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The effect of hot plastic deformation on the structure and properties of surface-modified layers after non-vacuum electron beam surfacing of a powder mixture of composition 10Cr-30B on steel 0.12 C-18 Cr-9 Ni-Ti

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ABSTRACT

Introduction. Currently, austenitic *Ni-Cr* steels are widely used in the oil and gas industry for drilling wells due to its high corrosion resistance, non-magnetic properties, high impact strength, ductility and weldability. However, in order to increase the service life of products, it is necessary to increase the abrasive resistance of the surface layers while maintaining chemical resistance, which is a difficult technological task. The solution to such a problem can be the creation of sheet blanks "austenitic *Ni-Cr* steel - modified layer" subjected to hot plastic deformation. **The purpose of the work** is to study the effect of hot plastic deformation on the structure and phase composition of "modified layer – base metal" compositions obtained by the method of non-vacuum electron beam surfacing of a powder mixture of boron and chromium on austenitic *Ni-Cr* steel *0.12 C-18 Cr-9 Ni-Ti*. **Material and methods of research.** The work investigated specimens made of steel *0.12 C-18 Cr-9 Ni-Ti* with a modified *10Cr-30B* layer formed by non-vacuum electron beam surfacing of a powder mixture of chromium and boron, and subsequent hot plastic deformation at a temperature of 950 °C. The research methods are mechanical tests for microhardness, X-ray spectral analysis of the modified layer, metallographic studies, profile analysis, calculation of lattice parameters. **Results and discussion.** It is revealed that after deformation, defect-free compositions are obtained, the surface layer of which is a matrix composite material containing oriented chromium carbide particles with altered crystal lattice parameters. After plastic deformation, cracks and delamination are not recorded, which allows us to speak about the high quality of the "modified layer – base metal" compositions with increased hardness values exceeding 6.5 times as-delivered steel *0.12 C-18 Cr-9 Ni-Ti* (3…11 GPa and 2 GPa, respectively). In the modified layer, complex borides of type $(Fe_xCr_y)B$ are formed and located in a γ-solid solution of iron. The lattice parameter decreases for γ-iron from 3.588 Å to 3.580 Å, for boride parameter ***a*** from 5.126 Å to 5.111 Å, parameter ***c*** from 4.228 Å to 4.199 Å.

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Introduction

Non-vacuum electron beam surfacing makes it possible to obtain compositions consisting of a base metal and a surface-modified strengthening layer of various thicknesses containing boride particles. The thickness of the layer is regulated by the parameters [1–12].

In the process of non-vacuum electron beam surfacing, it is necessary to use flat blanks with a thickness of at least 9 mm as the base material, in order to exclude warping during surfacing of the powder mixture. The large thickness and shape of the surface to be strengthened limit the possibilities of various surfacing options. For example, for drilling wells in the oil and gas industry, telemetry systems are used to monitor the condition and direction of movement of drilling tools, the parts of which have a complex structure with cylindrical and shaped surfaces. Chromium-nickel austenitic steel with non-magnetic properties is used as a material for these parts, which are operated in conditions of corrosive environments and abrasive effects of rock particles. Increasing the hydroabrasion wear resistance of the surface layers of such products (the inner surfaces of body parts, pipes and valves of telemetry systems) while maintaining chemical resistance and the absence of magnetization is an important technical task [13–15].

The use of hot plastic deformation of compositions consisting of relatively ductile austenitic steel and a wear-resistant modified layer makes it possible to obtain thin-sheet products and products with shaped surfaces that will combine high corrosion and wear-resistant properties [16–20].

The purpose of this work is to study the effect of hot plastic deformation on the structure and phase composition of “modified layer – base metal” compositions obtained by non-vacuum electron beam surfacing of a powder mixture of boron and chromium on chromium-nickel austenitic steel *0.12 C-18 Cr-9 Ni-Ti*.

The goal requires solving the following tasks:

– to obtain blanks from austenitic stainless steel strengthened by the method of non-vacuum electron beam surfacing of powder mixtures *10Cr- 30B*;

– to evaluate the effect of the degree of plastic deformation on the structure and properties of boride layers;

– to investigate the effect of hot plastic deformation on the phase composition and lattice parameters of the modified layer.

