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Features of the formation of Ni-Cr coatings obtained by diffusion alloying from low-melting liquid metal solutions

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ABSTRACT

Introduction. The main ways to increase steel parts properties are considered. The rationale for choosing *Ni* and *Cr* as the main components of the coating is given. The technology of diffusion alloying from low-melting liquid metal solutions (*DALMMS*) is given. **The purpose of this work** is to identify the features of the coatings formation with simultaneous diffusion saturation of nickel and chromium structural steels using the *DALMMS* technology. **Methodology.** Cylindrical specimens with a diameter of 20 mm and a length of 30 mm were subjected to *DALMMS*. The specimens were manufactured of the following structural steels: carbon steel *St3*, alloyed carbon steels *40Cr*, *40Cr13*, and extrafine steel *30CrMnSiNi2*. As a technological medium with *DALMMS* (transport melt), a *Pb-Li* eutectic melt with the specified content of *Ni* and *Cr* was used. Metallographic studies were carried out on microsections prepared according to the standard methodology. Studies to determine the thickness of coatings and its structure were carried out on the *Dura Scan Falcon 500* microhardness tester. The elemental composition of the coatings was determined by the method of X-ray microanalysis on a *Tescan Lyra 3* scanning electron microscope with the *Oxford Ultim MAX PCMA* system. **Results and discuss.** It is revealed that the formation of diffusive *Ni-Cr* coatings occurs with *DALMMS*. With *DALMMS* of structural steels contained carbon in cementite form two-layers coatings are formed: surface carbide layer and transition solid-soluble one. At the same time, the chromium content in the surface layers reaches 80 % with a nickel content of 1.5 %. The maximum *Ni* concentration is observed in the transition layer and amounts to 21 % at a depth of 5 μm on steel *30CrMnSiNi2* and 13 % at a depth of 4.5 μm for steel *40Cr*. Carrying out the *DALMMS* on steels containing carbon in the form of chromium carbides, or containing carbon in small amounts, leads to the formation of single-layer coatings based on solid solutions. The *Ni* content in the coating reaches 40 %, the chromium content for steel *St3* is 14.5 %; for steel *40Cr13* it was 9 %.

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Introduction

One of the leading scientific and technical problems in modern mechanical engineering is the development of technologies for applying functional coatings on the surface of steel products [1–5]. The properties of the surface layers, such as wear resistance, corrosion resistance, crack resistance, and strength, are determined by the composition and structure of the surface layers of products. The impact of surface layers on the corrosion resistance of products is especially important [6]. The application of corrosion-resistant coatings

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enables the use of structural carbon, low- and medium-alloy steels instead of high-alloy ones in equipment operating in aggressive environments.

With regard to increase the corrosion resistance of structural materials, elements such as *Ni*, *Cr*, *Al*, *Ti*, *Cu*, etc. are of interest [7–9]. At the same time, the current trend in the formation of functional coatings is the use of elemental compositions based on two or three or more components [10]. From the variety of possible elemental compositions, it is worth highlighting compositions based on nickel and chromium. Nickel forms a continuous series of solid solutions with iron. Nickel coatings are often used as corrosion-resistant, heat-resistant, high-temperature resistant ones. Chromium also forms a continuous series of solid solutions with iron. However, chromium is a carbide-forming element, which allows it to be considered not only for elements that form corrosion-resistant coatings, but also wear-resistant ones. Therefore, the formation of coatings based on the *Ni-Cr* composition is promising.

