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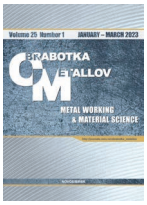

The effect of borocoppering duration on the composition, microstructure and microhardness of the surface of carbon and alloy steels

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ABSTRACT

Introduction. Borocoppering is one of the methods of thermochemical treatment (*TCT*) aimed at forming diffusion layers with high physical and mechanical properties on the surface of carbon and alloy steels. The thickness of the diffusion layer is the most important characteristic of the *TCT*, which determines the depth of hardening. Consequently, the intensity and main characteristics of the *TCT* (layer thickness, alloying element concentration profile) depend on the process conditions (temperature, duration, and amount of alloying element). **The purpose of this work** is to determine the temperature-time parameters of diffusion borocoppering, which contribute to the formation of diffusion layers with a maximum thickness. The paper considers the results of surface hardening of carbon and alloy steels (for example, *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)*, and *0.5C-Cr-Ni-Mn* steel) by high-temperature soaking in powder mixtures containing boron and copper. Borocoppering was carried out in sealed containers with the powder mixture consisting of boron carbide, copper oxide, and sodium fluoride as an activator at a temperature of 950 °C for 3–5 h. The resulting specimens with a diffusion layer were examined using an optical microscope and a scanning electron microscope (*SEM*); the microhardness, elemental and phase composition of the layers were also determined, as well as the roughness of the obtained surfaces. **Results and discussions.** The microstructure of the obtained diffusion layers is studied; diagrams of the changes in the layers' thickness and the microhardness distribution over the layers' thickness are shown. It is established that with an increase in the soaking time from 3 to 5 h, the thickness of the diffusion layer increases from 120 to 170 μm on *Steel 45 (0.45% C)*; from 110 to 155 μm on *Steel U10 (1.0% C)* and from 130 to 230 μm on *0.5C-Cr-Ni-Mn* steel. A gradual decrease in the concentration of boron and copper along the layer thickness from 15–16% and 2–3% on the surface, respectively, to zero values at the boundary with the base metal is revealed. It is established that borocoppering to the formation of more thick boride layers on the surface of carbon and alloy steels compared to pure boriding. Moreover, an increase in the duration of soaking during the process contributes to the greatest increase in the thickness of the layer on *0.5C-Cr-Ni-Mn* steel. A study of microgeometry is carried out, microtopographies and profilograms of specimens' surfaces are shown before and after borocoppering. It is established that the roughness after borocoppering increases by 2-3 times compared to the initial one, and an increase in the duration of the process does not have a significant effect on the roughness.

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Introduction

The tasks of improving the reliability, operability and durability of machine parts, structures and tools are among the priorities in science and technology. To solve these problems, it is necessary to develop and implement effective methods that can improve many operational characteristics (corrosion resistance and wear resistance) by surface hardening.

One of the most common methods of surface hardening is thermochemical treatment (*TCT*), which is aimed at improving a wide range of physical and mechanical properties during the operation of machine parts and tools. The essence of any *TCT* method consists in thermal and chemical effects on the material in order to change the composition, structure and properties of the surface layer.

From the analysis of the literature data, it follows that one of the most common methods of *TCT* is boriding [1–6]. The boriding process has been known for more than half a century, but is not widely used compared to carburizing [7–10], nitriding and nitrocarburizing [11–14]. As a result of saturation of iron-carbon alloys with boron, layers with high hardness (1,600–2,000 HV) are formed on the surface. The widespread use of boriding in mechanical engineering is limited by high brittleness and tendency to cracking of surface layers after various chemical and thermal processing methods [15–17]. There are several ways to reduce the brittleness of the boride layer:

1) obtaining single-phase layers consisting of Fe_2B phase;

2) obtaining thinner layers;

3) the use of such elements as chromium, copper, nickel, aluminum, etc. in the composition of the saturating mixture together with boron [21–24].

Of particular interest is one of the methods of *TCT – borocoppering*. This method is aimed at increasing the thickness of the diffusion layer, as well as increasing the plasticity of the diffusion layer. The authors of [21–23] found that an increase in the concentration of copper in the composition of the saturating mixture contributes to an increase in the thickness of the diffusion layer.

The purpose of this work is to determine the temperature-time parameters of diffusion borocoppering, which contribute to the formation of diffusion layers with a maximum thickness. The paper considers the results of surface hardening of carbon and alloy steels (for example, *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)*, and *0.5C-Cr-Ni-Mn* steel) by high-temperature soaking in powder mixtures containing boron and copper.

The purpose of this work is to study the structure of the diffusion layer depending on the duration of complex saturation of the surface of specimens made of *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)* and *0.5C-Cr-Ni-Mn* steel with boron and copper.

Research methodology

The diffusion saturation process was carried out in a powder medium. *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)* and *0.5C-Cr-Ni-Mn* steel were used as test specimens, the chemical composition of which is shown in Table 1.

Table 1

Chemical composition of *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)*, *0.5C-Cr-Ni-Mn*, wt.%

	C	Si	Mn	Ni	S	P	Cr	Cu	Fe	Mo
Steel 45 (0.45% C)	0.42–0.5	0.17–0.37	0.5–0.8	up to 0.25	up to 0.04	up to 0.035	up to 0.25	up to 0.25	~97	–
Steel U10 (1.0% C)	0.96–1.03	0.17–0.33	0.17–0.33	up to 0.25	up to 0.028	up to 0.03	up to 0.2	up to 0.25	~97	–
0.5C-Cr-Ni-Mn steel	0.5–0.6	0.1–0.4	0.5–0.8	1.4–1.8	up to 0.03	up to 0.03	0.5–0.8	up to 0.3	~95	0.15–0.3

The saturating mixture included powders of boron carbide, aluminum and copper oxide. Sodium fluoride acted as an activator of the saturation process.

The composition of the saturating mixture had the following percentage of components: 47 % B_4C + + 28 % CuO + 23% Al + 2 % NaF. The optimal amount of copper oxide was chosen based on the works [21–23], where diffusion layers with maximum thickness were obtained.

The prepared specimens were placed in a container, filled with a saturating mixture (Fig. 1, *a*) and placed in a muffle furnace (Fig. 1, *b*). To prevent oxidative processes, the lid of the container was sealed with fusible glass. Diffusion saturation was carried out at a temperature of 950 °C, for 3, 4 and 5 hours. Further, the container was cooled in air; specimens were extracted, cleaned out of the remnants of the saturating mixture. This was followed by the preparation of specimens for metallographic studies.

a *b*

Fig. 1. Packed containers (*a*), muffle furnace *EKPS-50* (*b*)