Research methodology

To create a modified layer on *0.12 C-18 Cr-9 Ni-Ti* steel reinforced with boride particles, a powder mixture (Table 1) was surfaced with a beam of relativistic electrons released into the air atmosphere using an industrial electron accelerator *ELV-6* at the Budker Institute of Nuclear Physics SB RAS. The parameters of non-vacuum electron beam surfacing are presented in Table 2.

It should be noted that austenitic steel was used as a reference material in durometric studies.

After non-vacuum electron beam surfacing, the specimens were subjected to hot plastic deformation at 950 °C on a *Quarto* rolling mill with a working roll diameter of 330 mm, a speed of 60 mm/s, a deformation

Table 1

Composition of the powder mixture

Name of the powder system	The composition of the powder mixture, wt. %		
	Cr	*B*	MgF_2*
10Cr-30B	10	30	60
	The size of the powder particles, µm		
	5–20	40–80	200–300

* Since the surfacing is carried out without the use of vacuum and protective gases, MgF_2 was used as a flux.

Table 2

Modes of non-vacuum electron beam surfacing

Parameter	Meaning
The energy of the electron beam, *E*	1.4 MeV
Specific energy, *Ese*	6.44 kJ/cm^2
Powder weight per unit area, *m*	0.33 g/cm^2
The scanning frequency of the electron beam, *v*	50 Hz
The distance from the outlet to the workpiece, *h*	90 mm
The speed of movement of the table with the specimen, *V*	10 mm/s

step of 5 % and the minimum deformation degree of 30 %, the maximum degree of deformation of 80%. When deformed by less than 30 %, there were no significant changes in the structure and appearance of the workpieces. When deformed by 80 %, the maximum possible degree of plastic deformation is observed for the specimens "modified layer – base metal".

Based on the scientific literature, the temperature for plastic deformation was chosen as 950 °C as the minimum temperature to ensure the plasticity of the substrate (*0.12 C-18 Cr-9 Ni-Ti*) and the relative plasticity of the modified layer. In addition, deformation at 950 °C avoids overheating of the material and grain coarsening in the base metal.

Metallographic studies were performed using the *Axio Observer Z1m Carl Zeiss* microscope.

The determination of the phase composition and structure was studied at the station "*Rigid Fluoroscopy*" of the Siberian Center for Synchrotron and Terahertz Radiation at the G.I. Budker Institute of the INP SB RAS. Diffraction analysis was performed at room temperature in a translucent mode. The radiation energy was 56.35 keV, the beam size was 500×500 µm, and the distance to the studied material was 353 mm. The *Mar345* detector was used to register diffracted radiation. After the study, two-dimensional diffraction patterns were integrated using the open source *pyFAI* software [21].

The profile analysis of diffraction maxima was carried out using the *pseudo-Voight* function, with subsequent calculation of the crystal lattice parameter using the matrix method.

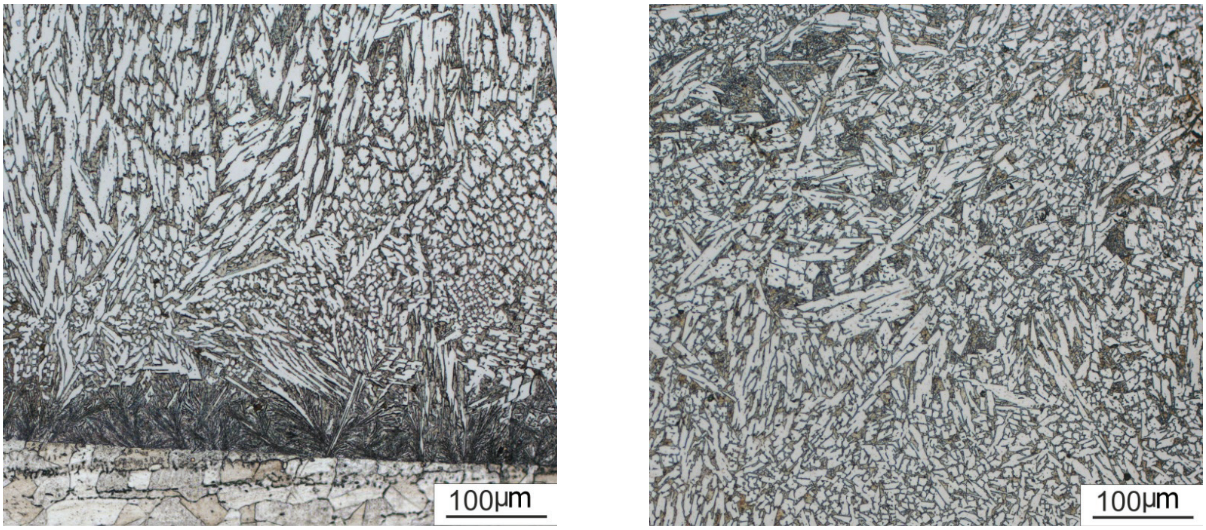
To identify the features of the boride particles location in the structure of the modified layer, a *Carl Zeiss EVO50 XVP* scanning microscope was used. The studies were carried out in the backscattered electron diffraction mode, the chemical composition was determined using the *EDX X-Act* energy dispersion analyzer.