There are numerous techniques for forming coatings based on *Ni* and *Cr*, namely: *CVD*, *PVD*, flame spraying, galvanic methods, and thermochemical treatment (*TCT*) [11–15]. At the same time, it is worth paying attention to *TCT* technologies in connection with the following technological capabilities: simplicity of processing methods; the ability to coat products of complex shapes; a high degree of compatibility of the coating and the material being coated; ensuring a smooth gradient of concentration and properties from the coating to the material being coated [16–17]. Among the promising *TCT* technologies, *diffusion alloying from the medium of low-melting liquid metal solutions (DALMMS)* stands out. The technology is based on the phenomenon of isothermal mass transfer of coating elements in a medium of low-melting metals [18–19]. The melt of low-melting metal acts as a transport medium for bringing the diffusant to the coated product's surface. Simultaneously, the melt elements are unable to diffuse into the specimen being coated. Isothermal mass transfer of a diffusing element consists of the following elementary processes: dissolution of diffusing elements in the melt; its diffusion to the coating material in the liquid phase; adsorption on the surface; diffusion in the solid phase.

Based on previous studies, it is worth noting that an important factor influencing the formation of a coating is the elemental composition of the materials being coated. The microstructure of the specimens has less influence on the formation process due to the fact that the *DALMMS* technology is characterized by high temperatures (900–1,100 °C) and duration (60–600 minutes), which causes phase transformations to occur in the specimen, bringing the structure to an equilibrium state.

The formation of coatings based on two-component diffusion saturation with nickel and chromium using *DALMMS* technology has not been extensively studied. Previous studies indicate that coatings were formed on materials like Armco, *Steel 10*, *Cr6WV*, *12Cr18Ni10Ti* [20, 21].

The purpose of this work is to identify the features of the coatings formation with simultaneous diffusion saturation of nickel and chromium structural steels using the *DALMMS* technology.

The research objectives are as follows:

- 1) to identify the influence of the elemental composition of steels on the structure of the surface layers of the coated specimens after *DALMMS* process.
- 2) to identify the influence of the elemental composition of steels on the microhardness of the coatings obtained.
- 3) to identify the influence of the elemental composition of steels on the concentration distribution of coating elements.

Methods

Cylindrical specimens with a diameter of 20 mm and a length of 30 mm were subjected to *DALMMS*. The composition of the steels is shown in table.

These steels were selected due to the possibility of detecting the influence of carbon and other alloying elements (in particular, chromium, nickel) on the *DALMMS* process.

The *DALMMS* was carried out in a facility developed by our team, in a *Pb-Li* eutectic melt. Chromium powder (5 wt. %) and nickel powder (10 wt. %) were added to the melt [20]. The coated products were kept

Elemental composition of specimens

Steel grade	C	Si	Mn	Ni	S	P	Cr	Cu
<i>St3</i>	0.14–0.22	0.15–0.3	0.4–0.65	≤ 0.3	≤ 0.05	≤ 0.04	≤ 0.3	≤ 0.3
<i>40Cr</i>	0.36–0.44	0.17–0.37	0.5–0.8	≤ 0.3	≤ 0.035	≤ 0.035	0.8–1.1	≤ 0.3
<i>40Cr13</i>	0.35–0.44	≤ 0.6	≤ 0.6	≤ 0.6	≤ 0.025	≤ 0.03	12–14	–
<i>30CrMnSiNi2</i>	0.27–0.34	0.9–1.2	1–1.3	1.4–1.8	≤ 0.025	≤ 0.025	0.9–1.2	≤ 0.3

for 480 minutes in the melt at a temperature of 1,050 °C in an isothermal mode, while argon was above the surface of the bath.

After *DALMMS*, the surface of the specimens was cleaned of melt residues by etching in a mixture of acetic acid and hydrogen peroxide.

The chemical composition of the diffusion coatings was analyzed using X-ray microanalysis. An *INCA x-sight* X-ray spectrometer by Oxford Instruments Analytical was installed on a *JEOL JSM-7500F* scanning electron microscope for this purpose.

To identify the structure of specimens made of steel *St3*, *40Cr*, etching was carried out in a 4 % solution of nitric acid in ethyl alcohol. *Grechko* reagent was used for specimens made of *40Cr13*, *30CrMnSiNi2* steels.

Microhardness was measured with a *Dura Scan Falcon 500* electronic hardness tester. The microhardness of the coatings, as well as the transition layers and the base material, was measured at a load of 10 grams (GOST 9450).