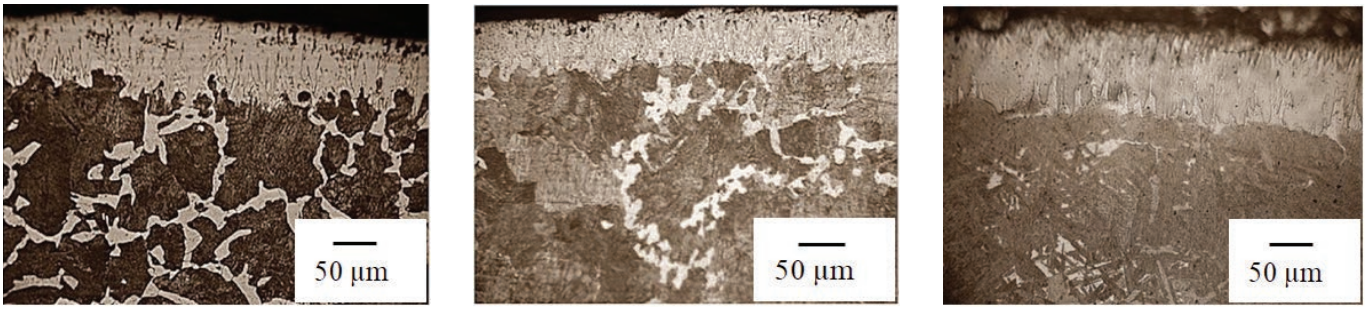
The specimens were fixed in clamps, then grinding and polishing were carried out. To identify the microstructure of the studied specimens, a chemically active solution consisting of nitric acid (4 %) and alcohol (the rest) was used. Metallographic studies were carried out on an optical microscope *Altami MET 2C*. Microhardness measurements were carried out on a *PMT-3M* microhardness meter, the load on the diamond pyramid was 50 g. Elemental analysis was conducted on a *JEOL JCM-6000* scanning electron microscope (*SEM*) with an elemental dispersion analyzer. To study the structure, the etched surface of the specimens was studied in the mode of secondary electrons. X-ray phase analysis was performed on a *D2 PHASER* diffractometer with a *LYNXEYE* linear detector. The measurement step was 0.02°, the processing time of one step was 1.2 s. The study of the topography with the determination of the surface roughness parameters of the obtained specimens was carried out on an optical profilometer *Bruker Contour GT-K1* with *Vision64* software [24, 25].

Results and discussion

As a result of diffusion surface saturation of specimens with boron and copper for 3 hours, diffusion layers with a thickness of 110–130 μm were obtained (Fig. 2).

After diffusion borocoppering for 4 hours, diffusion layers with a thickness of 140–220 μm were obtained on the surface of the specimens (Fig. 3).

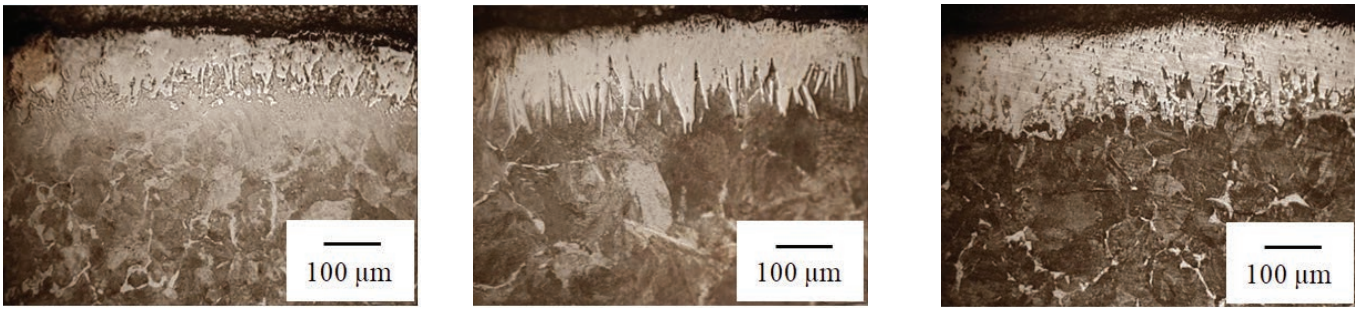
Fig. 2, *a* shows a 120 μm thick diffusion layer of *Steel 45 (0.45% C)* with a hardness of 1,800–1,600 HV. The diffusion layer has a needle-like structure typical of the boride layer. A characteristic feature is the deep insertion of needles into the steel base, which many authors point out as the reason for the strong adhesion of the diffusion layer to the metal base [26–29]. In this case, the needles at the ends have rounding. The carboboride phase is isolated directly from boride needles, the hardness of which was 1,200–1,750 HV. The transition zone between the layer and the steel base does not differ from the ferrite-pearlite structure of the base.



a *b* *c*

Fig. 2. Microstructure of the diffusion layer after complex surface saturation with boron and copper for 3 hours of soaking:

a – Steel 45 (0.45% C), layer thickness is 120 μm; *b – Steel U10 (1.0% C)*, layer thickness is 110 μm; *c – 0.5C-Cr-Ni-Mn* steel, layer thickness is 130 μm



a *b* *c*

Fig. 3. Microstructure of the diffusion layer after complex surface saturation with boron and copper for 4 hours of soaking:

a – Steel 45 (0.45% C), layer thickness is 160 μm; *b – Steel U10 (1.0% C)*, layer thickness is 140 μm; *c – 0.5C-Cr-Ni-Mn* steel, layer thickness is 220 μm

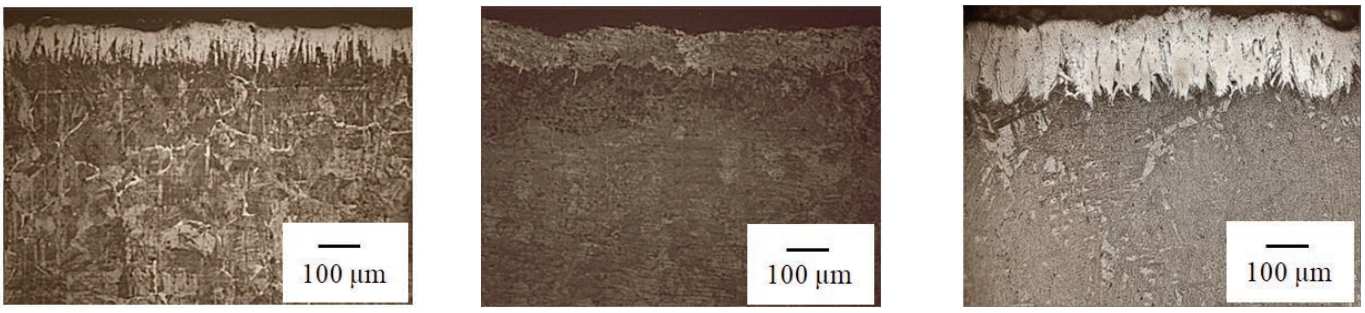
After borocoppering of *Steel 45 (0.45% C)* for 4 hours, the layer thickness was 140 μm, which is 20 μm greater compared to the soaking this steel for 3 hours (Fig. 3, *a*). The microhardness was 2,000 HV at the surface, followed by a decrease to 1,600 HV at the layer/base interface. There is a fusion of needles at the base with the formation of a continuous layer. There is no carboboride phase adjacent to the boride needles. The transition zone is more clearly represented in the form of a light ferrite layer, where the maximum concentration of boron reaches 4 %, and then it gradually decreases towards the core of the specimen. The steel structure retains proeutectoid ferrite (light inclusions); martensite with a small content of residual austenite is also observed.

On the surface of the carbon tool *Steel U10 (1.0% C)*, after 3 hours of *TCT*, a diffusion layer with a thickness of 110 μm was obtained, the hardness of which was 1,975–1,575 HV (Fig. 2, *b*). The layer consists of tightly pressed needles with an unexpressed transition zone, which is represented by perlite with low boron content. The steel structure consists of a plate-like perlite surrounded by a thin cementite mesh. It is necessary to note the presence of light coagulated inclusions, apparently being austenite.