The microhardness of the modified layers obtained was measured using the *Vickers* method in accordance with *GOST 9450-76* at a load of 0.98 N on a *Wolpert Group 402MVD* micro-hardness tester. At least 5 tracks with 10 indentations were made on each specimen.

Results and discussion

The most effective temperature range for deformation of chromium-nickel steel is 950–1,100 °C; it is in this temperature range that the processes of dynamic recovery and recrystallization have time to occur, and there are no local melting areas with defects that lead to destruction.

Figure 1 shows the structure of transverse sections of modified layers obtained after surfacing a powder mixture of composition *10Cr-30B*, which is a composite material with a dense arrangement of boride particles. A matrix composite material is understood to be a sample of a "modified layer – base metal" (Figure 1, *a*). Borides act as a strengthening phase in the modified layer. The density of the boride particles was assessed visually (Figure 1, *b*). The modified surface layer with a thickness of up to 2.5 mm is connected to the main material by a transition zone with a thickness of 100-150 microns. The structure of the modified layer contains borides that do not have the correct geometric shape, which can be explained by the collision of crystals during their growth. The transition layer is a eutectic, the components of which are austenite and boride crystals.



a *b*

Fig. 1. Structure of the specimens before plastic deformation:

a – transverse section; *b* – frontal section

According to experimental data, with an increase in the degree of plastic deformation of compositions, cracks and delamination are formed in relatively large boride particles, which can contribute to its spalling and the formation of defects in the form of discontinuities, although no defects in the structure were observed before deformation (Figure 2). With an increase in the degree of plastic deformation, the boride particles are crushed with its alignment in the rolling direction (Figure 2, *a*, *c*, *e*). The chaotic distribution of boride particles after non-vacuum electron beam surfacing (Figure 2, *a*) and the rolling texture with boride orientation (Figure 2, *b*, *e*) confirm the assumption above. Under the influence of high temperatures (950 °C) and large deformations (80 %), crushed borides that do not come into contact with the matrix material become sources of structural defects in the form of a grid of cracks and spalling during the preparation of thin sections (Figure 2, *b, d, f*).

In addition, after hot plastic deformation, with an increase in the degree of deformation, borides with an irregular geometric shape (Figure 2, *b*) become smoother due to high temperatures and partial diffusion of elements (Figure 2, *e*, *f*).

In accordance with scanning electron microscopy (Figure 3, *a, b*), the surface of the specimen after maximum plastic deformation is characterized by clearly marked traces of plastic flow and destruction of high-strength boride particles (Figure 3, *c*). Longitudinal broadening of the specimens is also observed (Figure 3, *g*) and the texture of the base metal *0.12 C-18 Cr-9 Ni-Ti* (Figure 3, *a*), which is the result of high ductility of steel and is further confirmed by X-ray phase analysis. Image analysis at maximum plastic deformation shows the presence of small cracks between the modified layer and the base metal in the transition zone (Figure 2, *b*).

