
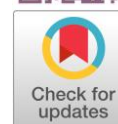


Composite electrochemical coatings of Ni-CrB₂ from sulfuric acid and sulfamate electrolytes: formation, structure and properties

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Abstract

Electrodeposited coatings are in demand in many industries: due to the high adhesive strength to the base metal, they can be applied to complex-shaped products, and the thickness of the deposited coating can be controlled. However, standard electrolytic coatings do not always meet the necessary operating requirements. In this case, the formation of composite electrolytic coatings, which consist in the modification of electrolytes by various dispersed particles, becomes an urgent task. These particles are introduced into the electrolyte and deposited together with the precipitate metals, which leads to changes in the coating properties. In this article, the structure, adhesive and corrosion properties of nickel-chromium diboride composite coatings obtained in the electrodeposition process are studied. Precipitation was carried out in sulfuric acid and sulfamate electrolytes with a concentration of dispersed chromium diboride particles of 10-40 g/l. The coatings consist of nickel and CrB₂ phases. The adhesive strength of the coating with a steel base is satisfactory; chipping and peeling were not observed. The corrosion resistance of sulfamate electrolyte coatings is higher than that of the coatings obtained from sulfuric acid electrolyte due to the compactness of nickel precipitates. Chromium diboride in the coating provides 1.5-2 times greater corrosion resistance.

Keywords

electrodeposition
nickel plating
composite electrolytic coatings
chromium diboride
corrosion resistance

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Key findings

- Composite coatings of nickel-chromium diboride obtained by the electrodeposition method from a sulfamate electrolyte have a uniform structure, without pores, cracks and discontinuities.
- The corrosion resistance of composite electrodeposited coatings is ensured by the presence of CrB₂.
- The developed coatings can be recommended to prevent corrosion of products made of carbon structural steels.

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1. Introduction

Currently, science and technology are developing very rapidly, which has led to the creation of new machines and mechanisms that are used in many fields. This, in turn, requires the use of new solutions, materials and technologies to improve machine parts and increase their reliability and strength. As industry develops, the importance of extending the service life of materials is becoming more significant. During operation, destruction of a material mainly occurs due to the interaction of its surface with an aggressive

environment [1]. Thus, to increase the service life of a product and increase its performance characteristics, it is necessary to form a corrosion-resistant coating.

Currently, there are many methods for obtaining coatings, such as the hydrothermal method [2, 3], non-vacuum electron beam surfacing [4-6], plasma spraying [7, 8], laser surfacing [9, 10], electrodeposition [11-13] and others. It is worth noting that the electrodeposition technology is relatively simple, and that the deposition of coatings can be carried out at low cost and low temperature at atmospheric pressure via easy-to-handle equipment with a small amount of production waste, which makes this coating method

widely used in industrial applications [14]. Coatings such as pure metals, alloys, and composites can be applied to many metal substrates, which makes it possible to improve surface properties, namely, corrosion and wear resistance [15].

Composite electrolytic coatings (CEPs) are of particular interest for the machine-building industry since they can process products of complex geometry, control the required thickness of coatings, and combine the properties of a metal matrix and a dispersed phase [16].

Nickel has a wide range of applications as a metal matrix because of its good toughness, chemical stability, corrosion resistance and easy codeposition with dispersed particles of any nature. Nickel coatings have a high tendency to passivate, which leads to a significant improvement in corrosion resistance in media such as aqueous, saline, organic, alkaline and mineral acids [17, 18].

The inclusion of dispersed particles in the metal matrix of the coating improves properties such as hardness, wear resistance, and corrosion resistance.

Various carbides, borides, nitrides, nanowires, nanotubes, diamonds, etc., can be used as the dispersed phase (DP) of a composite electrolytic coating [19]. In previous studies [20–23], the introduction of SiC into the nickel matrix increased the hardness and wear resistance of the composite coating. In addition, an increased amount of silicon carbide contributes to an increase in oxidation resistance due to the formation of SiO₂ [24]. Additionally, the introduction of TiO₂ into the metal matrix has a positive effect on the mechanical properties [25]. It was reported in [26] that alloying the zinc matrix with nickel contributes to an increase in the corrosion resistance of coatings. Notably, chromium diborides are of significant interest because they have high hardness and corrosion resistance. Additionally, the properties of Ni–CrB₂ composite electrolytic coatings have not been studied yet.

