published by Ural Federal University
eISSN 2411-1414
chimicatechnoacta.ru

ARTICLE
2024, vol. 11(4), No. 202411408
DOI: 10.15826/chimtech.2024.11.4.08

Electrodeposition of Ni-WC composite coatings: formation, structure and properties

Polina M. Petukhova *, Evdokiya G. Bushueva,
Oksana N. Novgorodtseva, Vladislav D. Kizimov

Faculty of Mechanical Engineering and Technologies, Novosibirsk State Technical University, Novosibirsk 630073, Russia
* Corresponding author: polina.ptkhv@gmail.com

This paper belongs to a Regular Issue.

Abstract

Currently, electroplating is actively developing, and one of its promising areas is the study of composite electrochemical coatings (CEC). They are of great interest to the industrial sector, especially for mechanical engineering, due to their unique properties. CEC contain microscopic particles as additives that can significantly improve coating properties such as hardness and wear resistance. This is especially important for the manufacturing of high-quality parts of industrial mechanisms. These areas can lead to new advances in the development of better and more durable materials. In this work, composite coatings of nickel-tungsten carbide were obtained by the method of electrodeposition. The deposition was carried out in a sulfuric acid electrolyte with different concentrations of tungsten carbide. Formation of composite coatings was carried out at a concentration of dispersed particles in a sulfate electrolyte of 10 g/L. The coatings have no pores or cracks. Nickel and tungsten carbide phases were detected in the coatings by X-ray phase and micro-X-ray spectral analyses. Inclusions of WC in the nickel matrix lead to an increase in the microhardness of the coating by 10–15 times. Wear resistance was assessed under dry sliding friction with reciprocating motion; a decrease in the volume of worn material by 2–3 times was noted when tungsten carbide was added into the nickel matrix.

Keywords

electrodeposition
nickel plating
composite electrolytic coatings
tungsten carbide
microhardness
wear resistance

Received: 06.09.24
Revised: 03.10.24
Accepted: 10.10.24
Available online: 19.10.24

Key findings

- The formation of a Ni-WC composite coating by the method of electrodeposition was carried out at a concentration of particles in a sulfate electrolyte of 10 g/L.
- An increase in the microhardness and wear resistance of composite electrolytic coatings is ensured by the presence of tungsten carbide particles in a nickel matrix.
- The developed coatings can be recommended to enhance wear resistance of products made of carbon structural steels.

1. Introduction

With the development of industry, higher demands are placed on the strength, hardness and wear resistance of products. Insufficient efficiency of surface protection of metal products reduces its service life. Thus, to ensure the durability of parts, coatings with the necessary mechanical properties are formed on their surface [1–3]. The methods of coating application include: vapor deposition [4–6], laser cladding [7–9], non-vacuum electron beam cladding [10–12], electrical discharge coating [13–15], electrodeposition [16–20], and others.

It is worth noting that electrodeposition, in comparison with the above methods, has the following characteristics: the ability to process products of complex configuration, ease of equipment operating, control of the required coating thickness, the possibility of applying several layers of coatings.

However, electrodeposited pure metal has a limited range of wear resistance. For this reason, composite electrolytic coatings (CEC), which are metal matrices with embedded dispersed particles, are of particular interest. The combination of the properties of the deposited metal and dispersed particles makes it possible to obtain composite coatings with unique properties [21].

Also, an equally important task is the complete replacement of chromium-containing coatings due to the toxicity of chromium (VI). Similar or superior, compared to the chromium-containing ones, coatings can be obtained by forming composite electrodeposited coatings [22].

The process of forming composite coatings is multistage, requiring interaction at three different boundaries. The first boundary at which the interaction occurs is the boundary between the dispersed phase and the electrolyte. Here, the process of ionization of the dispersed phase and its transfer to the surface of the material takes place. At the second boundary, between the electrode and the electrolyte, a number of electrochemical reactions occur, including redox processes. The electrode serves as a cathode or an anode, depending on the purpose of coating formation. Here, the dispersed phase is deposited on the electrode surface. The third boundary in which the interaction occurs is the boundary layer between the dispersed phase and the electrode. At this stage, the dispersed phase adheres firmly to the electrode surface, forming a stable coating. The transportation of dispersed particles to the cathode surface can be carried out by mixing, brownian motion, electrophoresis, gravitational forces, or adsorption of metal cations or other ions onto its surface (for example. H^+, NH^+, etc.). During the formation of nuclei in the areas of contact with the electrode surface, the particles actively interact, contributing to its coating with metal [23. 24].

