

Effect of Electrodeposition Parameters on the Composition and Surface Topography of Nanostructured Coatings by Tungsten with Iron and Cobalt

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Abstract

The electrodeposition of binary and ternary coatings Fe-W and Fe-Co-W from mono ligand citrate electrolyte has been investigated. The Fe-Co-W coatings were formed from electrolytes, which composition differs in the ratio of the concentrations of the alloying components and the ligand content. The investigation results indicate a competitive reduction of iron, cobalt and tungsten, the nature of which depends both on the ratio of electrolyte components, and electrolysis parameters. The effect of both current density amplitude and pulse on off time on quality, composition and surface morphology of the galvanic alloys was determined. Coatings deposited on a direct current with a density of more than 6.5 A/dm², crack and peel off from the substrate due to the inclusion of Fe (III) compounds containing hydroxide anions. The use of non-stationary electrolysis allows us to extend the working range of current density to 8.0 A/dm² and form electrolytic coatings of sufficient quality with significant current efficiency and the content of the refractory component. The presence of the Co₇W₆, Fe₇W₆, α-Fe, and Fe₃C phases detected in the Fe-Co-W deposits reflects the competition between the alloying metals reducing from hetero-nuclear complexes. The surface of binary and ternary coatings is characterized by the presence of spherical agglomerates and is more developed in comparison with steel substrate. The parameters *Ra* and *Rq* for electrolytic alloy Fe-W are of 0.1, for Fe-Co-W are 0.3, which exceeds the performance of a polished steel substrate (*Ra* = 0.007 and *Rq* = 0.010). These properties prospect such alloys as a multifunctional layer are associated with structural features, surface morphology, and phase composition.

1. Introduction

Electrochemical deposition of alloys is an important and promising direction of electroplating on several grounds. Due to the combination of valuable qualities of individual metals, coatings with alloys [1, 2] give the surface of products various chemical and physic-mechanical properties, the level of which can be quite easily varied. Electrodeposited multicomponent and synergic alloys are characterized by super additive enhancement of functional properties – catalytic [3–5], corrosion

resistant [6–9], hard and wear resistant [10–12], magnetic [13–15] etc. Among the practically important alloys, the main place is occupied by electrolytic compositions formed by the metals of the iron triad (iron, cobalt and nickel) with refractory metals, namely tungsten, molybdenum, vanadium and zirconium [16–18].

For electroplating of iron alloys with refractory metals, ammonium-citrate electrolytes based on Fe (II) are mainly used. While Fe (II) in such electrolytes oxidized by oxygen, oxometalates and in the anodic process to the Fe (III). That is why the use of electrolytes based on iron (III) for the coatings with cobalt and tungsten deposition is seen as a modern trend in electroplating [13, 19].

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The stability of the electrolyte, the quality of the coatings, the morphology of the surface and the current efficiency are significantly influenced by the choice of ligand and the ratio of the components in the electrolyte. Solutions of citrate, chloride-citrate, diphosphate and diphosphate-citrate complexes are more commonly used to deposit binary and ternary cobalt alloys with tungsten and molybdenum, and the addition of ethylene diamine tetraacetate (EDTA) electrolytic bath to the composition increases the content of refractory components [20, 21].

Significant influence on the process of alloys electrodeposition can be achieved using pulsed electrolysis, which is one of the most effective methods for controlling electrode polarization and redistributing the partial discharge currents of electrolyte components and, thus, changing the composition and properties of the alloy in the desired direction within relatively wide limits [16, 22–24].

Despite a fairly significant amount of research, the issues of control and management of the composition, surface morphology of the coatings remain relevant. This paper presents the investigation results on the effect of electrolyte composition and electrolysis mode on the composition, surface morphology and topography of binary Fe-W and ternary Fe-Co-W coatings.