After borocoppering of *Steel U10 (1.0% C)* for 4 hours, a diffusion layer with a thickness of 140 μm was obtained, which is 30 μm greater compared to the soaking this steel for 3 hours (Fig. 3, *b*). The microhardness also increased slightly to 2,050 HV at the surface, followed by a decrease to 1,600 HV at the layer/base interface. The microstructure indicates the fusion of needles and the formation of a continuous layer in the upper and middle part of the layer with the preservation of the needle structure at the layer/base interface. The presence of a transition zone is not observed, and the microstructure of the base metal is represented by a lamellar perlite with a cementite mesh.

Metallographic studies of the structure and diffusion layer of *0.5C-Cr-Ni-Mn* steel showed the presence of a diffusion layer with a thickness of 130 μm and 220 μm at 3- and 4-hour borocoppering, respectively (Fig. 2, *b*, Fig. 3. *b*). The microhardness was 1,800–1,500 HV at 3-hour soaking and 2,000–1,650 HV at 4-hour soaking.

When conducting diffusion saturation with boron and copper of specimens from *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)* and *0.5C-Cr-Ni-Mn* steel for 5 hours, an increase in the thickness of the diffusion layer by 10–15 μm is observed (Fig. 5). Fig. 4, *a* shows the structure of *Steel 45 (0.45% C)*, where, in contrast to the previous borocoppering modes, the layer has a pronounced needle-like structure in the form of enlarged needles with a rectilinear direction to the core of the specimen. There is an increase in microhardness in the near-surface part of the layer, where its maximum value is 2,100 HV (Fig. 6, *a*).



a *b* *c*

Fig. 4. Microstructure of the diffusion layer after complex surface saturation with boron and copper for 5 hours of exposure:

a – Steel 45 (0.45% C), layer thickness is 170 μm; *b – Steel U10 (1.0% C)*, layer thickness is 155 μm; *c – 0.5C-Cr-Ni-Mn* steel, layer thickness is 230 μm

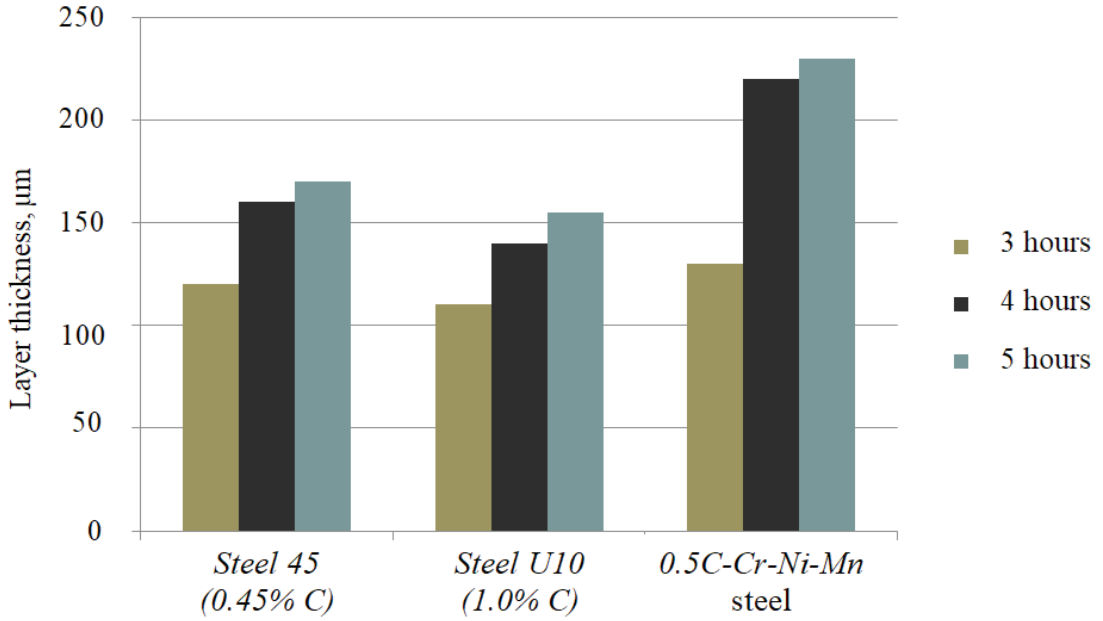
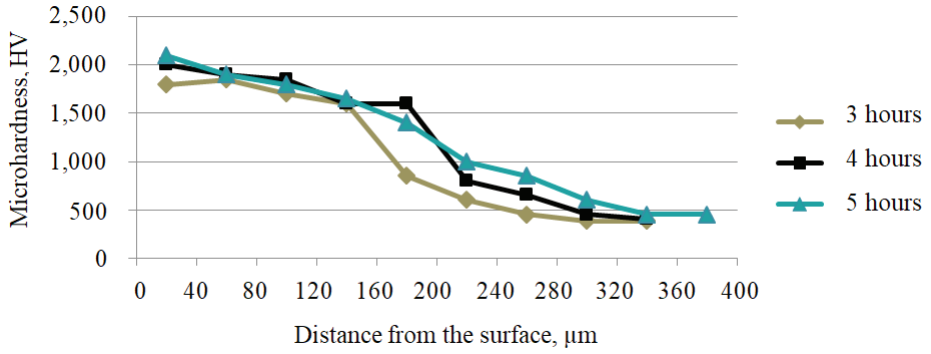


Fig. 5. The thickness of the diffusion layer formed after borocoppering of *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)*, and *0.5C-Cr-Ni-Mn* steel for 3, 4 and 5 hours

On the surface of the specimens from *Steel U10 (1.0% C)*, after 5 hours of borocoppering, the diffusion layer loses its needle-like structure and takes the form of a continuous layer, as evidenced by Fig. 4b. The increase in thickness was 15 μm, and the maximum value of microhardness was equal to 2,000 HV (Fig. 6, *b*).



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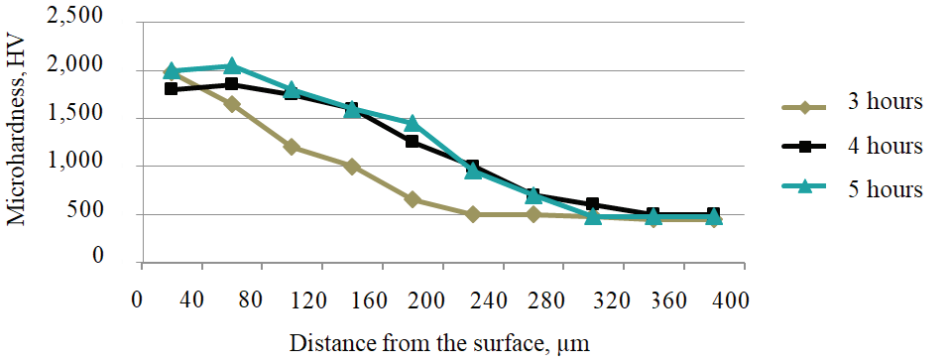