Electrodeposited composite coatings are metal or alloy matrices containing dispersed particles. These particles can be oxides, nitrides or carbides used to increase the wear resistance of the coating. The inclusion of such particles in the coating matrix improves properties such as hardness, wear resistance, friction resistance and corrosion resistance [25].

Nickel and its alloys are widely used as a metal matrix due to their high strength, good toughness, chemical stability and corrosion resistance [26]. Tungsten carbide has a high hardness (92–94 HRA), which makes the compound promising for the manufacture of tools and wear resistant parts.

The purpose of this work is to form composite coatings of nickel-tungsten carbide, increase the wear resistance of the nickel coating by adding dispersed high-strength particles of tungsten carbide, and evaluate the adhesive strength.

2. Materials and methods

The composite coatings were formed from a standard sulfuric acid electrolyte of the following composition: 245 g/L $NiSO_4 \cdot 7H_2O$, 30 g/L H_3BO_3, 20 g/L NaCl, 1 g/L OS-20. The concentration of tungsten carbide powder was: 1, 3, 5, 7, 10, 20, 30, 40 g/L. The particle size was 2–20 μm. The pH value was 5.0–5.5; the cathode density was 0.7 A/cm²; the temperature was 50 °C; the deposition time was 60 minutes. The constant stirring of the electrolyte was carried out by a magnetic stirrer. The CEC was deposited on a steel plate (steel 20); the anode was a graphite electrode.

Preparation before deposition consisted of a standard technique, including the plate surface machining, degreasing in a solution of soda ash and decapping in a solution of hydrochloric acid with rinsing in running water after each operation.

The structure of composite electrolytic coatings was studied by scanning electron microscopy on Carl Zeiss EVO50 XVP with 5.000 to 10.000 times magnification. The chemical composition of the local sites was determined using the EDS X-Act X-ray microanalysis (Oxford Instruments). The diffraction pattern of the specimens was obtained in the θ-θ system using a high-resolution ARX'TRA diffractometer. The diffraction patterns were recorded using Cu Ka radiation with the wavelengths of $K\alpha_1 = 1.540562$ Å and $K\alpha_2 = 1.542390$ Å. The final diffraction pattern was recorded using a detector in the angular range from 20° to 100° with a recording step of $2\theta = 0.05°$. The recording speed was 0.075° per minute. The phases present in the studied materials were identified using the ICDD PDF-4 database.

The determination of adhesive properties was carried out by applying a scratch grid (marks) according to State Standard 9.302-88 [27]. The essence of the method consists in making at least 3 parallel scratches with a steel tip with a distance of 2–3 mm and similar marks perpendicular to it. Scratches should be made at an angle of 30° and a depth to the base metal of the substrate. The absence of peeling in the grid of square areas indicates satisfactory strength.

The microhardness of composite coatings and steel substrate was measured in cross-section using the Vickers method with an indentation load of 10 g on a DuraScan-50 hardness tester. Ten measurements were made for each sample.

The wear resistance of composite coatings was evaluated under conditions of dry friction with reciprocating motion on a universal friction machine UMT-2 (Bruker Nano GmbH, Germany) according to ASTM G133A at room temperature (298 K) using a “ball-plane” scheme with a non-rotating ball. The counterbody and comparison material were carbide balls (VK6) with a diameter of 6.35 mm and plates of steel 20, respectively. The following test parameters were used: travel length of 5 mm, sliding speed of 5 mm/s, load applied to the counterbody of 25 N, total sliding distance of 100 m. The friction surface was studied using the Contour GT-K1 optical 3D system (Bruker Nano GmbH, Germany) to determine the amount of worn material.

3. Results and Discussion

The nickel coating from the sulfuric acid electrolyte is characterized by homogeneity and has no defects (Figure 1a). The coating thickness is 15 μm.