2. Experimental

Coating with alloys was applied to substrates made of copper M0 and mild steel. Preparatory operations for treating the surface were carried out according to the generally accepted method following the nature of the material. Fe-W and Fe-Co-W coatings were deposited from citrate complex electrolytes based on Fe(III) at various ratios of complexing ions and ligand. Electrodeposition was carried out in two modes: at direct current (DC) of density i 3.0–9.0 A/dm²; and at pulse current (PC) when varying the amplitude of the unipolar current 3.5–9.5 A/dm², the pulse duration (on time) t_{on} was 5–10 ms and the pause one (off time) t_{off} was 10–20 ms. The electrolyte temperature was in the range of 20–25 °C. The electrolysis was carried out using insoluble anodes made of stainless steel AISI 304. DC electroplating was performed using stabilized DC source B5-47; and an IPC– PRO M potentiostat was used for PC deposition.

The chemical composition of the coatings (in mass and mol %) was determined by energy dispersive X-ray spectroscopy by an Oxford INCA Energy 350 electron probe microanalysis integrated into the system of the SEM. The X-rays were

excited by exposure of the samples to a beam of 15 keV electrons. The surface morphology of the deposits was studied with a Zeiss EVO 40XVP scanning electron microscope (SEM). The surface roughness of the coatings was determined using an atomic force probe microscope (AFM) NT-206. Scanning was performed by the contact method using a CSC cantilever B probe with a lateral resolution of 3 nm. Analysis of AFM images was performed using Explorer software. The height distribution and particle diameter, surface asymmetry, mean deviation (R_q), and R_a and R_z parameters, which reflect surface roughness, were determined.

The X-ray spectroscopic analysis of the phase composition was performed on a DRON-3M X-ray diffractometer in the emission of cobalt anode $\text{Cu-K}\alpha$ ($\lambda = 1.5405 \text{ \AA}$). X-ray diffractograms were recorded in a discrete mode with a step of $2\theta = 0.1^\circ$ and an exposure at each point of 20 s at a current voltage of 35 kV and a current of 20 mA.

2. Results and discussion

Coatings with Fe-W and Fe-Co-W alloys were deposited from complex electrolytes, and citrate ions ($\text{C}_6\text{H}_5\text{O}_7^{3-}$) were used as a ligand. The coatings Fe-W were deposited from electrolyte of composition, mol/dm³: $\text{Fe}_2(\text{SO}_4)_3 - 0.1-0.15$; $\text{Na}_2\text{WO}_4 - 0.04-0.06$; $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 - 0.2-0.3$; $\text{Na}_2\text{SO}_4 - 0.1$; $\text{H}_3\text{BO}_3 - 0.1$. The acidity of the electrolyte was regulated by the addition of NaOH or H_2SO_4 and maintained in the pH range 3.0–4.0. It was established that a higher content of tungsten in coatings corresponds to an electrolyte with a content, mol/dm³ of $\text{Fe}_2(\text{SO}_4)_3 - 0.1$ and $\text{Na}_2\text{WO}_4 - 0.06$. The increasing tungstate concentration in the electrolyte over 0.06 mol/dm³, is impractical because of coatings cracking due to high internal stresses. Raising the pH to 5.0–6.0 reflects in the increase in oxygen content, and a decrease in current efficiency (to 50–60%), and the stability of electrolyte deteriorates.

The dependences of the tungsten content in Fe-W coatings and CE from the current density are of extreme nature. Optimal indicators of the electrodeposition for both the DC and PC modes are observed at current densities in the range from 4.5 to 8.5 A/dm². Coatings deposited by a DC with a density of more than 6.5 A/dm², crack and peel off from the substrate due to the inclusion of Fe (III) compounds containing hydroxide anions. The PC electrolysis allows us to extend the working range of current density to 8.0 A/dm² and produces coatings of sufficient quality, current efficiency and the content of the refractory component.