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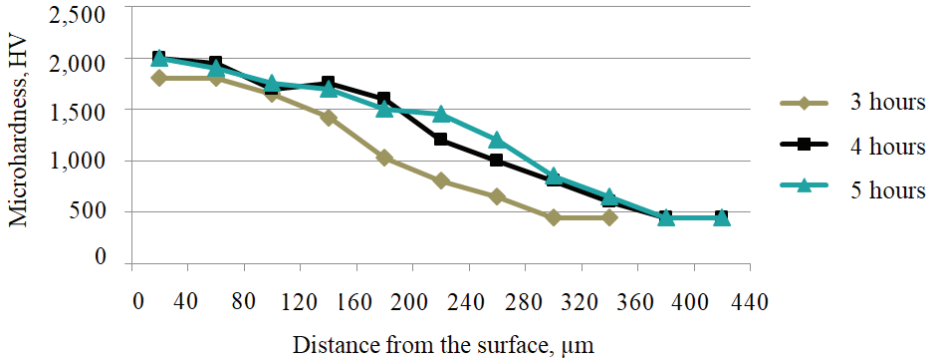

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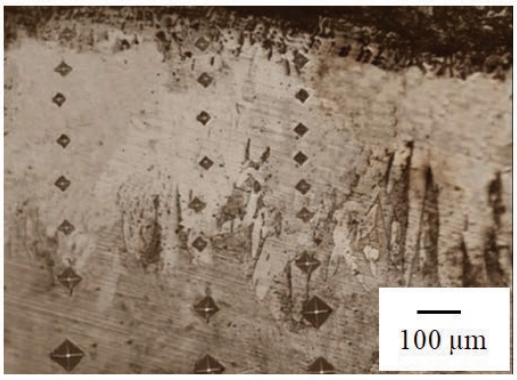


d

Fig. 6. Microhardness distribution:
a – for *Steel 45 (0.45% C)*; *b* – for *Steel U10 (1.0% C)*; *c* – for *0.5C-Cr-Ni-Mn* steel; *d* – microstructure of *Steel 45 (0.45% C)* after 4 hours of soaking with points of indentation

Minor changes in the structure of the diffusion layer after 5-hour borocoppering undergo specimens of *0.5C-Cr-Ni-Mn* steel (Fig. 4, *c*). The thickness of the layer was increased by 10 μm (Fig. 5). The needle-like structure of the layer remains unchanged, but the needles' growth is observed. It is also worth noting that directly adjacent to the boride needles are some secretions, presumably of a carboboride structure, which have a direction at some angle relative to the needles themselves. The microhardness and the nature of its distribution remain unchanged (Fig. 6, *c*).

An increase in the carbon content in *Steel 45 (0.45% C)* and *Steel U10 (1.0% C)* reduces the average layer thickness at both soaking modes. The thickness of the layer is greatest on *0.5C-Cr-Ni-Mn* steel specimens, despite the intermediate carbon content (Fig. 4). It is likely that alloying elements in steel take part in the intensification of diffusion during borocoppering.

As can be seen in Fig. 6, the distribution of microhardness after borocoppering for 3, 4 and 5 hours on all steels is similar and is characterized by a gradual decrease in values from the surface to the base metal. It should be noted that the microhardness of all specimens over the entire thickness of the layer after 5-hour borocoppering is higher by 100–150 HV, compared with the microhardness of specimens after borocoppering for 3 and 4 hours. Presumably, this is due to an increase in the content of the harder phase of *FeB* after a 5-hour borocoppering.

The data given in Table 2 (Fig. 7, *a*) confirm the presence of boron and copper in the diffusion layer on the test specimen made of *Steel 45 (0.45% C)*. There is a decrease in the concentration of boron and copper in the direction from the surface to the interface with the base metal. Carbon is pushed into the transition zone, where its concentration is maximum – 0.56%. Nickel and manganese are almost evenly distributed over the entire thickness of the diffusion layer. The presence of chromium was detected in the transition zone. Consequently, the elemental analysis shows the nature of the distribution of alloying elements corresponding to the chemical composition of *Steel 45 (0.45% C)*.

The results presented in Table 3 (Fig. 7, *b*) for *Steel U10 (1.0% C)* indicate the presence of boron on the surface in the amount of 16.81 % and a gradual decrease in its concentration to 0.68 %. The maximum amount of copper is observed on the surface of the diffusion layer and directly under the boride needles. Carbon is pushed under the boride layer, where its content reaches 1.69 %. Chromium and manganese are evenly distributed over the entire thickness of the diffusion layer.

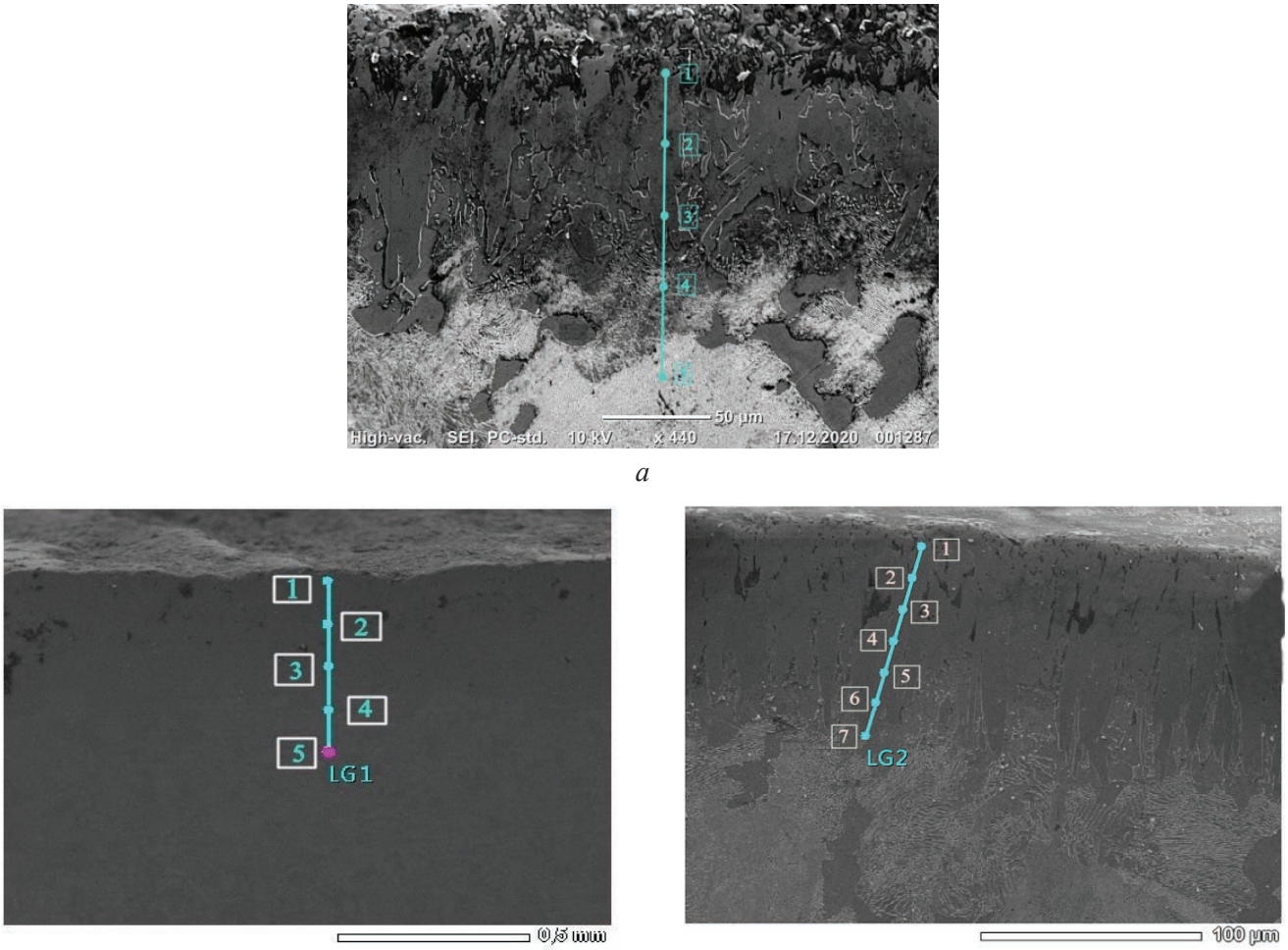
Table 4 shows the elemental composition of *0.5C-Cr-Ni-Mn* steel after borocoppering for 4 hours (Fig. 7, *c*). As in the previous specimens, the maximum concentration of boron is observed on the surface, followed by its decrease towards the boundary with the base. The maximum carbon concentration is visible on the surface and in the transition zone. Aluminum, chromium, nickel, molybdenum and copper are concentrated in the same zones as carbon.