A more detailed examination of the transition zone (Figure 3, *b*) reveals a deformation texture, multiple grooves and etching pits. In the modified layer, partial cracking of high-strength particles is observed simultaneously with its grinding (Figure 3, *c*). It can be assumed that such a structure is formed as a result of critical stresses and accompanying deformations. The thickness of the modified layer decreases from 2.5 mm (Figure 1, *a*) to 0.5 mm (Figure 3, *a*). During deformation, the composition acquires a complex layered morphology and decreases in thickness by 7–8 times (Figure 3, *a*).

The analysis of the results of the study showed that the plastic deformation of the composition begins with the base material, and then continues in the modified layer. Also, with an increase in the degree of compression, borides are crushed by a brittle mechanism, and the base metal and matrix of the modified layer are mainly viscous. The mechanism of destruction of high-strength boride particles and the matrix is the same at all degrees of deformation. Starting from 30 % deformation, the degree of crushing of boride particles and its partial destruction continues with an increase in subsequent rolling.

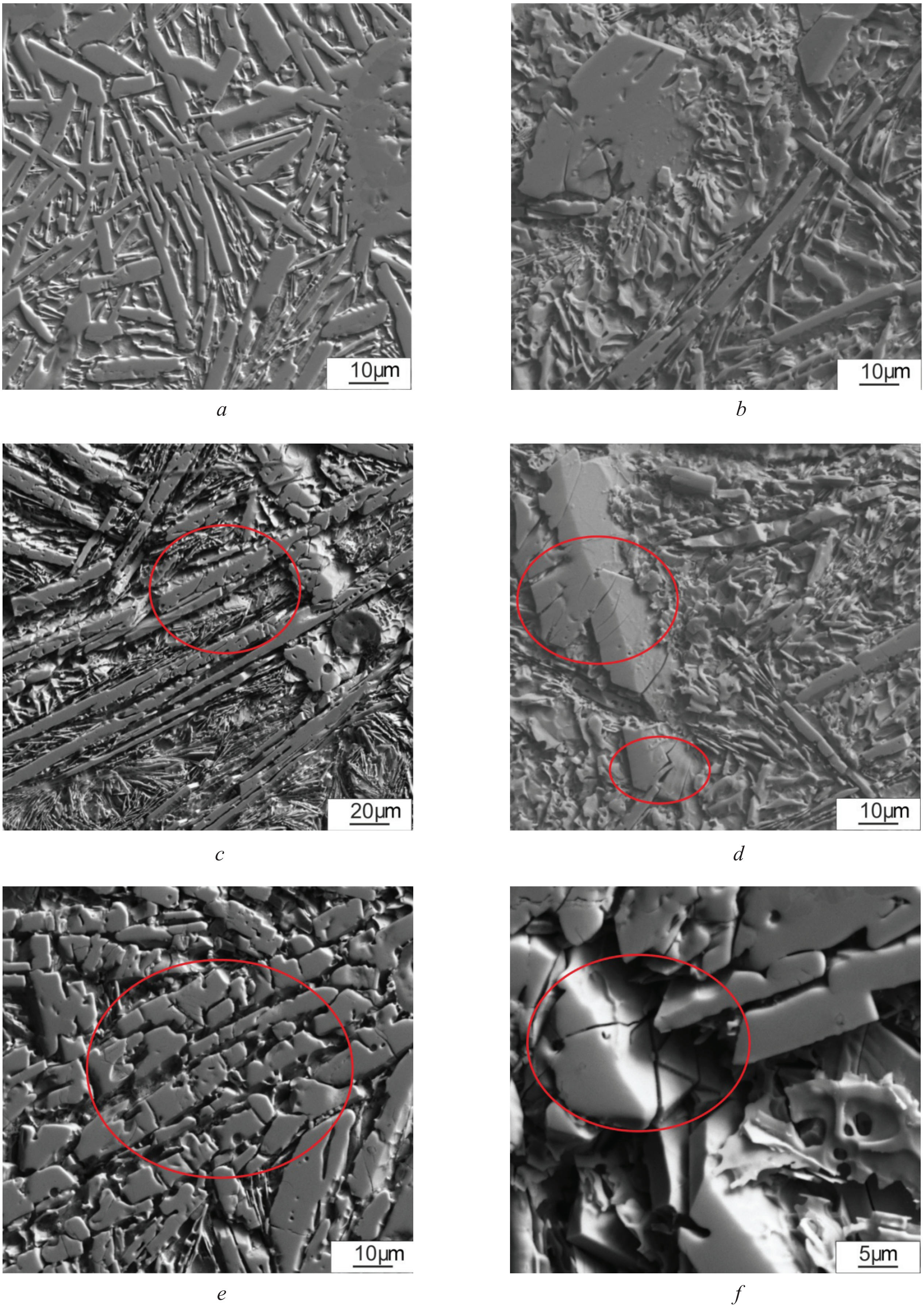


Fig. 2. Change in the shape of boride particles depending on the degree of plastic deformation:
a, *b* – before plastic deformation; *c*, *d* – 30 %; *e*, *f* – 80 %. The places of cracks, delamination and discontinuities in boride particles are highlighted in a red oval