Table
Electrolytes for electrodeposition Fe-Co-W coatings

The concentration of components, mol/dm ³	Ratio of concentrations: $c(\text{Fe}^{3+}) : c(\text{Co}^{2+}) : c(\text{WO}_4^{2-}) : c(\text{Cit}^{3-})$		
	W1	W2	W3
	1 : 1 : 0.4 : 2	1 : 1.3 : 0.6 : 2.7	1 : 1.3 : 0.6 : 3.3
Fe ₂ (SO ₄) ₃ ·9H ₂ O	0.075	0.075	0.075
CoSO ₄ ·7H ₂ O	0.15	0.2	0.2
Na ₂ WO ₄ ·2H ₂ O	0.06	0.06	0.06
Na ₃ Cit·2H ₂ O	0.3	0.4	0.5
Na ₂ SO ₄	0.15	0.15	0.15
H ₃ BO ₃	0.1	0.1	0.1
pH	3.8	4.3	4.55

Analysis of the coatings composition indicates that, in addition to the main components (Fe, W), they contain some amount of oxygen. It should be stated uneven distribution of the alloy components on the surface of the covers deposited at a DC mode: on the hills of the relief there is a high content of metals; in the valleys, the oxygen concentration is rather high. Coatings deposited in a pulsed mode differ by a more uniform distribution of components over the surface and lower oxygen content, which is explained by the features of the electro-crystallization. During the pulse, Fe(III) is reduced to metallic iron in two steps [6, 9, 22]; and tungstates produce oxides in intermediate oxidation states. During the period of current interruption, the reagents adsorption, chemical reduction of tungsten oxides by hydrogen atoms and ligand release from complexes are realized more fully. Thus, the use of a programmable electrolysis allows obtaining more uniform covers due to the chemical stages progress and more complete reduction of alloying metals. The pulse duration in the range of 5–10 ms and the pause one in the interval of 10–20 ms is the ranges that are recommended as optimal for the deposition of Fe-W alloy.

The Fe-Co-W coatings were formed from electrolytes (Table), which composition differs in the ratio of the concentrations of the alloying components and the ligand content. The investigation results indicate a competitive reduction of iron, cobalt and tungsten, the nature of which depends both on the ratio of electrolyte components and electrolysis parameters (Fig. 1). In the coatings deposited from the electrolyte W1 at current densities 2.0–4.0 A/dm², there is a slight variation in the content of iron and cobalt, and the content of tungsten increases with increasing i , but does not exceed 8 mol%.

An increase in the cobalt (II) concentration with a simultaneous increase in the ligand to 0.4 mol/dm³ (W2, Table) leads to an inversion of the ratio of metals in the alloy (Fig. 1b). This is due to changes in the composition of heteronuclear complexes, discharged at the electrode. When the current density is from 3 to 6 A/dm², the cobalt content in the coating exceeds the iron content. The tendency to a decrease in the cobalt content is observed with an increase in the current density (Fig. 1b). Iron and cobalt co-deposits in the alloy in a ratio 1:1 at a current density of 6 A/dm². A further increase in i contributes to a significant increase in the iron content due to cobalt and tungsten. The content of the refractory component in the alloy varies within 8–10 at% with a tendency to decrease with increasing current density.

When the alloy is deposited from the electrolyte W3 *ceteris paribus* (Fig. 1c), the tungsten content in the coatings slightly increases (9–11 at.%). However, the tendency to decrease W content in the coatings with increasing current density is maintained. At the same time, the competitive reduction of iron and cobalt becomes more significant as the current density increases from 3 to 7 A/dm², as can be seen from Fig. 1c.

The Fe-Co-W deposited in the stationary mode has a spheroid surface morphology with a grain size of 2–6 μm (Fig. 2a). The use of a pulsed electrolysis mode allows the deposition of coatings with a more uniform surface (Fig. 2b) and expanding the range of the W content in the alloy. The coatings formed by the unipolar pulse current from the W3 electrolyte (Table) contain W of 12.5 at.% and are distinguished by a more developed surface on which the agglomerates formed from spheroids are visualized (Fig. 2b). As before, the time parameters of pulsed electrolysis are an effective

tool for controlling the composition and character of the surface of the coatings. The pulse duration at a fixed pause time contributes to an increase in the content of cobalt and tungsten in the alloy at the current densities studied. At the same time, there is a tendency to increase the iron content due to cobalt and tungsten with an increase in current

density, in contrast to DC electrolysis. The efficiency of the process is almost doubled when using a unipolar pulsed current compared to DC: at a current density of 3 A/dm², the current efficiency is 70–75%, and at 4 A/dm² the current efficiency (CE) decreases up to the level of 63–68%. But CE remains higher than at DC electrodeposition.