X-ray phase analysis performed on the surface of *Steel 45 (0.45% C)* (see Fig. 8) after borocoppering demonstrates the presence of phases *FeB*, Fe_2B. The inability to determine copper is most likely due to its small amount.

Table 2

The elemental composition of the diffusion layer on *Steel 45 (0.45% C)* after 4 hours of borocoppering (Fig. 7, *a*)

Points of the spectrum	Chemical elements, mass %						
	B	*C*	*Mn*	*Ni*	*Cr*	*Cu*	*Fe*
1	16.73	0.2	0.29	0.41	–	2.39	79.98
2	11.37	0.06	0.38	0.44	–	–	87.75
3	6.2	0.32	0.22	0.51	–	–	92.75
4	–	0.56	0.24	0.31	–	0.36	98.53
5	–	0.47	0.35	0.51	0.12	0.17	98.38



a

b *c*

Fig. 7. The points of the spectra counting in the diffusion layer on the sample during elemental analysis: *a – Steel 45 (0.45% C)*; *b – Steel U10 (1.0% C)*; c – *0.5C-Cr-Ni-Mn* steel after 4 hours of borocoppering

Table 3

The elemental composition of the diffusion layer on *Steel U10 (1.0% C)* after 4 hours of borocoppering (Fig. 7. *b*)

Points of the spectrum	Chemical elements, mass %								
	B	*C*	*Al*	*Si*	*Cr*	*Mn*	*Ni*	*Fe*	*Cu*
1	14.81	0.83	0.22	0.16	0.34	0.57	–	81.18	1.89
2	12.73	0.43	0.19	0.17	0.15	0.08	–	85.68	0.57
3	6.91	0.61	0.06	0.11	0.09	0.55	–	83.34	–
4	0.68	1.22	–	0.34	0.23	0.32	0.11	95.91	1.19
5	–	1.69	–	0.28	0.12	0.3	–	97.61	–

The X-ray obtained on *Steel U10 (1.0% C)* (see Fig. 9) demonstrates the presence of the Fe_2B phase and the Fe_3C carbide phases. It is worth paying attention to the absence of the *FeB* phase. The presence of copper is also not observed.

As a result of X-ray phase analysis of a *0.5C-Cr-Ni-Mn* steel specimen (see Fig. 10) the phase composition of the boride layer was established, which consists of three borides: *FeB*, Fe_2B and Cr_5B_3. It should be noted

Table 4

The elemental composition of the diffusion layer on *0.5C-Cr-Ni-Mn* steel after 4 hours of borocoppering (Fig. 7, *c*)

Points of the spectrum	Chemical elements, mass %							
	B	*C*	*Al*	*Cr*	*Ni*	*Cu*	*Mo*	*Fe*
1	16.43	0.35	0.3	0.66	0.67	2.6	0.57	78.42
2	14.77	0.15	0.51	0.66	0.67	-	0.14	83.1
3	12.05	0.06	–	0.53	0.51	0.51	0.27	86.07
4	5.98	0.03	–	0.62	0.31	–	0.34	92.72
5	1.35	0.41	–	0.63	0.46	–	0.25	96.9
6	0.21	0.37	0.56	0.59	0.57	0.09	0.07	97.54
7	–	0.4	0.58	0.4	0.56	0.54	–	97.52

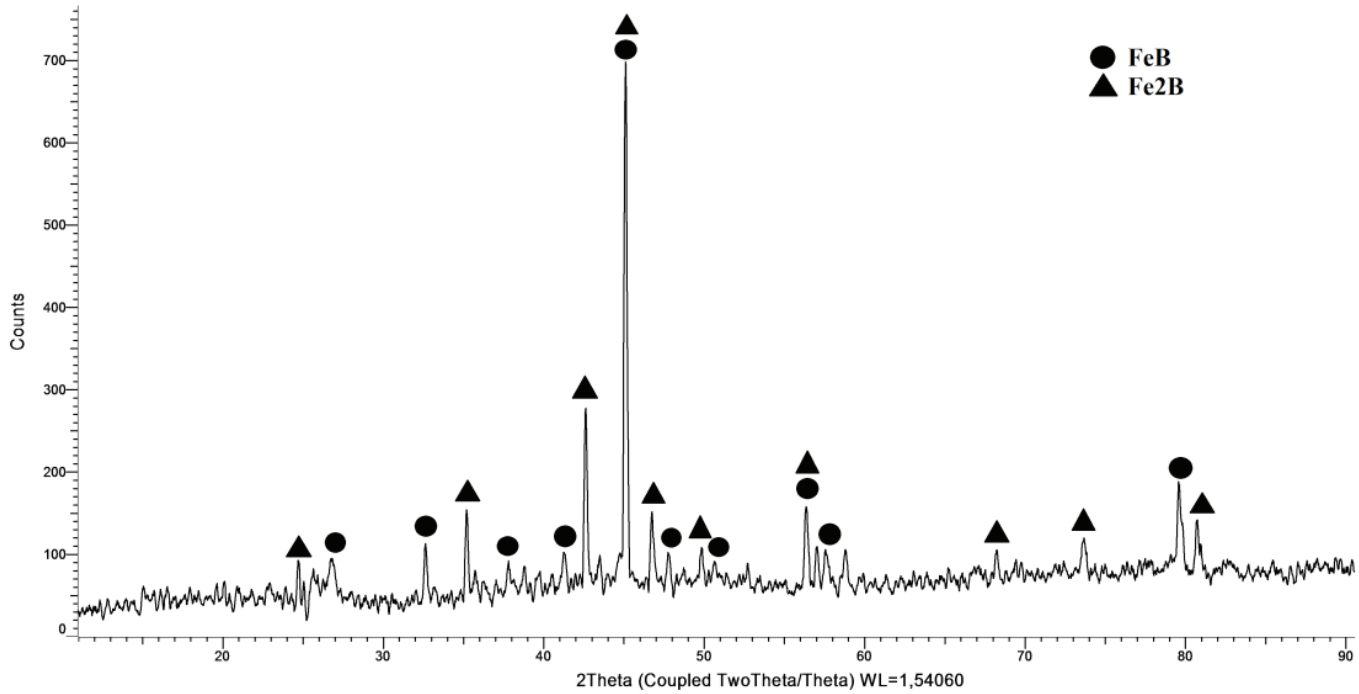


Fig. 8. XRD pattern of the specimen of *Steel 45 (0.45% C)* after borocoppering for 4 hours

that copper was detected in free form, which confirms the assumptions indicated in [21], where it does not form thermally stable compounds with boron, iron and carbon.