Results and discussion

It was revealed that *DALMMS* with nickel and chromium results in the formation of diffusion coatings on the surface of all the materials under study. Fig. 1 shows micrographs of the specimens.

It was revealed that diffusion coatings were formed on the surface of all the specimens under study during *DALMMS*. The coatings consist of a surface layer and a transition layer. The transition zone is characterized by different elemental composition, structure and microhardness from both the coating and the base material (figs. 1–3). However, the structure and elemental composition of these coatings were different and were determined by the elemental composition of the coated steel. Thus, a carbide layer is formed on the surface of *40Cr* and *30CrMnSiNi2* steels, which is confirmed by the results of microhardness measurement and the results of X-ray microanalysis (figs. 2, 3). In this case, the carbide grains are oriented perpendicular to the surface of the specimen (fig.1 a, d). The formation of a carbide layer did not occur on *St3* and *40Cr13* steels. The formation of the carbide layer is due to the fact that the carbon contained in the steels diffuses to chromium, which is a strong carbide-forming element. At the same time, the carbon contained in the steel *St3* was not enough to form a carbide layer. In the case of *40Cr13* steel, the absence of a carbide layer is explained by the fact that in this steel carbon is bound into chromium carbides, which does not allow it to actively diffuse to the surface of the specimen, as in the case of forming a coating on *40Cr* steel. The results of the microhardness measurement are shown in fig. 2.

The specimens were subjected to X-ray microanalysis to identify the features of the formation and structure of coatings. The results are shown in fig. 3.

From the X-ray microanalysis results presented above, it was revealed that the elemental composition of the coated materials had a significant impact on the concentration distribution of elements in the surface layers of products subjected to *DLLHR*. From the point of view of elemental composition, coatings on steels *40Cr* and *30CrMnSiNi2* can be considered as consisting of two layers: a surface layer enriched with chromium and an intermediate layer enriched with nickel. Thus, the chromium content in the surface layer of the coating on steel *40Cr* was 80 %, on steel *30CrMnSiNi2* the chromium content was 78 %. It is also worth noting the nature of the distribution of chromium over the coating. On steels *40Cr* and *30CrMnSiNi2*,