The initially selected concentrations of tungsten carbide in the sulfate electrolyte (1 g/L and 3 g/L) do not allow the formation of a composite coating. According to the results of scanning electron microscopy, there are no dispersed particles in the coating (Figure 1a, 1b). The thickness of the coatings is 10 µm.

A further increase in the concentration to 5 g/L does not lead to the formation of CEC (Figure 1c). A possible reason for this is its small distribution over the coating and WC particles are absent in this cross-section.

At a concentration of 7 g/L, it is possible to obtain a composite coating (Figure 1d). However, the particles are located unevenly throughout the coating, and their accumulation is observed in several areas.

An increase in the concentration of tungsten carbide particles from 10 to 40 g/L leads to the formation of Ni-WC composite coatings. which is confirmed by SEM (Figure 2). Tungsten carbide particles are sometimes large agglomerates with fuzzy geometries. The coating thickness is on average 20 µm but there are "build-ups" of greater thickness (30 µm), inside which WC particles are located. A non-uniform dispersion of conductive particles in bath could further increase the porosity of the coating, by introducing porosities from within large particle agglomerates as well as accelerating the growth of protrusions [28].

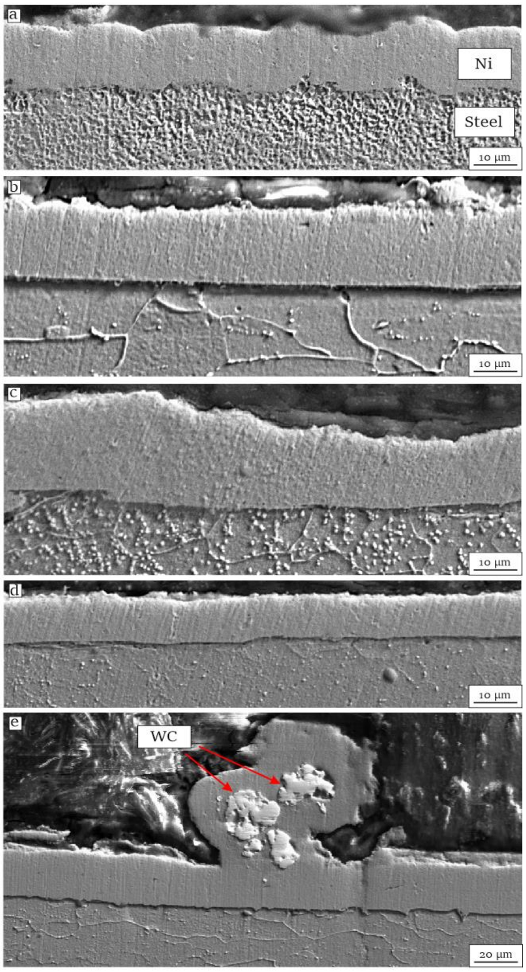


Figure 1 Images of a nickel coating with a concentration of tungsten carbide in an electrolyte: 0 g/L (a); 1 g/L (b); (c) 3 g/L; 5 g/L (d); 7 g/L (e).

X-ray microanalysis revealed that the particles embedded in the nickel matrix correspond to tungsten carbide (Figure 3). One image of the coating is provided to illustrate the phase composition of the detected particles.

X-ray images of specimens with a WC content from 1 to 7 g/L in the sulfuric acid electrolyte are shown in Figure 4. Two phases were found in the specimens: nickel and α-iron. Thus, XRF confirms previous studies, namely, that WC concentrations from 1 to 7 g/L do not allow the formation of composite electrolytic coatings. Single particles of tungsten carbides in the coating do not affect the X-ray pattern with 7 g/L, which is due to the low particle content in the nickel layer.