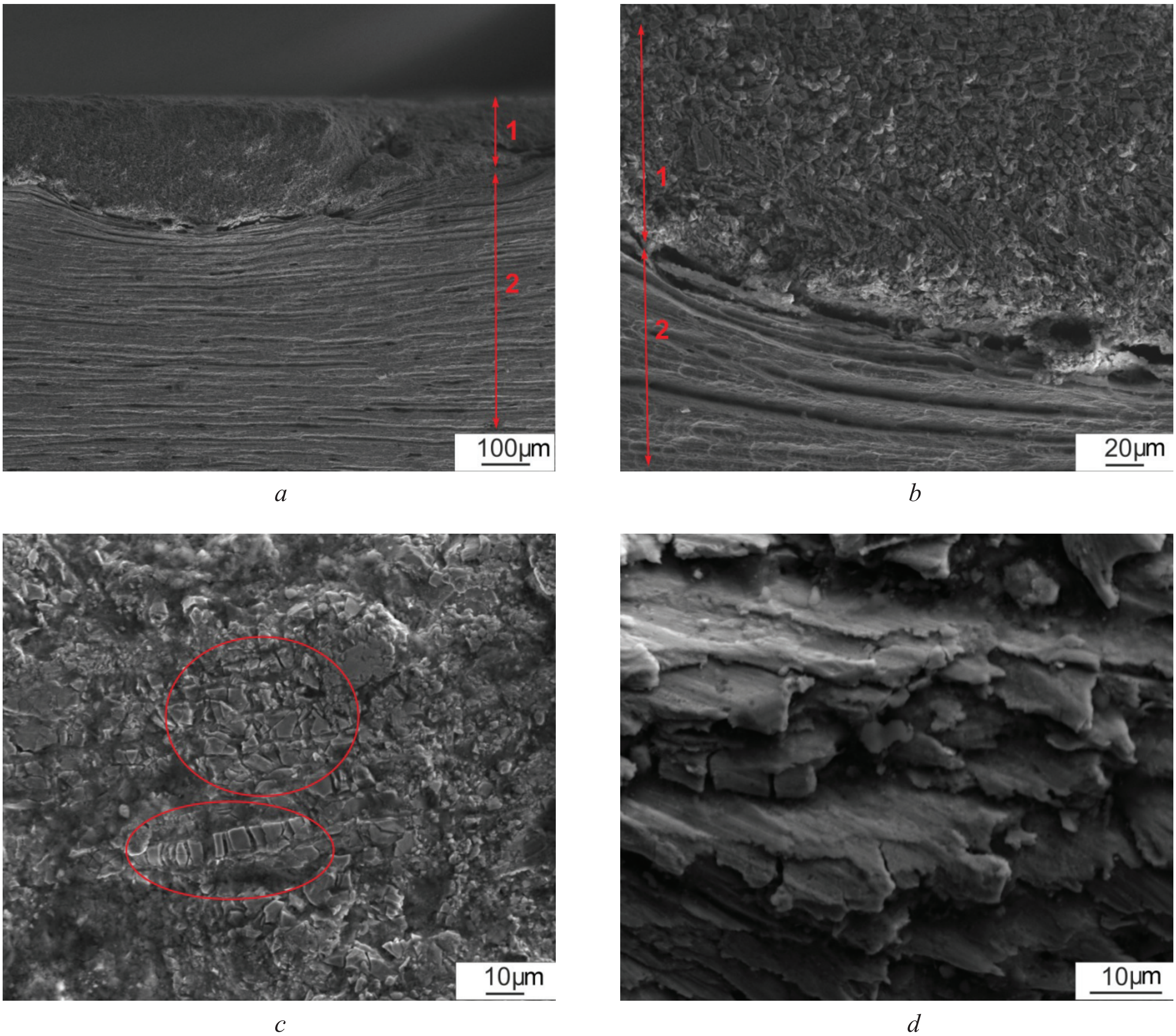


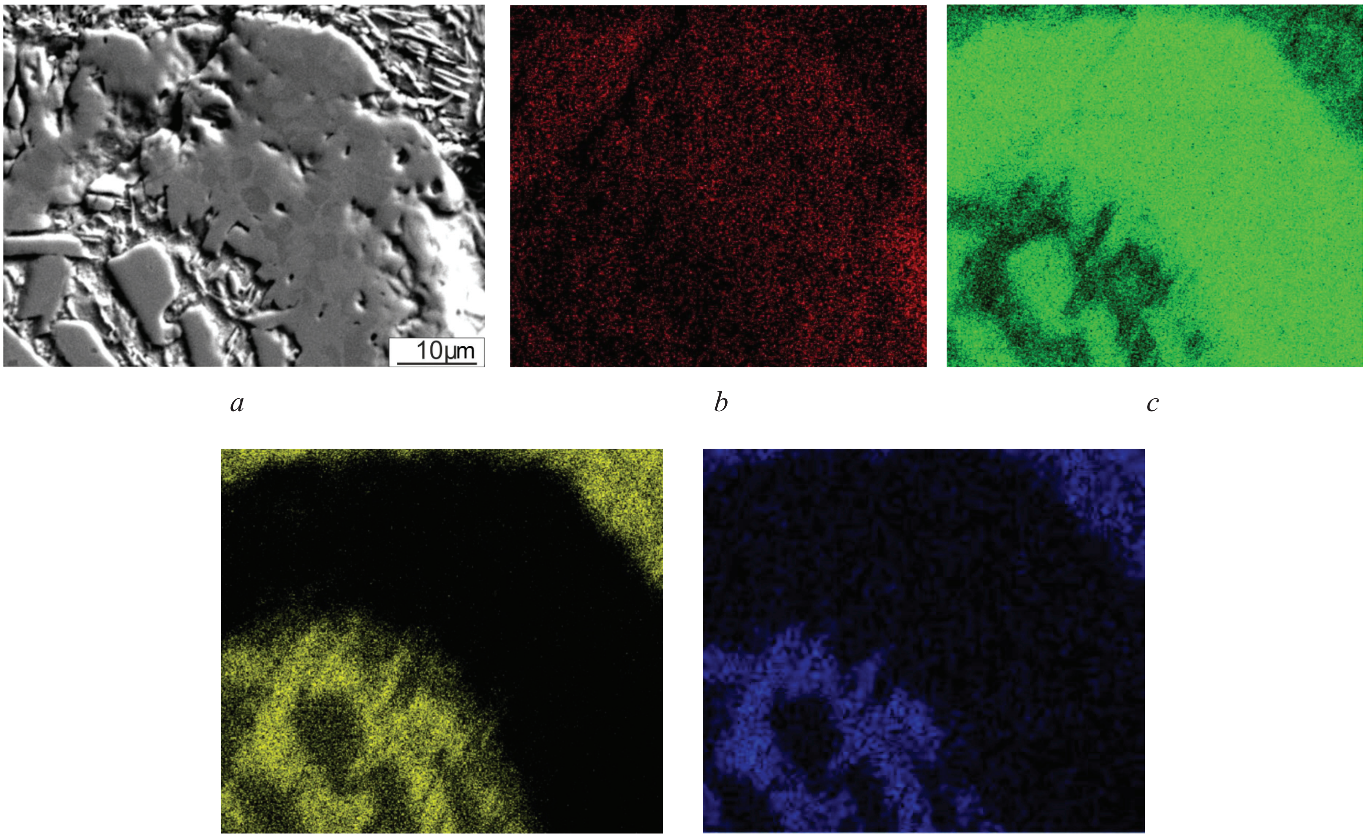
Fig. 3. The structure of the composition after 80 % deformation after etching with a mixture of HNO_3 and *HCl* acids in a ratio of 1:3 by volume, where ***1*** is the modified layer, ***2*** is the base metal:

a, *b* – the transverse section of the composition after rolling; *c* – the structure of the modified layer, with isolated destroyed borides after plastic deformation; *d* – morphology of the side surface of the base material *0.12 C-18 Cr-9 Ni-Ti* along the rolling direction