As a result of the study of microgeometry, three-dimensional microtopographs were obtained, as well as profilograms of the surfaces of the specimens after *TCT* (see Fig. 11–13). Roughness was estimated by the parameter *Ra* (Table 5).

The roughness of *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)* and *0.5C-Cr-Ni-Mn* steel in the initial state before the *TCT* was comparable and the *Ra* values are in the range of 0.06–0.084 μm (Fig. 11, *a*, 12, *a*, 13, *a*). After *TCT*, there is an increase in the heights of micro-dimensions compared to the initial specimens for all the materials under consideration and the processing time (Fig. 11, *b–d*, 12, *b–d*, 13, *b–d*). After borocoppering, an increase in the *Ra* parameter was established by 2–3 times compared to the initial specimens

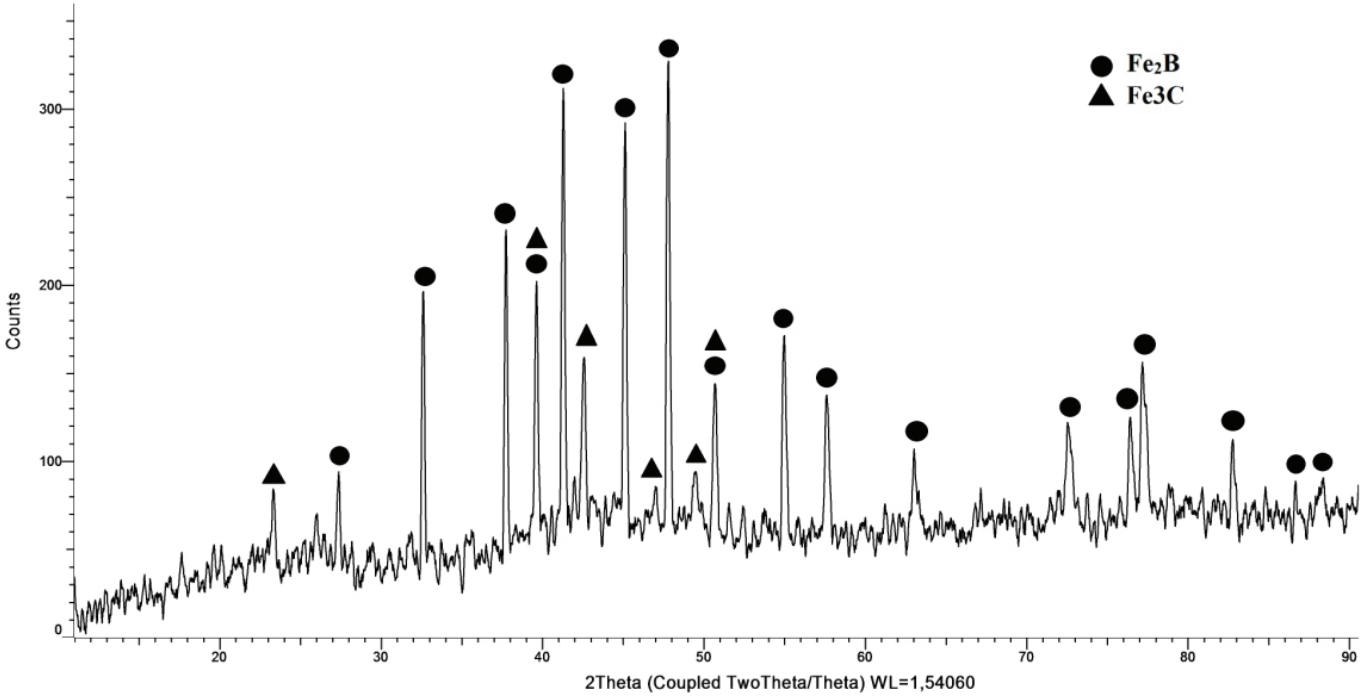


Fig. 9. XRD pattern of the specimen of *Steel U10 (1.0% C)* after borocoppering for 4 hours

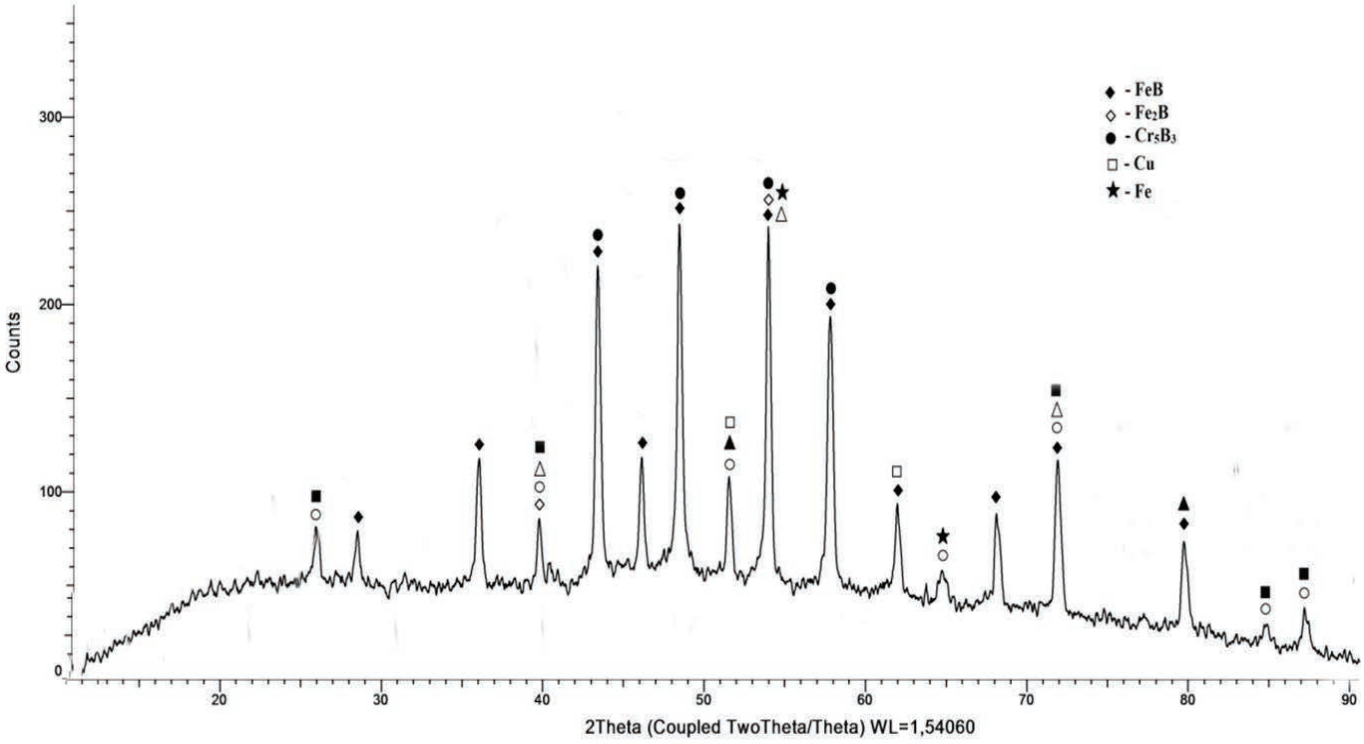


Fig. 10. XRD pattern of the sample made of *0.5C-Cr-Ni-Mn* steel after borocoppering for 4 hours

before treatment, while an increase in the *TCT* time from 3 to 5 hours does not lead to a further increase in roughness (Table 5).