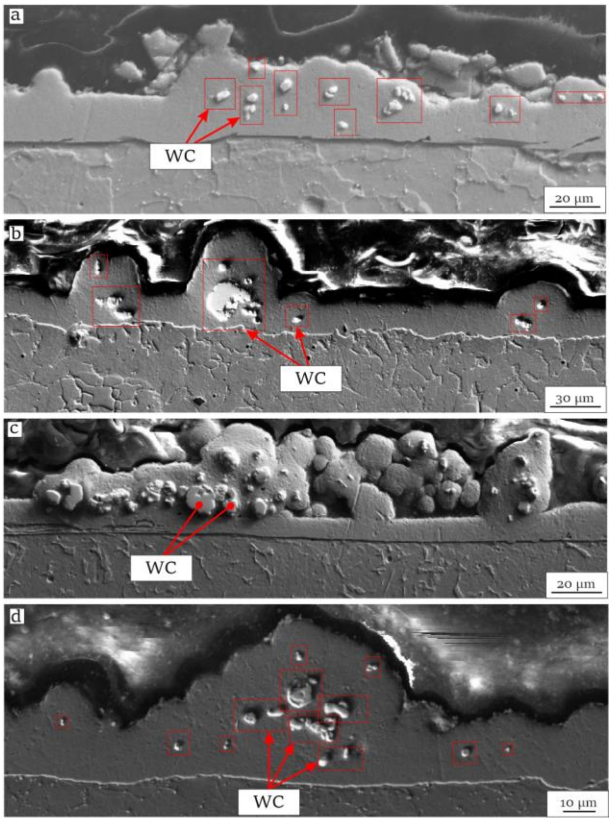
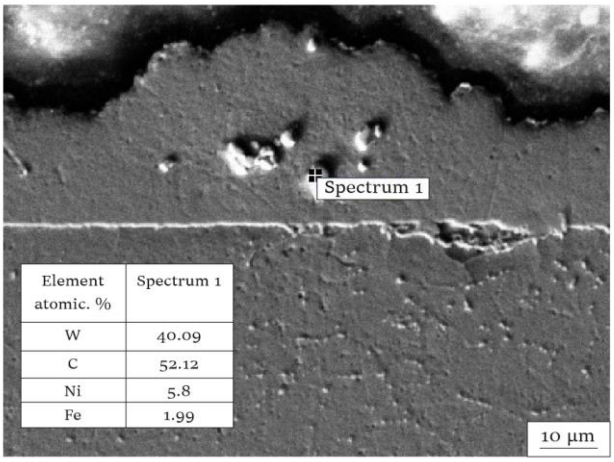


Figure 2 Images of a nickel coating with a concentration of tungsten carbide in an electrolyte: a) 10 g/L; b) 20 g/L; c) 30 g/L; d) 40 g/L.



Element atomic. %	Spectrum 1
W	40.09
C	52.12
Ni	5.8
Fe	1.99

Figure 3 X-ray microanalysis of coatings with different contents of tungsten carbide in the sulfuric acid electrolyte.

DOI: 10.15826/chimtech.2024.11.4.08

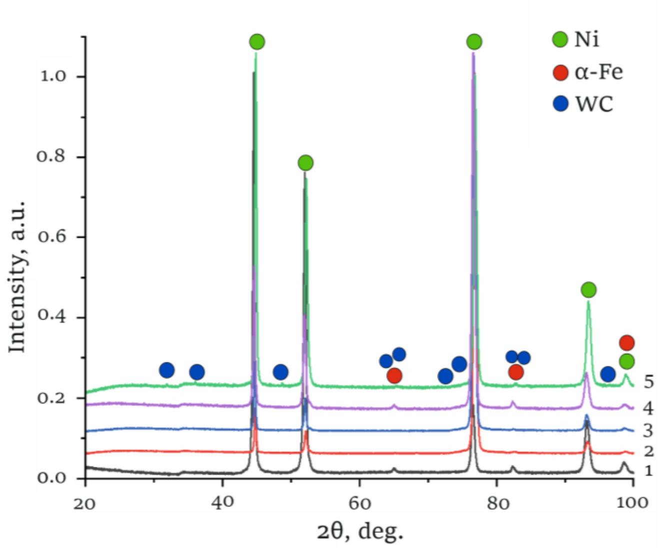


Figure 4 X-ray patterns of coatings with a concentration of tungsten carbide: 1) 0 g/L (nickel coating); 2) 1 g/L; 3) 3 g/L 4) 5 g/L; 5) 7 g/L.

X-ray patterns of coatings obtained from sulfuric acid electrolyte with a WC concentration from 10 to 40 g/L are shown in Figure 5. The highest intensity of tungsten carbide peaks corresponds to a coating with a concentration of 30 g/L of particles in the electrolyte. According to the results of scanning electron microscopy, it was noticed that the largest distribution of particles occurred precisely at this concentration.