In this work, the structure and properties of nickel-based composite coatings with dispersed chromium diboride particles (adhesive strength and corrosion resistance) are investigated.

2. Materials and methods

The deposition of Ni–CrB₂ composite coatings was carried out from sulfuric acid and sulfamate electrolytes of the following compositions:

Sulfuric acid electrolyte: 245 g/l NiSO₄·7H₂O; 30 g/l H₃BO₃; 20 g/l NaCl; 1 g/l organic supplement. The current density was 0.07 A/cm², the pH was 5.0–5.5, the electrolyte temperature was 50 °C, and the deposition time was 60 min.

It was found that an increase in the coating mass occurs in the current density range of 0.03–0.07 A/cm². With a further increase in the current density, deterioration in the coating layer appearance was noted. Thus, the optimal

value of the current density in the sulfuric acid electrolyte was assumed to be 0.07 A/cm².

According to the results of the study, the greatest weight gain was observed at 50 °C. The best coating quality was obtained at 50 °C and 70 °C. Analyzing the data obtained, the operating temperature of the sulfuric acid electrolyte was chosen to be 50 °C.

Sulfamate electrolyte: 400 g/l Ni(NH₂SO₃)₂; 25 g/l H₃BO₃; 15 g/l NiCl₂·6H₂O; 1 g/l organic supplement. The current density was 0.05 A/cm², the pH was 3.6–4.5, the electrolyte temperature was 35 °C, and the deposition time was 60 min.

In the sulfamate electrolyte, the maximum weight gain was observed at 0.05 A/cm², and a further increase in the current density led to a sharp decrease in the mass of the deposited nickel. This pattern is explained by the combined release of hydrogen with the reduction of nickel, which significantly suppresses metal deposition and negatively affects the quality of the coating.

The maximum weight gain in the sulfamate electrolyte was obtained at 35 °C. A further increase in the process temperature led to a gradual decrease in the weight gain of the nickel precipitate. The reason for this, on the basis of the literature data [27], is the growth of coupled kinetic transport phenomena leading to morphological and microstructural changes in the electrodes that occur with increasing temperature.

With an increase in the duration of the process, a directly proportional dependence of the increase in the mass of the deposited metal was observed. The optimal electrolysis duration for the deposition of a nickel coating with a thickness of 20 to 30 μm was 60 min. A duration of electrolysis of less than 60 min does not result in either the full surface coverage or the desired deposit thickness. An increase in the duration of electrolysis above the optimal duration leads to a deterioration of the adhesion of the precipitate to the substrate due to the excessive thickness of the coating.

The concentrations of CrB₂ powder used were 10, 20, 30, and 40 g/l. The particle size ranged from 2–20 μm.

The formation of composite coatings was carried out in an electrochemical cell; a graphic electrode was used as the anode, and a steel plate (steel 20) was used as the cathode. The dispersed particles were held and mixed in the electrolyte in a suspended state via a magnetic stirrer.

The methods used to prepare the surfaces of the samples included surface machining followed by rinsing in running water, chemical degreasing in caustic soda solution (50 g/l) followed by rinsing, and chemical etching in 10% hydrochloric acid solution to remove the oxide film from the surface of the product, increase the adhesive strength of the coating with the base, and rinsing.

The structure of the composite electrolytic coatings was studied by scanning electron microscopy on a Carl Zeiss EVO50 XVP at magnifications ranging from 5,000 to 10,000

times. The chemical composition of the local sites was determined via an EDS X-Act (Oxford Instruments) electron microprobe analyzer. The thickness of the coatings was determined on the cross-section of the sample via microscopy.