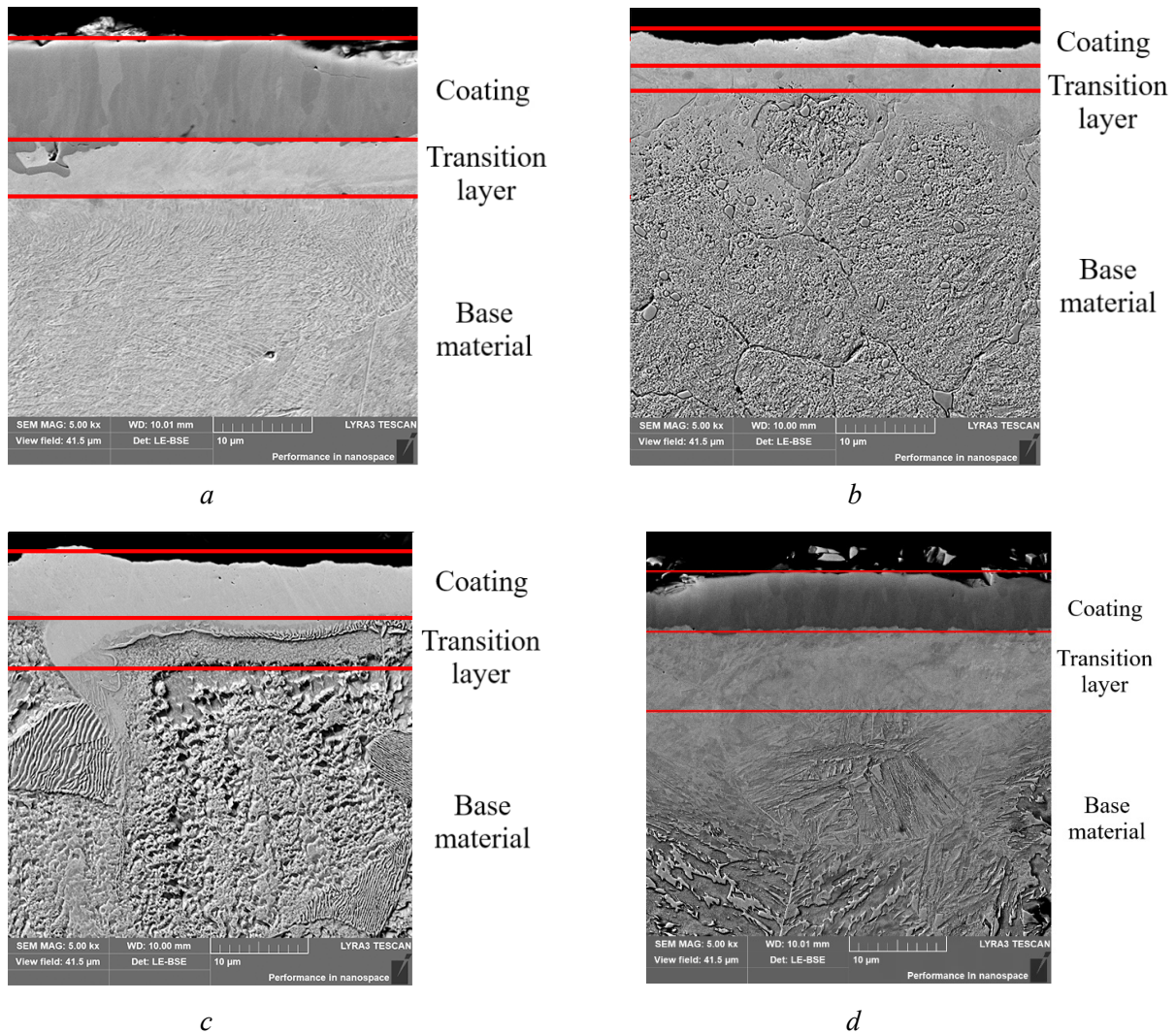


Fig. 1. DALMMS results on steel: 40Cr (a); 40Cr13 (b); St3 (c); 30CrMnSiNi2 (d)