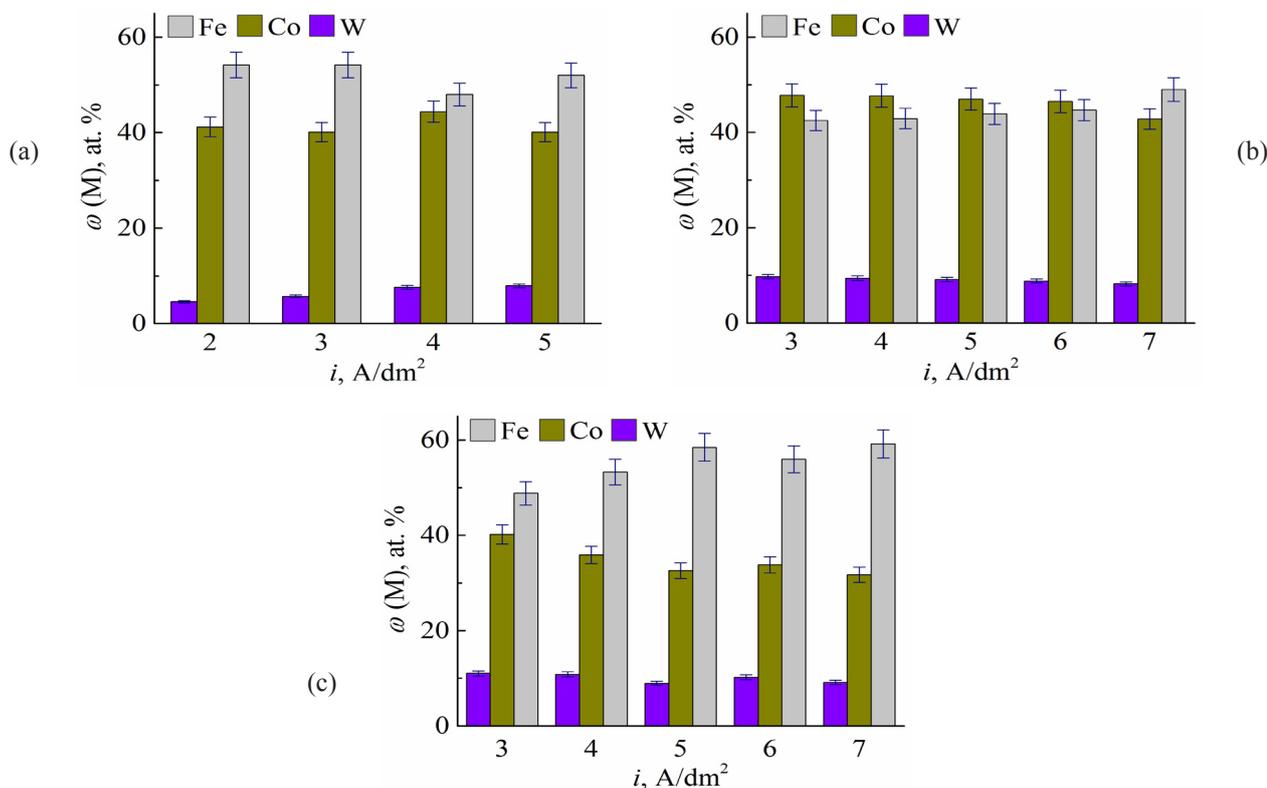


Fig. 1. Effect of current density on the composition of Fe-Co-W coatings deposited from electrolytes: (a) – W1; (b) – W2; (c) – W3.