It is worth noting that the WC phase is present in coatings at all concentrations. Thus X-ray phase analysis confirms the formation of a composite electrolytic Ni-WC coating.

The images of the scratch grid on composite coatings are shown in Figure 6. As a result of the assessment of the adhesive strength of the coating with a steel plate, it was found that coatings with a low concentration of tungsten carbide have satisfactory adhesion. Peeling and chipping of coatings, including at the intersections of scratches, are not observed (Figure 6a, 6b). However, it was not possible to evaluate the adhesive strength of the coatings formed from the electrolyte at WC concentrations from 10 to 40 g/L, since these coatings have increased hardness and do not allow the tip to reach the base metal. Thus, it can be assumed that tungsten carbide increases the hardness of the composite coating, which suggests an increase in wear resistance.

The results of the microhardness measurement are shown in Table 1. During the tests, it was found that the average microhardness value for steel 20 is 1.65 GPa, for the nickel coating – 0.2 GPa, for the composite coating with an electrolyte WC concentration of 10 g/L – 2 GPa, and for the composite coating with an electrolyte WC concentration of 40 g/L it is 3 GPa. Thus, the introduction of tungsten carbide particles increases the microhardness of the nickel coating by 10–15 times.

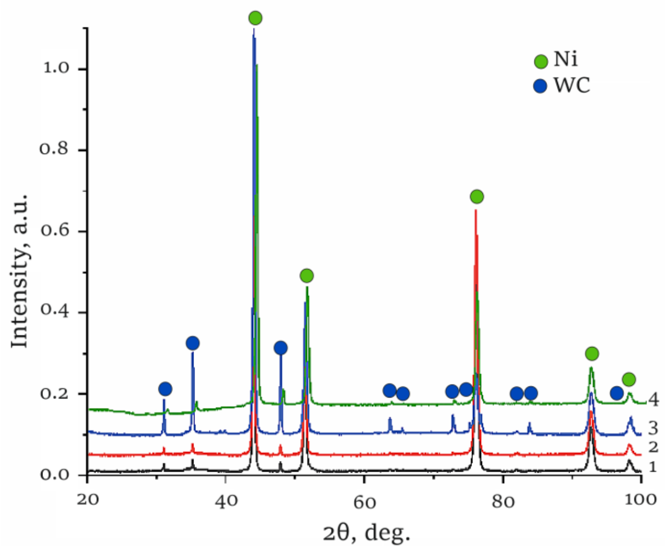


Figure 5 X-ray images of coatings with a concentration of tungsten carbide: 1) 10 g/L; 2) 20 g/L; 3) 30 g/L; 4) 40 g/L.

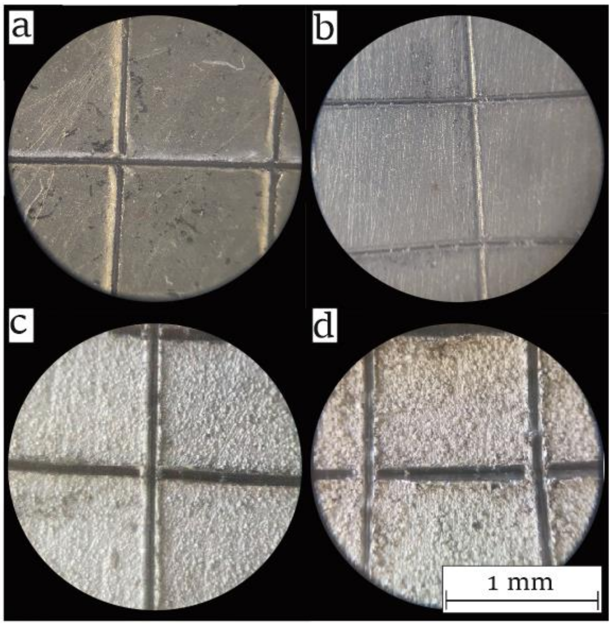


Figure 6 Images of a grid of scratches on the surface of samples with WC content in the electrolyte: 0 g/L (nickel coating) (a), 7 g/L (b), 20 g/L (c), 40 g/L (d).