The diffraction patterns of the samples were obtained in the θ - θ system via a high-resolution ARX' TRA diffractometer. The diffraction pattern was captured via a Cu $K\alpha$ probe, the wavelength of which was $K\alpha_1 = 1.540562 \text{ \AA}$ and $K\alpha_2 = 1.542390 \text{ \AA}$. The final diffraction pattern was recorded via a detector in the angular range from 20° to 100° , with a shooting step of $2\theta = 0.05^\circ$. The shooting speed was 0.075° per minute. The phases present in the studied materials were identified via the ICDD PDF-4 database.

The determination of adhesive properties was carried out by applying a scratch grid (drawings) according to GOST 9.302-88 [20]. The essence of the method consists of making at least 3 parallel scratches with a steel tip with a distance of 2–3 mm and similar risks perpendicular to it. The scratches should be made at an angle of 30° and deep to the base metal of the substrate. The absence of peeling in the grid of square areas indicates satisfactory strength.

Corrosion resistance tests of steel, "pure" nickel and Ni-CrB₂ coatings obtained from sulfuric acid and sulfamate electrolytes were carried out using a potentiostat-galvanostat Elins P-40X. The samples were plates with a size of $20 \times 10 \times 2 \text{ mm}$. A three-electrode cell was used for the electrochemical studies. The working electrode was a coated sample, the reference electrode was a silver chloride electrode, and the counterelectrode was graphite. Potentiodynamic studies were performed at room temperature (25°C) with natural aeration of the solution. The corrosion resistance was assessed in a solution of 3.5% sodium chloride. Immediately before the test, the samples were kept in an electrolyte to establish a steady state and then polarized in the anode direction at a potential sweep rate of 1 mV/s . The corrosion potential (E_{corr}) and corrosion current density (i_{corr}) were determined by extrapolating the cathode sites onto polarization curves, which demonstrated Tafelian behavior.

3. Results and Discussion

The structure of the electrolytic nickel coating without the addition of a dispersed phase is shown in Figure 1. The thickness of the layer of the sulfuric acid electrolyte is $30 \mu\text{m}$, and that of the sulfamate is $\sim 20 \mu\text{m}$. The coatings are homogeneous, and no pores or cracks are found, which indicates that the deposition parameters were selected correctly.

According to the results of preliminary studies, the addition of chromium diboride at concentrations ranging from 0.3 to 10 g/l does not allow the formation of a composite coating because of the low content of dispersed particles in the electrolyte; thus, the electrode layer does not have enough time for enrichment.

Increasing the concentration of dispersed particles in the electrolyte from 10 to 40 g/l made it possible to obtain a nickel coating with inclusions in the form of particles (Figure 2).

The coating obtained at a concentration of 10 g/l was characterized by the presence of large agglomerates with unprecise geometries and sizes ranging from 5 to $20 \mu\text{m}$ (Figure 2a).

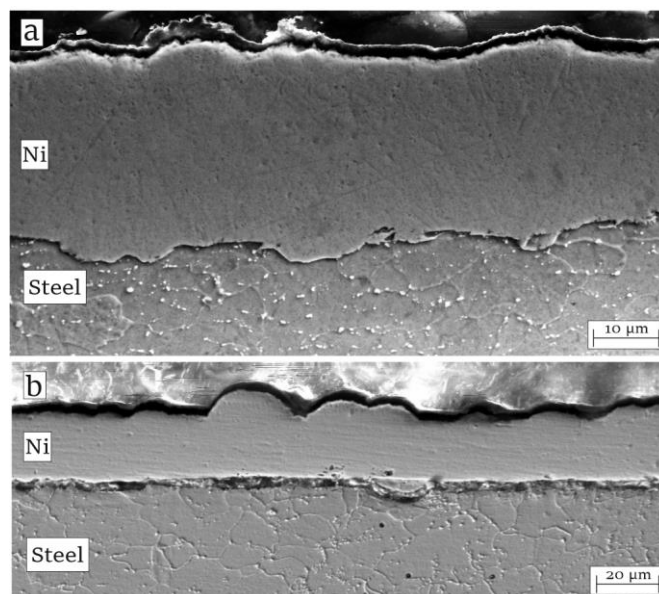


Figure 1 Images of nickel coatings obtained from sulfuric acid electrolyte (a) and sulfamate electrolyte (b).