In addition, when examining boride particles in the austenite matrix using X-ray spectral analysis of the modified layer, high proportions of chromium in borides and iron with nickel in the matrix are recorded, this presumably indicates the diffusion of boron from borides into the matrix material (Figure 4).

To evaluate the mechanical properties, the microhardness of the specimens was measured. The average microhardness of the modified layer for specimens without deformation is 13 GPa, 12 GPa for of 30 % deformation, 11 GPa for 80 % deformation. The microhardness of chromium-nickel austenitic steel *0.12 C-18 Cr-9 Ni-Ti* is 2.3 GPa. It can be noted that the greater the degree of deformation of the composition, the lower the microhardness values, which is explained by the crushing of high-strength particles and the formation of a grid of small cracks between it with a lack of matrix material between it.

The phase composition of the specimens was determined using hard X-ray radiation with a wavelength of 1.783 Å. Only diffraction peaks of austenite and complex borides *(FexCry)B* phases are observed on X-ray images (Figure 5). The analysis of X-ray images allows us to roughly analyze the defect of the structure by broadening the X-ray lines. The peaks widen with an increase in the degree of plastic deformation. In this



a *b* *c*

d *e*

Fig. 4. Micro X-ray spectral analysis of specimens after 80 % deformation:
a – shooting surface; *b* – *B* distribution; *c* – *Cr* distribution; *d* – *Fe* distribution; *e* – *Ni* distribution

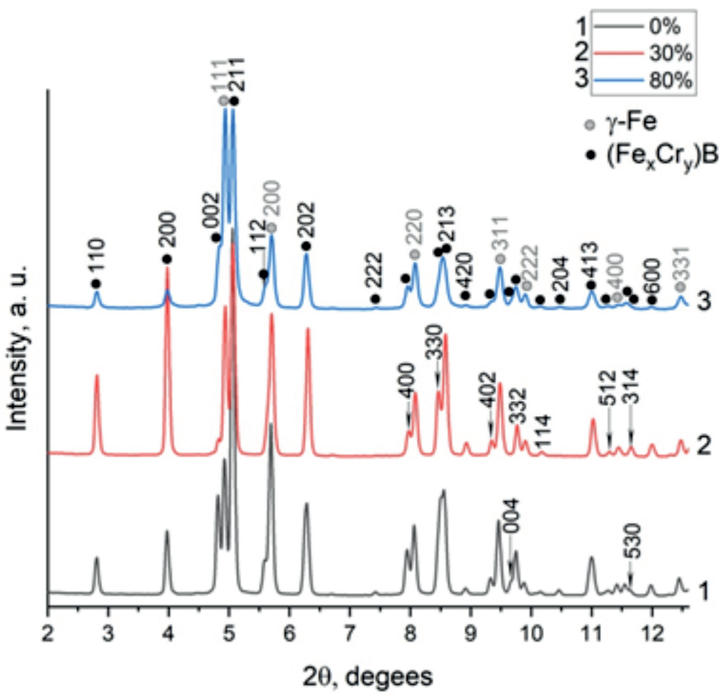


Fig. 5. Specimens after synchrotron radiation diffraction

case, it is necessary to note a decrease in the intensity of boride peaks relative to the intensity of austenite peaks and the textured nature of its distribution in the austenite matrix, as well as an increase in the intensity of diffraction peaks 200 and 202 at 30 % degree of deformation. In the process after non-vacuum electron beam surfacing and subsequent plastic deformation, a texture is present, which means that the specimens for synchrotron studies were cut and removed with accumulation in different currents and in some places there could be a more pronounced texture.