The resulting roughness after borocoppering (*Ra* 0.16–0.2 μm), with an initial *Ra* of 0.06–0.08 μm, meets the requirements for surface roughness of most mechanical engineering products and does not require additional subsequent machining.

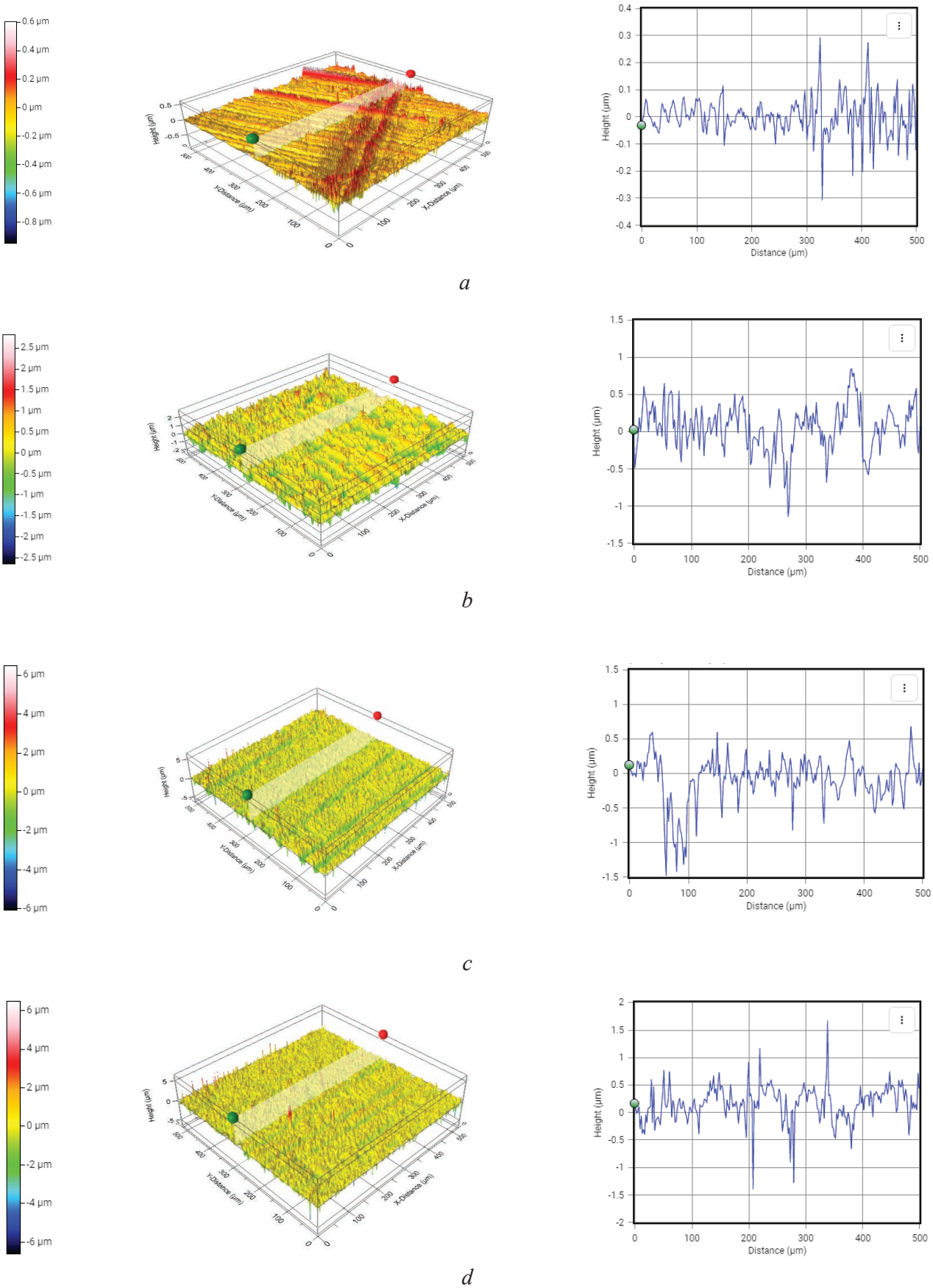


Fig. 11. Microtopography of the surface of *Steel 45 (0.45% C)* specimens:
a – initial, without treatment; *b* – after borocoppering for 3 hours; *c* – after borocoppering for 4 hours; *d* – after borocoppering for 5 hours