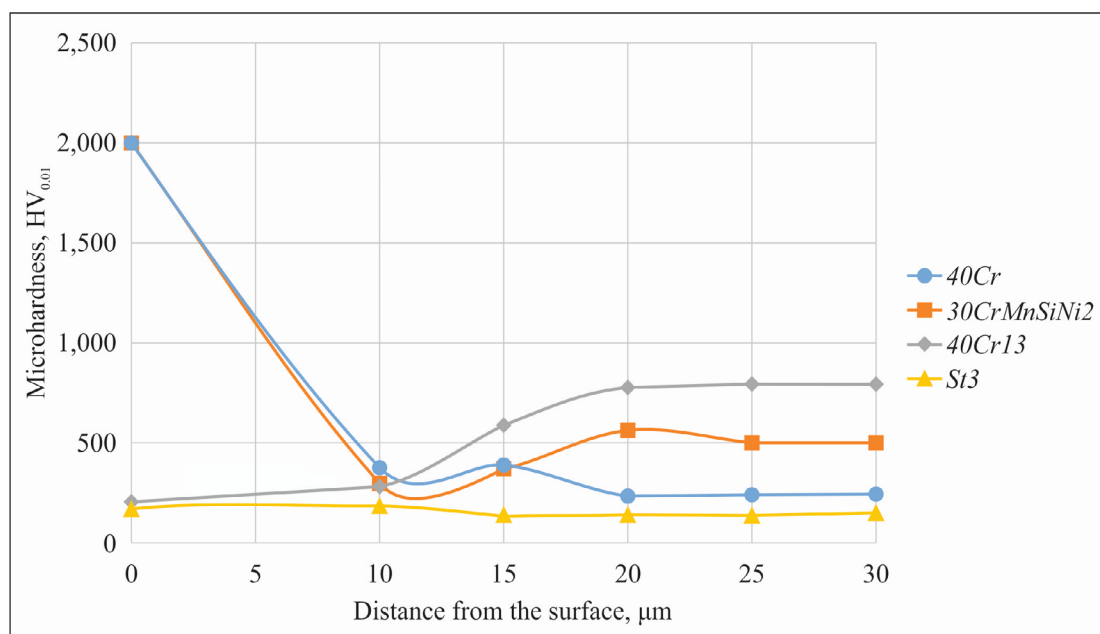


Fig. 2. Microhardness of specimens after DALMMS

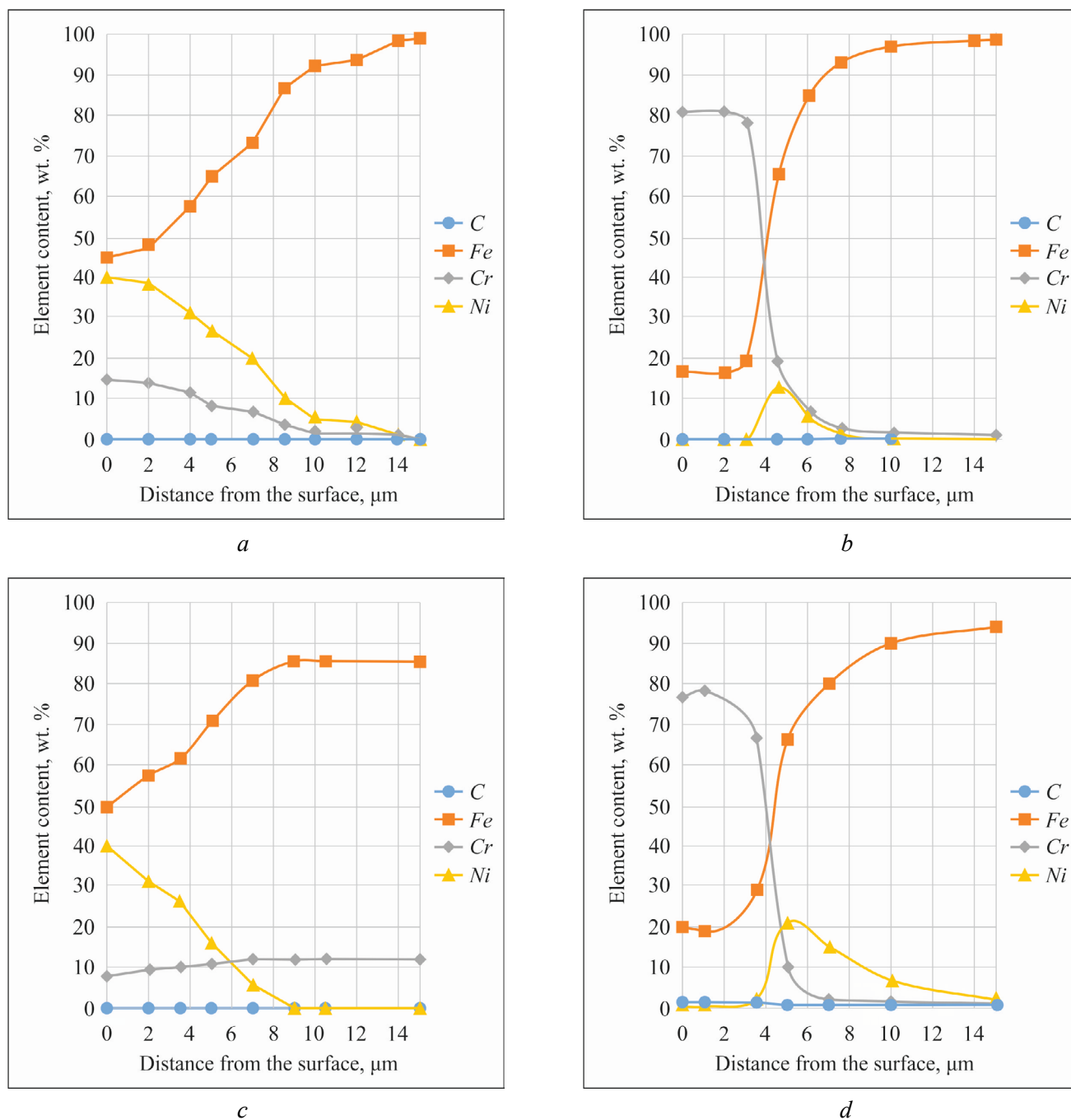


Fig. 3. X-ray microanalysis results after DALMMS: 40Cr (a); 40Cr13 (b); St3 (c); 30CrMnSiNi2 (d)

a sharp decrease in chromium concentration is observed during the transition from the surface layer to the layer enriched with nickel. Thus, at a depth of 5 μm for steel 40Cr the chromium concentration decreases from 80 % to 15 %, for steel 30CrMnSiNi2 it decreases from 78 % to 10 %. For steel 30CrMnSiNi2, the depth at which the chromium content corresponds to the base concentration is 10 μm. For steel 40Cr, the chromium diffusion depth was 15 μm. At the same time, in the surface layer of coatings on these steels the nickel content was quite low: 1.5 % for steel 30CrMnSiNi2 and 0.5 % for steel 40Cr. The maximum concentration of nickel was detected at a distance of 5 μm from the surface and amounted to 21 % for steel 30CrMnSiNi2 and 13 % for steel 40Cr.

In coatings formed on steels St3 and 40Cr13, the chromium content was significantly lower. Thus, on steel St3 the chromium content was 15 %, on steel 40Cr13 it was 9 %. At the same time, the change in chromium concentration occurred more smoothly than on steels 40Cr and 30CrMnSiNi2. Also, a higher nickel content was revealed in the coatings on steels St3 and 40Cr13. Thus, on steels St3 and 40Cr13, the

nickel content on the surface was 40 %. The depth of diffusion of nickel and chromium on steel *St3* was 14 μm , and on steel *40Cr13* it was 9 μm . At the same time, on steel *40Cr13* the chromium content in the coating was 9 %. The chromium concentration corresponding to the concentration of the uncoated material was detected at a depth of 7 μm . On *St3* steel, the maximum chromium concentration was 14.5 %. The chromium concentration corresponding to the concentration of the uncoated material was detected at a depth of 15 μm .