For borides, the most developed planes are the (110), (200), (002), (211) and (202) planes.

Estimating the width of the spectral line at half the height of its maximum or half-width allows only an approximate estimate of the defect structure of the strengthening phase of the modified layer due to the presence of many factors affecting the accuracy of the calculated parameters (possible boron diffusion from the strengthening phase during hot plastic deformation and the appearance of texture).

Table 3 shows the change in the *FWHM* of borides and the matrix of the material. The broadening of the interference lines may be related to the heterogeneity of the change in the interplane distance due to a change in the chemical composition of the phases.

Table 3

Change in *FWHM* diffraction patterns of modified layers after plastic deformation

The angular position is 2θ, deg.	*FWHM* at the degree of plastic deformation		
	0 %	30 %	80 %
For austenite			
4.94	0.068	0.070	0.076
5.71	0.073	0.079	0.091
8.08	0.075	0.077	0.092
9.48	0.077	0.080	0.089
9.90	0.076	0.084	0.081
For borides			
2.81	0.069	0.069	0.077
3.98	0.07	0.068	0.088
4.82	0.07	0.065	0.079
5.06	0.07	0.076	0.085
5.59	0.07	0.062	0.083
6.28	0.088	0.101	0.084

With a plastic deformation degree of 80 %, the *FWHM* values are maximal for both the matrix and borides. *FWHM* is minimal for the specimens before deformation when calculating the matrix and at 30% when calculating the borides. It can be assumed that with a degree of deformation of 30 %, the change in the peak half-width for borides is less intense than for austenite. This can be explained by the fact that the plasticity of the matrix is higher for austenite than for borides.

After plastic deformation, a decrease in the unit cell parameters is typical. Austenite is characterized by cubic syngony with the space group *Fm-3m* (225), while boride is characterized by tetragonal syngony with *I4/mcm* (140). The decrease in the parameters of the austenite unit cell can be explained by the fact that an ion with a larger radius is replaced by an ion with a smaller radius. At the same time, the volume of the boride unit cell changes, which in turn indicates an increase in the content of metal atoms in it (Table 4).

Conclusions

The results of the study of the effect of hot plastic deformation on the structure and properties of the composition of the "modified layer *10Cr-30B* – chromium-nickel austenitic steel *0.12 C-18 Cr-9 Ni-Ti*" obtained by the non-vacuum electron beam surfacing method allow us to draw the following conclusions:

1. Specimens of the "modified layer – base metal" are obtained using the technology of non-vacuum electron beam surfacing of powder compositions on the surface of steel *0.12 C-18 Cr-9 Ni-Ti* followed by hot plastic deformation at a temperature of 950 °C. The thickness of the modified layer is 2.5 mm after the non-vacuum electron beam surfacing, and about 0.5 mm after 80 % hot plastic deformation.

Table 4

Change in lattice parameter after hot plastic deformation

The lattice parameter	The lattice parameter for the degree of plastic deformation, Å		
	0 %	30 %	80 %
For austenite			
a	3.588	3.580	3.580
For borides			
a	5.126	5.113	5.111
c	4.228	4.238	4.199

2. The structure of the modified surface layer after hot plastic deformation is a composite material with dispersed particles of the strengthening phase in the form of borides $(Fe_xCr_y)B$. The transition layer between this material and the base metal has no cracks or pores. Borides are crushed during plastic deformation and oriented towards rolling. According to the results of durometric studies, it is found that the microhardness of the modified layers after deformation is 6.5–5.5 times higher (13–11 GPa) than the microhardness of the base material *0.12 C-18 Cr-9 Ni-Ti* (2 GPa), which acted as the reference material. To eliminate the spalling of particles of the strengthening phase of the modified layer, it is necessary to increase the content of matrix material in it by increasing the content of chromium in the powder mixture being surfaced and reducing the content of boron.

3. Synchrotron research methods show that complex borides of type $(Fe_xCr_y)B$ are formed in the modified layer, located in a **γ**-solid solution of iron. With an increase in the degree of plastic deformation, there is a broadening of the diffraction maxima and the volume of the elementary cells of austenite and borides increases due to the accumulation of defects in the crystal lattice.

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Conflicts of Interest

The authors declare no conflict of interest.