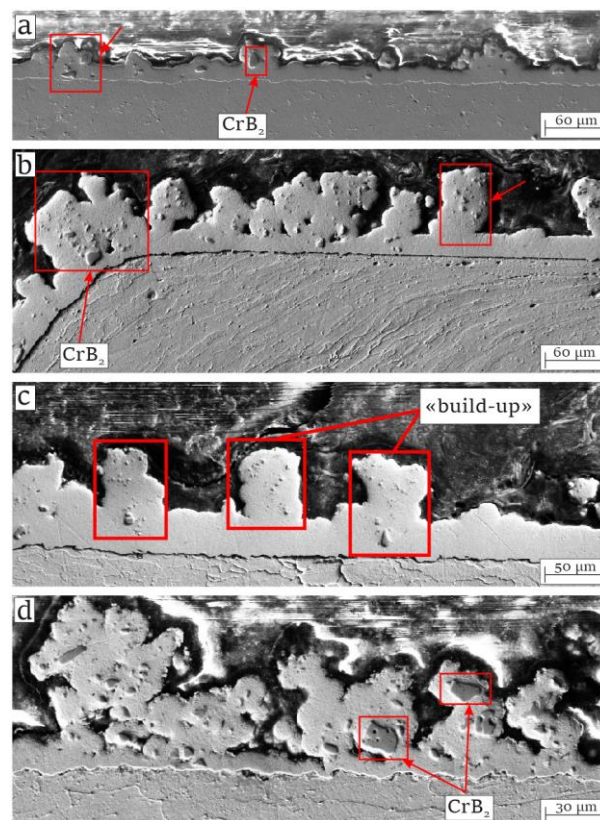


Figure 2 Images of nickel coatings from sulfuric acid electrolytes with different concentrations of chromium diboride: 10 g/l (a); 20 g/l (b); 30 g/l (c); 40 g/l (d).

With an increase in the concentration of DP to 20 g/l, a “build-up” layer with a thickness of 50–60 μm formed in the coating structure (Figure 2b). Chromium borides are the smaller particles.

Figure 2c shows the coating structure with a CrB_2 concentration of 30 g/l in a sulfuric acid electrolyte. The regularity of the increase in the content of the dispersed phase and the size of the “build-up” phase was observed. The thickness of these sections ranged from 60–70 μm , and their pitch also decreased. Notably, precipitation occurred on the sides of the steel plate, which indicates a high scattering capacity of the electrolyte.

At a boride concentration of 40 g/l, “build-up” with a thickness of approximately 70 μm containing inclusions of borides also formed (Figure 2d). The particles were distributed evenly throughout the volume of the “build-up”. No pores, cracks, or discolorations were found.

Composite coatings obtained from a sulfamate electrolyte with a concentration of chromium diboride ranging from 10 to 40 g/l are shown in Figure 3. The marked areas in the images correspond to chromium diboride particles. The coatings were uniform over the entire surface of the substrate with a thickness of 20 μm , and pores and cracks were not detected. An increase in the concentration of chromium diboride in the electrolyte did not lead to an increase in the number of particles in the nickel matrix. This is because this electrolyte is complex; that is, denser precipitation formed from it, and the introduction of diborides into it was more difficult. A similar compactness of nickel deposits from a sulfamate electrolyte was also described in other works [28]. This property may be associated with the low internal stresses of the nickel coatings obtained from the sulfamate electrolyte [29].

