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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS OF ACTIVE ELECTROCATALYTIC SYSTEMS ON Ni–Mo BASE FOR WATER ELECTROLYSIS

Specialty: 2316.01 – Chemical kinetics and catalysis

                         2308.01 – Electrochemistry

Field of science: Chemistry

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Baku – 2023

The dissertation work was performed in the "Nanoelectrochemistry and electrocatalysis" laboratory of the Institute of Catalysis and Inorganic Chemistry, named after Acad. M.Nagiyev of the Ministry of Science and Education of the Republic of Azerbaijan

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Dissertation Council BED 1.15 on specialties 2316.01 – "Chemical kinetics and catalysis" and 2308.01 – "Electrochemistry" established based on ED 1.15 Dissertation Council of the Supreme Attestation Commission under the President of the Republic of Azerbaijan under the Institute of Catalysis and Inorganic Chemistry named after acad. M.Nagiyev of the Ministry of Science and Education of the Republic of Azerbaijan
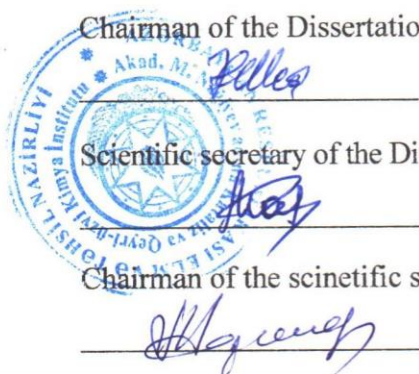
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GENERAL FEATURES OF THE RESEARCH WORK

The relevance and scientific elaboration of the topic. In the modern era, it is impossible to imagine the development of humanity without energy; even "energy revolutions" have occurred several times in human history. Recently, energy demand has doubled every 30 years. Energy consumption is constantly proliferating, with the bulk of this consumption coming from oil and gas products, which makes it urgent to switch to alternative energy sources that will be more profitable from an economic and environmental point of view.

Since the 80s of the last century, due to limited oil resources, attention to hydrogen energy has sharply increased, and petroleum products are proposed to be replaced with hydrogen from coal. In connection with the warming of the planet and the protection of the ecological environment, hydrogen energy has again become a pressing issue in recent years.

The purity of hydrogen depends on the raw materials from which it is obtained. Hydrogen can be produced in different ways. These methods can be divided into two groups: renewable and non-renewable resources. Non-renewable resources include methane and coal. Hydrogen can be obtained from them by gasification and thermocatalytic processes. An example of a renewable resource is hydrogen production through water electrolysis. Hydrogen produced from coal and natural gas is called "grey" and "blue" hydrogen, respectively, while hydrogen produced by electrolysis of water using alternative energy sources is called "green" hydrogen.

One of the main problems of materials science in the modern era is the creation of technologies for producing new multifunctional materials that are competitive in the high-tech sector of the economy. For this purpose, one of the urgent tasks is the development of technologies for producing thin films with high electrocatalytic properties. In this regard, recent alloys based on nickel and molybdenum have attracted increasing attention from researchers since these alloys meet all the requirements for catalysts, possessing the abovementioned properties.

The electrochemical method is more promising for producing thin layers of alloys with electrocatalytic properties and high perfor-

mance. Therefore, the presented dissertation examines the production of multifunctional thin films containing predominantly nickel and molybdenum by the electrochemical method and the study of their electrocatalytic properties in water electrolysis.

The object and subject of the research.

The preparation of Ni - Mo thin films by electrochemical method and research in the process of water electrolysis.

The aim and tasks of the study. The main goal of the dissertation work is the electrochemical application of thin layers of Ni-Mo of various compositions from an alkaline electrolyte and the study of the electrocatalytic properties of the resulting thin layers. Therefore, to achieve this goal, the research work planned to perform tasks in the following sequence:

- Study of the kinetics and mechanism of the process of electrochemical deposition of Ni and Mo, both separately and during codeposition;

- Study of the influence of various factors - temperature, electrolyte composition, current density, electrode materials, etc. on the process of coelectrodeposition of Ni and Mo;

- Study of the phase and elemental composition, morphological structure of NiMo thin films synthesized by the electrochemical method;

- Study of the electrocatalytic properties and corrosion resistance of thin films of Ni - Mo of various compositions obtained by the electrochemical method in the reaction of water electrolysis.