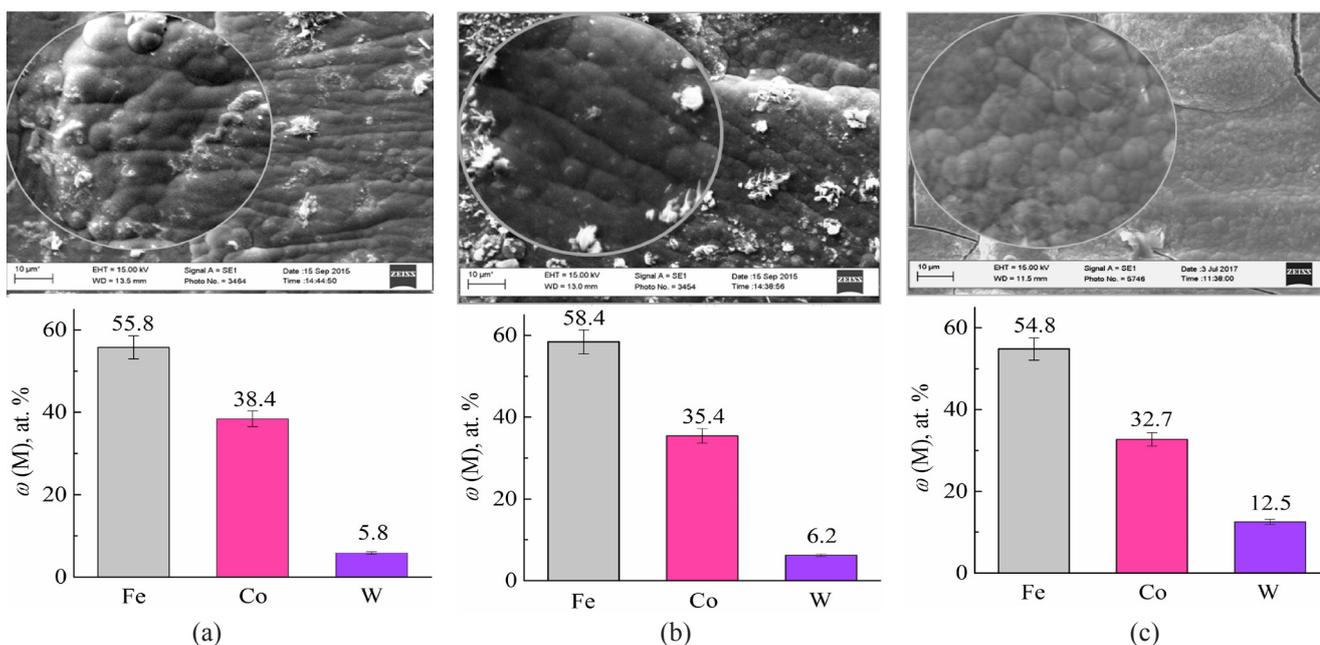


Fig. 2. Morphology and composition of Fe-Co-W alloys deposited from electrolytes: (a), (b) – W1; (c) – W3. Electrolyses parameters: (a) – $i = 3$ A/dm²; (b) – $i = 3$ A/dm², $t_{on}/t_{off} = 50/50$ ms; (c) – $i = 5$ A/dm², $t_{on}/t_{off} = 20/20$ ms.

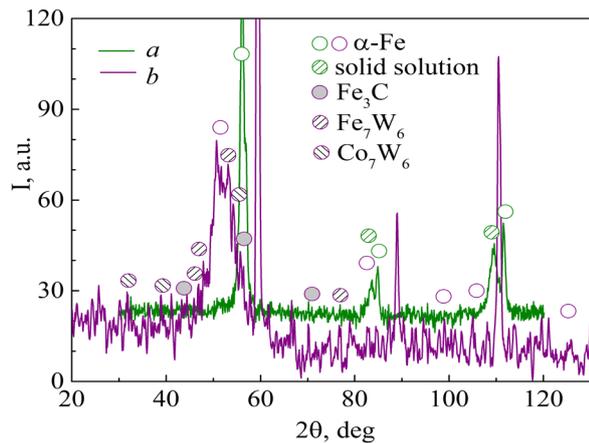


Fig. 3. X-ray diffraction patterns for the covers Fe-W (a) and Fe-Co-W (b) deposited by DC mode.