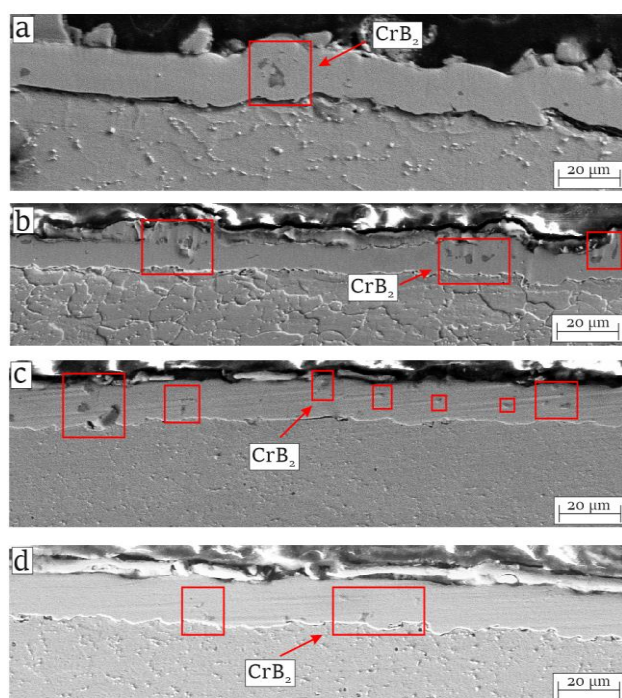


Figure 3 Images of nickel coatings from sulfamate electrolytes with different concentrations of chromium diboride: 10 g/l (a); 20 g/l (b); 30 g/l (c); 40 g/l (d).

Microinformatic analysis revealed that the detected particles in the nickel coating corresponded to chromium diborides (Figure 4).

X-ray diffraction patterns of composite electrolytic coatings with chromium diboride contents in electrolytes ranging from 10 to 40 g/l are shown in Figure 5.

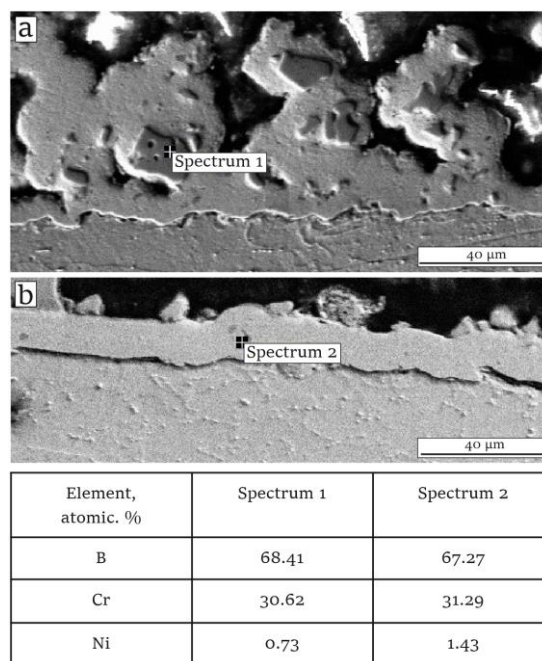


Figure 4 Results of electron microscopic analysis of composite coatings: sulfuric acid electrolyte (a); sulfamate electrolyte (b).