It can be concluded that one of the main factors affecting the concentration distribution of elements in the coating, its structure and microhardness is the elemental composition of the coated material, especially the carbon content. Steels *40Cr* and *30CrMnSiNi2* contain a fairly large amount of carbon, while the chromium content is about 1 %. Thus, most of the carbon in these steels is in the form of cementite. Chromium diffusing during the *DALMMS* process forms its own carbides on the surface of the coated material due to its greater affinity for carbon than iron. This factor causes more intense diffusion of chromium into materials, elemental-phase composition of which allows the formation of chromium carbides. In this case, a layer with an increased concentration of nickel is formed under the carbide layer. The formation of this layer is due to the low mutual solubility of nickel and carbides. Also, it is worth noting that on steels *St3* and *40Cr13* the formation of a carbide layer did not occur. On *St3* steel, the absence of a carbide layer is explained by the insufficient carbon content for its formation. On steel *40Cr13*, due to the high chromium content, the absence of a carbide layer is explained by the fact that this steel contains carbide $(\text{Cr}, \text{Fe})_{23}\text{C}_6$, which does not allow carbon to actively diffuse to the chromium obtained during *DALMMS*, and thereby form a carbide layer. Thus, comparing the chromium content in coatings on steel *40Cr* and *40Cr13*, a difference in concentration values was revealed by 8.8 times, which indicates a significant influence of the percentage of carbon and chromium in steel. In the case when carbon is bound into carbides of elements that have a lower affinity for carbon than chromium, diffusion of carbon occurs to the chromium obtained during *DALMMS* and the formation of chromium-based carbides.

