 20% *Mo*), the following phases are formed in the structure of the surfaced layer: alloyed niobium carbide (*Nb*,*Mo*)*C*, an **α**-solid solution of iron, and an **α**-solid solution of molybdenum alloyed with iron. Despite the introduction of 10% boron, there are no signs of the formation of *Nb* or *Mo* borides, which indicates its transition to a solid solution based on iron and molybdenum. In addition, it was found that in the modified layer, when using the *Nb20* + *Mo10* powder surfacing mixture, alloyed niobium carbides with a size of 8 ± 2 μm are formed in an amount of 17.5%; when using the *Nb10* + *Mo20* powder surfacing mixture, alloyed niobium carbides with a size of 18 ± 5 μm are formed in an amount of 12.5%.

3. The formation of modified layers alloyed with niobium, molybdenum, and boron significantly increases the microhardness of the surface layer of *0.40% C-1% Cr* steel (the microhardness of unmodified steel is 330 HV). The average microhardness value (~970 HV) was obtained for the modified layer of the *Nb20* + *Mo10* composition, while for the *Nb10* + *Mo20* composition this value was ~522 HV. The high *Nb* content (20%) enhances the formation of niobium carbides alloyed with molybdenum, which increases the hardness.

4. Alloying the surface of *0.40% C-1% Cr* steel with niobium and molybdenum significantly improves the material's high-temperature oxidation resistance. Quantitative analysis showed that the addition of molybdenum (20%) and niobium (10%) increases high-temperature oxidation resistance by 3.9 times, while alloying with niobium (20%) and molybdenum (10%) increases this indicator by 2.3 times. Increasing the proportion of *Mo* (20%) facilitates the production of molybdenum-based solid solutions, which improve high-temperature oxidation resistance.

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Conflicts of Interest

The authors declare no conflict of interest.