Research methods. Experimental studies on the topic of the dissertation were carried out using the following methods of analysis and measuring instruments:

- To study the kinetics and mechanism of the process of joint electrodeposition of thin layers of Ni - Mo - potentiostat "IVIUMSTAT Electrochemical Interface";

- "D2 Phazer" diffractometer for X-ray phase analysis;

- To study the chemical elemental composition and morphology - a scanning electron microscope of the "Carel Zeiss Sigma" brand;

- To measure the thickness of thin layers - microscope "MII-4";

- To measure the hardness of thin layers - the "PMT-3" device.

The main provisions of the dissertation:

- The nature of polarization during the electroreduction of Ni and Mo separately;
- Determining the co-deposition potential field of Ni and Mo on Pt electrode from alkaline electrolyte by electrochemical method;
- The influence of various factors (current density, temperature, concentration of components, etc.) on the composition of thin layers of Ni - Mo deposited from an alkaline electrolyte by the electrochemical method;
- Chronoampermetric study of the electroreduction of molybdate ions by nickel ions from an alkaline electrolyte using the electrochemical method;
- Regression equation by the method of statistical planning of experiments for the process of obtaining Ni - Mo thin films from alkaline electrolyte by electrochemical method;
- Morphology, chemical element and phase composition of Ni - Mo thin films deposited from alkaline electrolyte by electrochemical method;
- Study of the electrocatalytic properties and corrosion resistance of Ni - Mo thin films of various compositions deposited from an alkaline electrolyte by the electrochemical method.

Scientific innovation of the study:

The scientific innovation of the dissertation work lies in the fact that the electrolyte composition and electrolysis conditions for synthesizing Ni-Mo thin films by electrochemical deposition from an alkaline electrolyte have been determined (Az.patent № I 2020065). The electrocatalytic properties of Ni - Mo thin films in the electrochemical reduction of water were studied using polarization curves drawn by the potentiodynamic method. This method determined the kinetics and mechanism of the electrodeposition of Ni, Mo, and Ni-Mo thin films. A regression equation was established by the mathematical modeling method for the co-electrodeposition of Ni - Mo thin films.

The theoretical and practical significance of the research. The theoretical significance of the dissertation work lies in the study of the kinetics and mechanism of the process of electrodeposition of thin la-

yers of Ni - Mo from an alkaline electrolyte and the study of the electro-catalytic activity of these layers both as a cathode and as an anode material in the process of water electrolysis. The practical significance of the research work consists of replacing noble metals (Pt, Pd, Au, Ag) with cheaper electrodes during water electrolysis with electrocatalytically active and corrosion-resistant thin films of Ni-Mo. For this purpose, the obtained data on the microhardness, corrosion resistance, and catalytic activity of electrodeposited thin layers may be necessary for researchers working in the alternative field.

Approbation of work: The main results of the dissertation were presented at the following conferences:

1. Materials of the scientific conference "Nagiyev Readings" dedicated to the 110th anniversary of academician M. Nagiyev (Baku, Azerbaijan, 2018);

2. International scientific-practical conference and school of young scientists. "Chemistry, chemical technologies and ecology: science, production, education". Collection of Abstracts (Makhachkala, Dagestan, 2018);

3. V International Scientific and Practical Conference. "Theory and practice of modern electrochemical production." Collection of Proceedings (St. Petersburg, Russia, 2018);

4. International scientific and technical conference. "Modern electrochemical technologies and equipment" (Minsk, Belarus, 2019);

5. Sixth All-Russian Conference with international participation. "Fuel cells and power plants based on them" (Moscow, Russia, 2019).

The main content of the dissertation has been published in 12 scientific works. 1 of the works is a patent, 6 are articles (2 articles in WEB of Science, 1 article in Scopus), and 5 are theses.

Name of the research organization of dissertation work. The dissertation work was carried out by the research plan of the "Nanoelectrochemistry and electrocatalysis" laboratory of the Institute of Catalysis and Inorganic Chemistry, named after academician M. Nagiyev of the Ministry of Science and Education of the Republic of Azerbaijan. Topic: 6 Work: 6.4 (2015–2020). (State registration No. 0115 Az 2096).

The total symbolic volume of the dissertation, indicating the unit volumes of the dissertation separately. The dissertation work - introduction (13559 characters), chapter I (48933 characters), chapter II (19250 characters), chapter III (60476 characters), chapter IV (21153 characters), general results (3475 characters), total - 166946 characters.

Volume and structure of the dissertation: The dissertation includes an introduction and four chapters - a literature review, an experimental part, the third and fourth chapters, and general results. The dissertation concludes with general results, a list of 234 cited sources, abbreviations, and legends. The volume of the dissertation is 149 pages with computer printing. The work contains 50 figures and five tables.