The distribution of nickel in the coatings also had its own characteristics. On specimens with a carbide layer on the surface, nickel was pushed into the zone under this layer. Thus, on a specimen made of steel *30CrMnSiNi2*, the maximum concentration of nickel was observed at a depth of 5 μm and amounted to 21 %, while the nickel content in the carbide layer was 1.5 % (fig. 3). The low nickel content in the carbide layer is explained by the low mutual solubility of nickel and chromium carbides. A similar layer enriched with nickel was observed on *40Cr* steel. The maximum nickel concentration was 13 % at a depth of 4.5 μm . Further, the nickel concentration gradually decreased to a concentration characteristic of the uncoated material. On steels *St3* and *40Cr13*, the nickel concentration was significantly higher and amounted to 40 % on the surface of the specimen. Then the concentration gradually decreased to concentration values characteristic of the uncoated material.

The obtained data on the structure of the coatings are in good agreement with the already known results of diffusion saturation with nickel and chromium using the *DALMMS* technology for materials such as *Armco*, *Steel 10*, *Cr6WV* [21].

Thus, when forming coatings on steels *40Cr*, *30CrMnSiNi2*, in the context of elemental composition and structure, the coatings consist of several functional layers: surface carbide layer and transition one. At the same time, the layers have a clear interface. For steels *40Cr13* and *St3*, a single-layer coating is formed. Fig. 4 shows *EDS* images of steels *40Cr* and *St3*, characterizing the elemental composition of diffusion coatings.

The presented images show the distribution of elements in the coating and between the diffusion layers. Thus, for a specimen made of *40Cr* steel with a *Ni-Cr* coating, the surface layer consists of carbide grains elongated in the direction of diffusion; the transition layer is a solid solution of $\text{Fe}(\text{Ni}, \text{Cr})$ (fig. 4 a). The coating on a specimen made of *St3* steel is formed on the basis of solid solutions of the $\text{Fe}(\text{Ni}, \text{Cr})$ system (fig. 4 b).

Thus, it was revealed that the formation of *Ni-Cr* coatings obtained using the *DALMMS* technology, provided that the technological conditions are constant, largely depends not only on the elemental composition of the coated materials, but also on its phase composition.