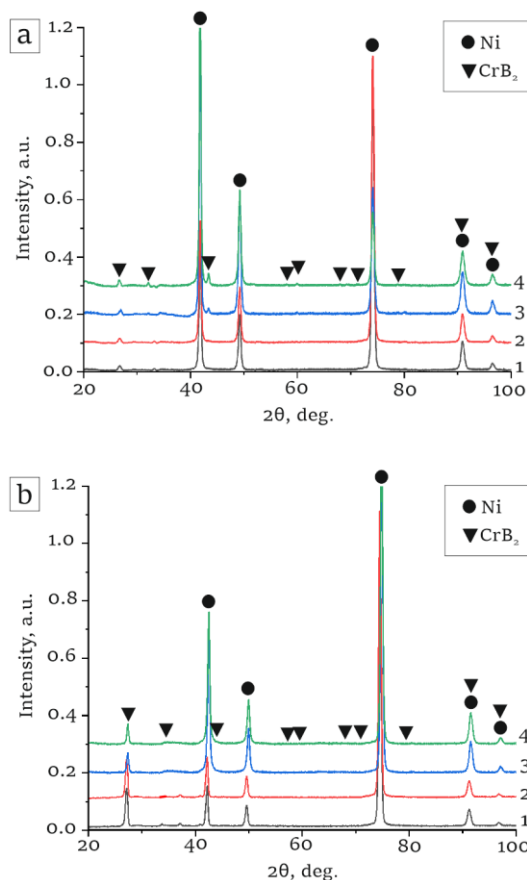


Figure 5 X-ray diffraction patterns of layers formed from sulfuric acid and sulfamate electrolytes containing chromium diboride: 1 – 10 g/l; 2 – 20 g/l; 3 – 30 g/l; 4 – 40 g/l.

X-ray phase analysis confirmed the formation of a nickel-based composite coating with chromium diboride particles.

As a result of the assessment of the adhesive strength of the coating with steel strength, the coatings were found to have satisfactory adhesion. The peeling of coatings and chips, including crosshairs, was not observed. Thus, increasing the concentration of chromium diboride in the electrolyte to 40 g/l did not adversely affect the bond strength (Figure 6).

The results of measuring the corrosion resistance of the materials in a solution of 3.5 % sodium chloride are shown in Table 1 and Figure 7. Figure 7 shows that the steel sample actively dissolved throughout the entire studied potential range. The application of a nickel coating on a steel substrate improved the corrosion properties of the product. The addition of chromium diboride at concentrations of 10 and 40 g/L led to a decrease in the corrosion current and a shift in the corrosion potential to the region of positive potentials, which increased the resistance of the samples.

Composite electrolytic coatings obtained from a sulfamate electrolyte had a wide passivation area, and unlike coatings obtained from a sulfuric acid electrolyte, there was no area of active dissolution up to the passivation potential.

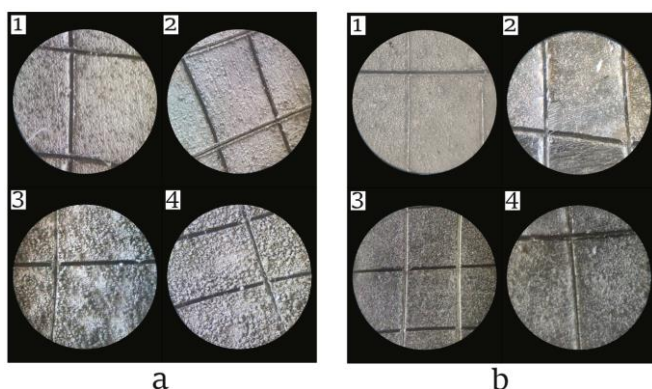


Figure 6 Images of the scratch grid on the surface of samples from sulfuric acid (a) and sulfamate (b) electrolytes containing chromium diboride: 1 – 10 g/l; 2 – 20 g/l; 3 – 30 g/l; 4 – 40 g/l.

Table 1 Results of corrosion tests.

Specimen series	I_{corr} , $\mu\text{A}/\text{cm}^2$	E_{corr} , mV
Sulfuric acid electrolyte		
steel	0.036	-0.348
nickel coating	0.076	-0.247
Ni + 10 g/l CrB_2	0.225	-0.192
Ni + 40 g/l CrB_2	0.0342	-0.247
Sulfamate electrolyte		
steel	0.036	-0.348
nickel coating	0.0031	-0.31
Ni + 10 g/l CrB_2	0.0043	-0.55
Ni + 40 g/l CrB_2	0.0073	0.1