The X-ray patterns for Fe-W coating deposited at substrate made from steel (Fig. 3a) shows two systems of diffraction lines corresponding to phases with a body-centered cubic crystal lattice (*bcc*) characteristic of alloying components. One system of narrower lines is characteristic for α -Fe, and the second one corresponds to a solid solution of tungsten in α -Fe. Figure 3b shows X-ray diffraction patterns for Fe-Co-W (composition in terms of metal, at.%: Fe – 54, Co – 36, W – 10) deposited by DC on a substrate made of copper; coating thickness of 30 μm . We can conclude the amorphous and crystalline structure of the electrolytic alloy. We see some copper lines and lines corresponding to the phases of the intermetallic compounds Co_7W_6 and Fe_7W_6 , as well as α -Fe and cementite Fe_3C . Also, at angles 2θ 50–55°, a low halo with a width of about 10°, corresponding to an amorphous structure, appears. The crystallite size of the amorphous part is $L = 77 \text{ \AA}$. The presence of the Co_7W_6 , Fe_7W_6 phases detected in the Fe-Co-W deposits reflects the competition between the alloying metals reducing. It is well known that cobalt, in contrast to tungsten and iron, is characterized by a hexagonal crystal lattice, therefore, the co-reduction of metals contributes to the formation of amorphous structures [25]. Let's note that the formation of the Co_7W_6 and Fe_7W_6 phases is a prerequisite for increasing the microhardness of the coatings.

An assessment of the surface topography of electrolytic alloys indicates that the Fe-W coating has a pronounced fine-crystalline surface consisting of a variety of grains 200–400 nm in size (Fig. 4a). For electrolytic alloy Fe-W the parameters R_a and R_q calculated at the scanning area of $48 \times 48 \mu\text{m}$ are 0.2, which exceeds the perfor-

mance of a polished steel substrate, the roughness of which is fixed at $R_a = 0.007$ and $R_q = 0.010$. Such indices indicate a significant development of the coating surface in comparison with the substrate. The surface of Fe-Co-W coatings is characterized by the presence of spherical agglomerates (Fig. 4b). The profile of the surface cross section allows you to determine the size of agglomerates 2.5–3.5 μm , which are formed from smaller spheroids with a diameter of 0.3–0.5 μm (Fig. 4c). The parameters R_a and R_q for the electrolytic alloy Fe-Co-W are of 0.3, which noticeably exceeds the performance of a binary coating and polished steel substrate. It was previously shown [26, 27] that the globular nature of the surface is due to the presence of tungsten in the coating and may be a prerequisite for the high microhardness and catalytic activity of the material.

4. Conclusions

1. It was shown that the composition and morphology of the surface of Fe-W and Fe-Co-W binary and ternary alloys are controlled by varying the ratio of the concentrations of the alloying components.

2. The ability to control the composition of Fe-W and Fe-Co-W coatings and their surface morphology using various electrolysis modes and parameters is proved: CD and PC with varying current density, pulse/pause duration, which provide the formation of coatings with high content alloying components at a deposition rate of 15–30 μm per hour with electrolysis efficiency of 60–85%. Coatings Fe-W and Fe-Co-W deposited by pulsed current contain less oxygen and have a more uniform distribution of alloy components over the surface and developed surface.

3. The surface of binary and ternary coatings is characterized by the presence of spherical agglomerates and is more developed in comparison with steel substrate. The roughness parameters for electrolytic alloys were determined by AFM measurements.