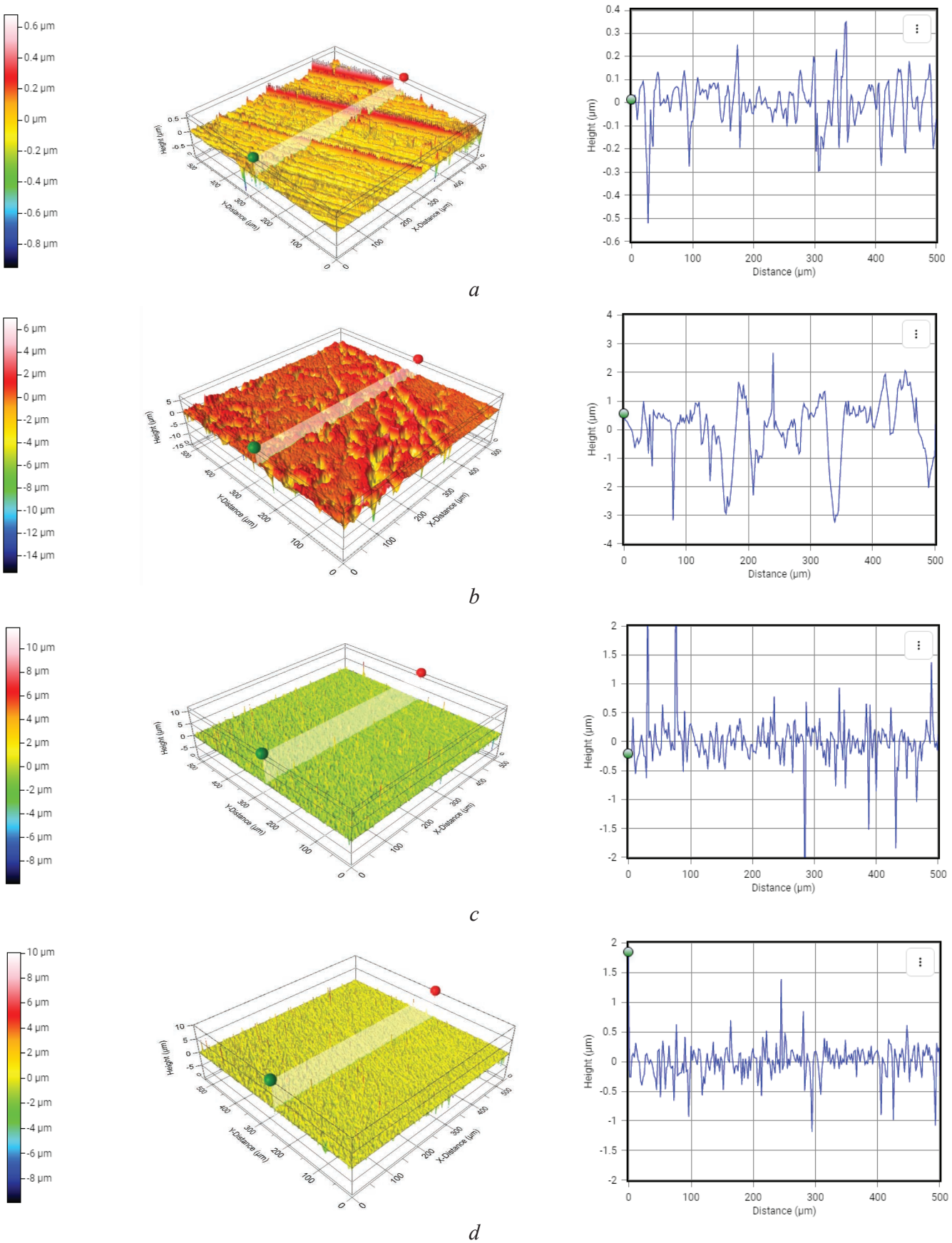


Fig. 12. Microtopography of the surface of *Steel U10 (1.0% C)* specimens:
a – initial, without treatment; *b* – after borocoppering for 3 hours; *c* – after borocoppering for 4 hours; *d* – after borocoppering for 5 hours

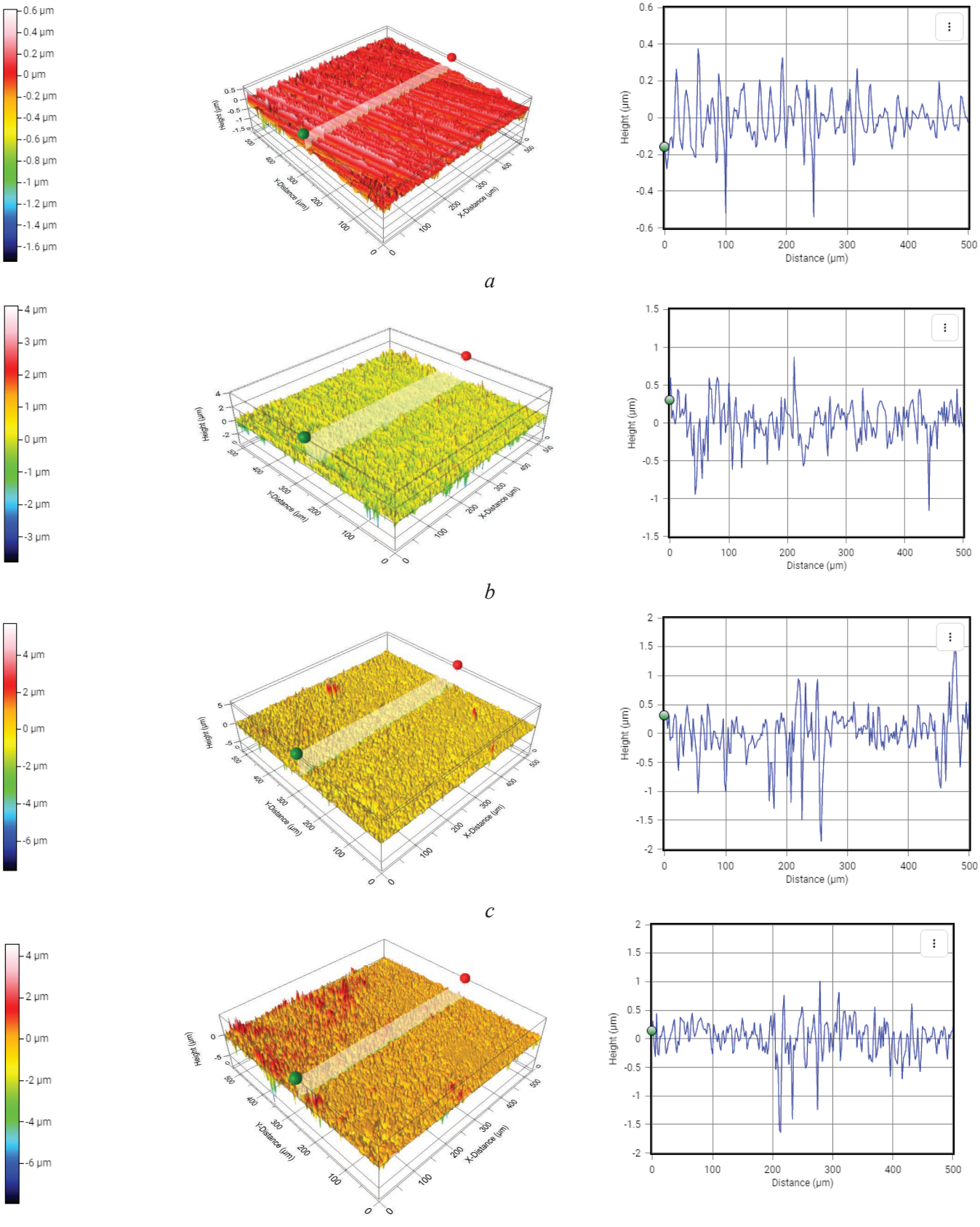


Fig. 13. Microtopography of the surface of *0.5C-Cr-Ni-Mn* steel specimens:
a – initial, without treatment; *b* – after borocoppering for 3 hours; *c* – after borocoppering for 4 hours; *d* – after borocoppering for 5 hours

Table 5

Roughness of the specimens after TCIT (Fig. 11-13)

Type of teatment	*Steel 45 (0.45% C)*	*Steel U10 (1.0% C)*	*0.5C-Cr-Ni-Mn* steel
	Ra, мкм		
Original, without teatment	0.06	0.062	0.084
Borocoppering for 3 hours	0.2	0.187	0.175
Borocoppering for 4 hours	0.16	0.201	0.273
Borocoppering for 5 hours	0.176	0.189	0.211

Conclusion

Based on the conducted studies, it is found that saturation of specimens from *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)* and *0.5C-Cr-Ni-Mn* steel for 3, 4 and 5 hours leads to the formation of diffusion layers, the thickness of which varies from 110 to 230 μm.

It is found that the increase in the thickness of the diffusion layer on *Steel 45 (0.45% C)* is 41%, with an increase in the time of treatment by 2 hours. On *Steel U10 (1.0% C)* and *0.5C-Cr-Ni-Mn* steel, the values of the layer thickness increase were 40 and 77 %, respectively. For these grades of steels, a longer soaking time during borocoppering is recommended.

It is established that during the diffusion boromedning for 4 hours, the greatest increase in the thickness of the diffusion layer is observed.

The study of microtopography revealed that the roughness after borocoppering increases to *Ra* 0.16–0.2 μm at the initial *Ra* 0.06–0.08 μm for *Steel 45 (0.45% C)*, *Steel U10 (1.0% C)* and *0.5C-Cr-Ni-Mn* steel, while the duration of the process does not affect the increase in roughness.

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Conflicts of Interest

The authors declare no conflict of interest.