Figure 7 shows the dependence of the coefficient of friction on the test time for a nickel coating when 10 g/L and 40 g/L of tungsten carbide are added to the electrolyte. The friction process can be divided into a running-in mode and a stationary mode. The coatings enter the stationary friction mode after ~1000 s. The presented curves of the dependence of the coefficient of friction on time are characteristic of all the coating patterns. The coefficient of friction of the coatings is quite stable in the stationary mode.

The average values of the coefficient of friction and the volume of worn material are shown in Table 2. According to the results, it can be noted that the coefficient of friction decreases only for a sample with a concentration of tungsten carbide of 10 g/L, and the amount of wear also decreases.

Table 1 Results of microhardness measurement.

Material	The value of microhardness, GPa	Average microhardness, GPa
Steel	1.742723	1.647957±0.11427
	1.620056	
	1.861446	
	1.677867	
	1.624400	
	1.601975	
	1.481895	
	1.817246	
	1.462469	
	1.589493	
Nickel coating	0.1864291	0.206531 ± 0.01003
	0.2031898	
	0.2252609	
	0.2140272	
	0.2109435	
	0.196387	
	0.2084738	
	0.1973625	
	0.2184725	
	0.2047639	
Ni + 10 g/L WC	2.328804	2.013152±0.20542
	2.331197	
	2.202411	
	1.913441	
	1.729156	
	1.720176	
	2.000607	
	2.178876	
	1.929898	
	1.796953	
Ni + 40 g/L WC	2.935656	3.064056±0.17595
	3.192084	
	2.855765	
	3.153707	
	2.971133	
	2.961443	
	3.55291	
	3.026181	
	3.064593	
	2.927091	

This is due to the fact that with this amount, a uniform distribution of particles is formed in the nickel coating, and there is no agglomeration. At the same time, for the sample with 40 g/L WC, a deterioration in wear resistance is established compared to a nickel coating. This may be due to the small amount of dispersed phase in the nickel deposit as well as the increased porosity of the coating. Also, an increase in the coefficient of friction for the sample with 40 g/L WC may be due to roughness, which affects this indicator. And an increase in volumetric wear is associated with the discoloration of dispersed particles.

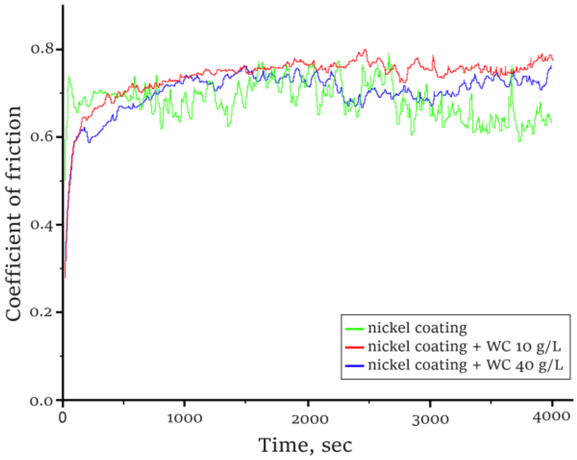


Figure 7 Dependence of the coefficient of friction on the test time of coatings obtained from sulfuric acid electrolyte.

Table 2 The average values of the volume of worn material are presented after dry sliding friction tests.

Material	Coefficient of friction	The average value of the volume of worn material mm³
Nickel coating	0.55	0.06
Ni + 10 g/L WC	0.49	0.042
Ni + 40 g/L WC	0.61	0.063

The average depth of the wear crater on the nickel coating is ≈34 µm (Figure 8a), on the Ni + 10 g/L WC specimen it is ≈20 µm (Figure 8b), and on the Ni + 40 g/L WC specimen it is ≈30 µm (Figure 8c). The friability of the well is due to the uneven distribution of the dispersed phase in the metal matrix. The presence of a rim at the well indicates the plasticity of the nickel matrix.

A sample with a tungsten carbide concentration of 10 g/L showed the highest wear resistance under dry friction conditions. The high result is due to the good resistance of the WC solid phase to plastic deformation and abrasion. In many cases, the wear resistance of a material is directly related to its hardness. Despite the fact that the sample with a concentration of WC 40 g/L had the highest microhardness, lower wear resistance was shown.