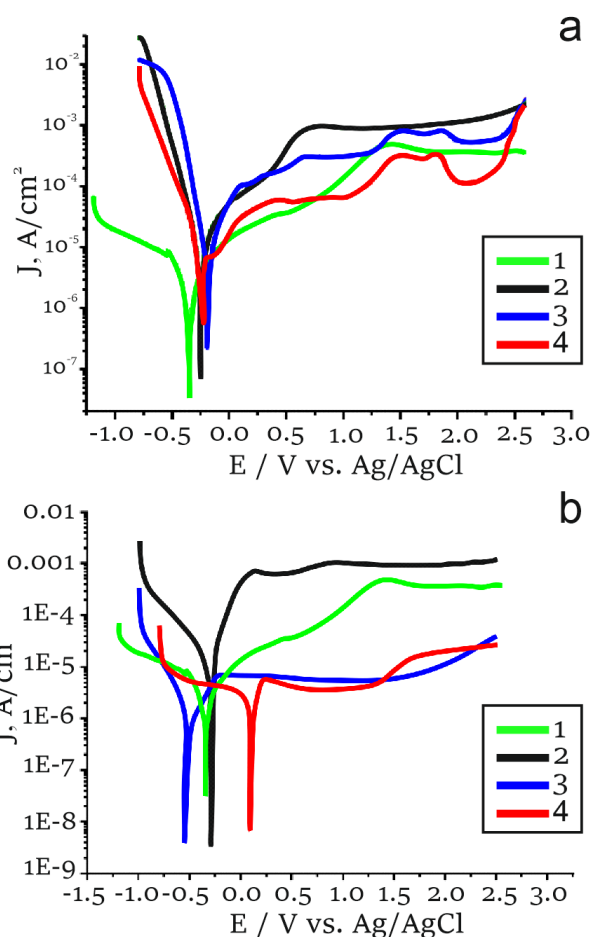


Figure 7 Corrosion diagram of samples from sulfuric acid (a) and sulfamate (b) electrolytes: 1 – steel base; 2 – nickel coating; 3 – CAP with 10 g/l CrB_2 ; 4 – CAP with 40 g/l CrB_2 .

4. Limitation

In this study, only one organic additive was used in the formation of composite electrolytic coatings. In the future, it is necessary to study in detail the effect of other various organic additives on the uniformity of the dispersed phase distribution in the metal matrix and determine the optimal number of the additives.

It is also planned to supplement the obtained results with the electrochemical impedance spectroscopy (EIS), which can provide a deeper understanding of the mechanisms of corrosion and protective properties of coatings.

5. Conclusions

Composite coatings are formed on the surface of structural steel by electrodeposition. The coatings consisted of a nickel matrix and dispersed CrB_2 particles. An increase in the concentration of the dispersed phase in the sulfuric acid electrolyte leads to an increase in roughness; this manifests itself in the form of “build-up”, and the coating thickness ranges from 20 to 70 μm . For the sulfamate electrolyte, the coatings are uniform in thickness and amount to 20 μm over the entire substrate area but have a smaller number of

chromium diboride particles in the nickel precipitate. Electron microprobe and X-ray phase analyses revealed that the coatings consisted of nickel and chromium diboride phases.

The adhesive strength of the coatings is considered satisfactory; thus, the introduction of chromium diboride into the nickel layer does not adversely affect the adhesive strength.

An increase in corrosion resistance is typical for all the samples. The corrosion current of the composite coatings obtained from the sulfuric acid electrolyte decreases in comparison with that of the steel and nickel coatings due to the passivation areas; that is, CrB₂ has a positive effect on the corrosion resistance of the coatings. It can be assumed that the pitting corrosion of the CEP data is minimal since the branches of the corrosion diagram are flat. The introduction of chromium diboride greatly lowers branch currents and provides passivation areas.

For the coatings obtained from the sulfamate electrolyte, the corrosion current decreases with increasing concentration of chromium diboride. A wide passivation area, since the branches run parallel to the abscissa axis (corrosion potential), increases the corrosion resistance of coatings by 1.5–2 times.

Thus, the use of a sulfamate electrolyte is recommended to obtain composite electrolytic coatings with a nickel matrix and chromium diboride particles with increased corrosion resistance. These coatings can be used for fasteners as well as for parts operating in severe conditions of high temperature and aggressive gas environments in automotive, ship, tool engineering, and other industries for parts requiring a multifold increase in durability.

• Supplementary materials

No supplementary materials are available.

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• Conflict of interest

The authors declare no conflict of interest.

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