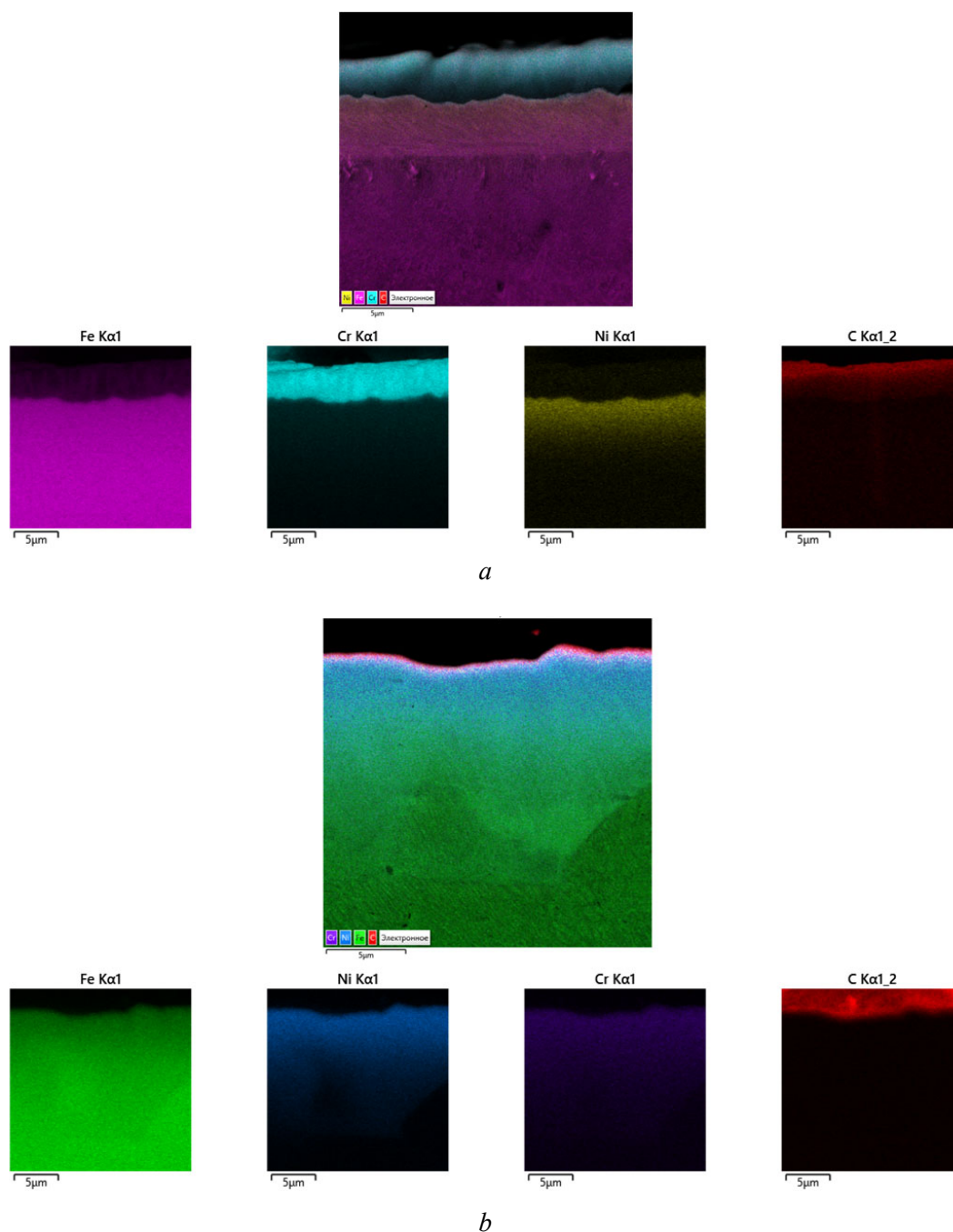


Fig. 4. EDS of diffusion coatings on steels: 40Cr (*a*); St3 (*b*)

Conclusions

1. It is found that the total thickness of nickel and chromium diffusion coatings applied to structural steels reaches 15 μm .

2. Conducting DALMMS on carbon and low-alloy steels leads to the formation of two-layer coatings: a surface carbide layer and a transition solid-soluble one. At the same time, the chromium content in the surface layers reaches 80 % with a nickel content of 1.5 %. The maximum concentration of nickel is observed in the transition layer and amounted to 21 % at a depth of 5 μm on steel 30CrMnSiNi2 and 13 % at a depth of 4.5 μm on steel 40Cr.

3. Conducting DALMMS on steels with carbide-forming elements or low carbon content leads to the formation of single-layer coatings based on solid solutions. At the same time, the nickel content in the coating reaches 40 %, the chromium content for steel St3 is 14.5 %, and for steel 40Cr13 it is equal to 9 %.

4. The thickness of the coatings, as well as its elemental composition, structure and microhardness, depended on the element-phase composition of the base material. The maximum thickness of the coating is achieved under the condition of the formation of single-layer coatings. The maximum microhardness of the coatings is achieved under the condition of the formation of a surface carbide layer, and is 20,000 MPa.

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Conflicts of Interest

The authors declare no conflict of interest.

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