4. Limitation

In the future, according to this study, it is necessary to: select the necessary organic additive and its concentration for a more uniform distribution of the dispersed phase in the nickel matrix; also adjust the mode to reduce hydrogen release. It is also planned to study in more detail the wear resistance of these composite coatings in other conditions.

5. Conclusions

Composite coatings are formed on the surface of structural steel by electrodeposition. The coatings are a nickel matrix with tungsten carbide particles. The coatings are characterized by increased roughness due to the formation of encapsulated particles in the nickel layer.

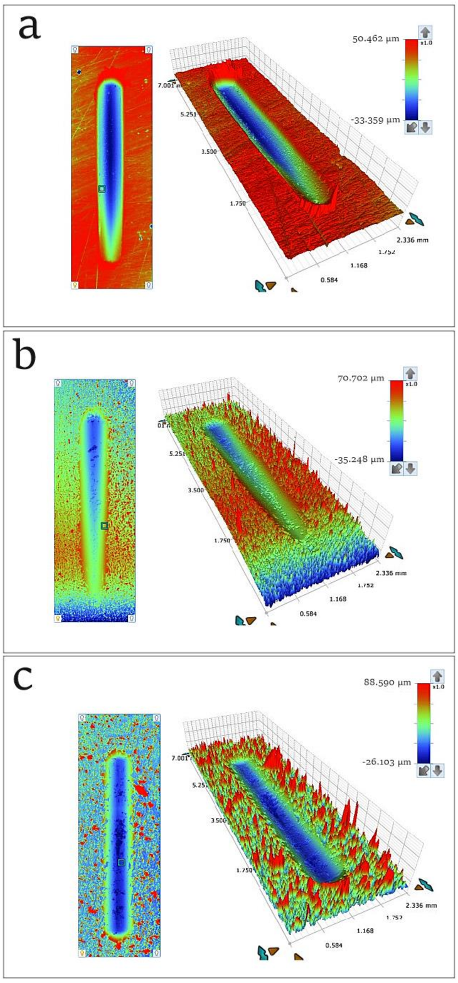


Figure 8 Reliefs of the wear craters after dry sliding wear tests. recorded using an optical profilometer nickel coating (a), Ni + 10 g/L WC (b), and Ni + 40 g/L WC (c).

The thickness of the CEC is 20–30 µm. The highest microhardness of 3.1 GPa was recorded in the specimen with the particle concentration of 40 g/L. Such microhardness is 15 times greater than that of the nickel coating and 1.8 times greater than that of steel 20. The specimen with a WC content of 10 g/L in the electrolyte has the best wear resistance. This specimen showed the smallest amount of worn material. This result is associated with a more uniform distribution of particles in the nickel coating. Thus, the results obtained allow us to conclude that Ni-WC composite coatings have higher mechanical characteristics than standard nickel coatings, although additional studies are currently being conducted to better understand the structure and properties of these coatings.

• Supplementary materials

No supplementary materials are available.

• Funding

The work was carried out in accordance with the state task of the Ministry of Education and Science of Russia (project FSUN-2023-0009).

• Acknowledgments

None.

• Author contributions

Conceptualization: E.B., O.N.
Data curation: P.P.
Formal Analysis: P.P., E.B., O.N, V.K.
Funding acquisition: E.B.
Investigation: P.P., E.B., O.N, V.K.
Methodology: P.P., E.B., O.N, V.K.
Project administration: E.B.
Resources: P.P., E.B., O.N.
Software: P.P., E.B., O.N.
Supervision: P.P.
Validation: E.B., O.N.
Visualization: P.M.
Writing – original draft: P.P.
Writing – review & editing: P.P., E.B., O.N.

• Conflict of interest

The authors declare no conflict of interest.

• Additional information

Author IDs:
Evdokiya G. Bushueva, Scopus ID 25627090600;
Oksana N. Nogorodtseva, Scopus ID 55796868200.

Website:
Novosibirsk State Technical University, https://en.nstu.ru/.

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DOI: 10.15826/chimtech.2024.11.4.08