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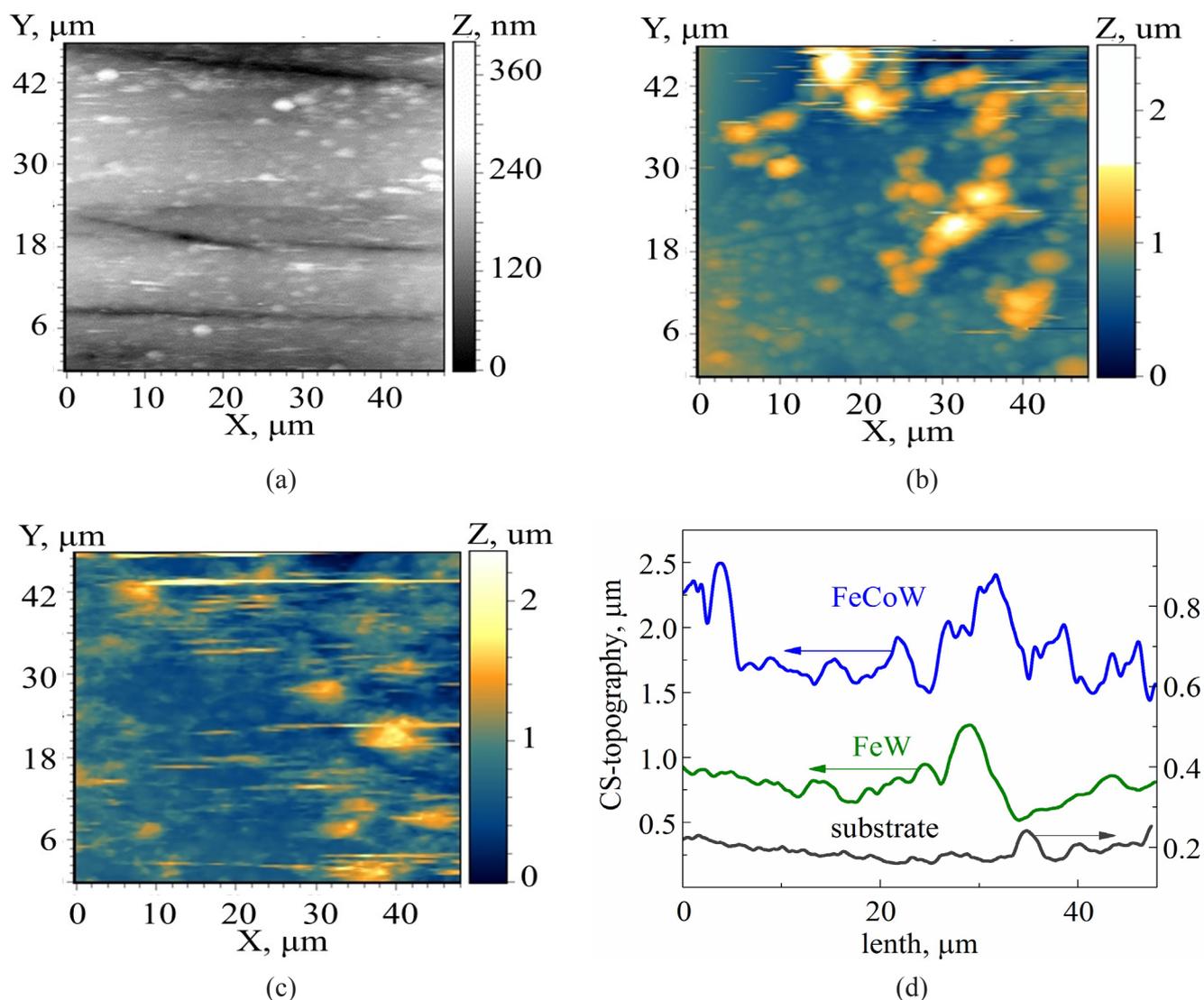


Fig. 4. Surface 2D maps for: (a) – mild steel substrate; (b) – Fe-W; (c) – Fe-Co-W; and (d) – cross section profile of substrate and electrodeposited coatings on scanning surface area $48 \times 48 \mu\text{m}$.

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