Personal participation of the author. The author independently participated in the dissertation work - planning the work, collecting literary materials, conducting basic experimental studies and mathematical calculations, and personally participating in writing theses, articles, and dissertations. The author's share in published scientific works is decisive.

THE MAIN CONTENT OF THE WORK

The introduction of the dissertation substantiates the relevant topic, scientific novelty, and practical significance of the obtained results.

The first chapter (literature review) provides data on the production of thin films by the electrochemical method, suitable as electrodes for water electrolysis, and an analysis of the properties of the resulting thin films [4].

The second chapter (experimental and methodological part) of the dissertation describes reagents, devices, and research methods used in the investigation.

The electrochemical deposition process of Ni-Mo thin films was carried out using the IVIUMSTAT Electrochemical Interface potentiostat consisting of a three-electrode glass cell equipped with a computer. The following electrode materials were used in the electrolysis process:

The working electrode was a platinum wire with a surface of $3 \cdot 10^{-3}$, $4 \cdot 10^{-3}$ dm^2, a nickel plate with a surface of $2 \cdot 10^{-2}$ dm^2, and a plate of Steel-3 with a surface of $7.6 \cdot 10^{-2}$ dm^2. Pt plate with a sur-

face area of $4 \cdot 10^{-2}$ dm^2 was used as an auxiliary electrode. Silver /silver chloride (Ag/AgCl/KCl) was used as a reference electrode.

Surface analysis, chemical elemental composition, relief, and morphology of electrodes with thin layers of Ni-Mo deposited on their surface by the electrochemical method were carried out using a scanning electron microscope of the "Carel Zeiss Sigma" brand. Phase analysis was carried out using a D2 Phazer diffractometer of the German company "Bruker." The thickness of Ni-Mo thin films was determined on an MII-4 device and hardness on a PMT-3 device.

The results of electrochemical studies are presented in **the third chapter**, and the electrocatalytic activity of Ni-Mo thin films, corrosion resistance, SEM, and X-ray diffraction analyses are discussed in **the fourth** chapter.

The short abstract of the experiments presented in the third chapter.

Chapter 3 presents the research results of the electrochemical deposition process of Ni-Mo thin films.

Figure 1 shows the polarization curves of the electroreduction of nickel ions at different concentrations of nickel-sulfate to study the kinetics and mechanism of electrochemical precipitation of nickel ions from an alkaline electrolyte.
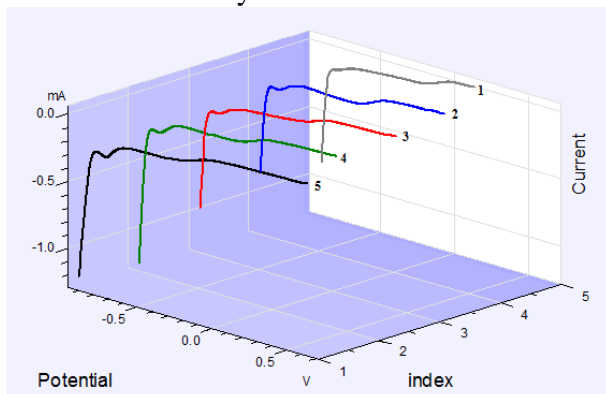

Figure 1. The voltammetry curves of electroreduction of nickel ions from alkaline electrolyte at different concentrations on Pt electrode. Electrolyte (M): NiSO$_4$: 1 – 0.05, 2 – 0.1, 3 – 0.2, 4 – 0.4. 5 – 0.8, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7, E$_\upsilon$ = 40 mV/s, pH = 11.2, T = 298 К.

The concentration of nickel sulfate was changed between $0.05 \div 0.8$ M. From the polarization curves, it is clear that at potential values of -0.2 $\div$ -0.48 V, a small "plateau" appears on the curves, associated with the reduction of nickel ions. At a potential of -0.2 V, the formation of a light gray nickel precipitate is observed on the electrode surface. A change in the concentration of nickel sulfate within the range of $0.05 \div 0.8$ M has virtually no effect on the height of this "plateau."

Figure 2 (a) shows the cyclic polarization curve of the electrochemical reduction process of nickel ions in an alkaline electrolyte, and for comparison, figure 2 (b) shows the cyclic polarization curve of the background without nickel ions. Comparing Figure 2 (a) and (b), it was determined that the potential range of nickel ions in the platinum electrode in the electrolyte given above and under the electrolysis conditions is $-0.65 \div -0.78$ V. The evolution of hydrogen begins from a potential value of -0.78 V.
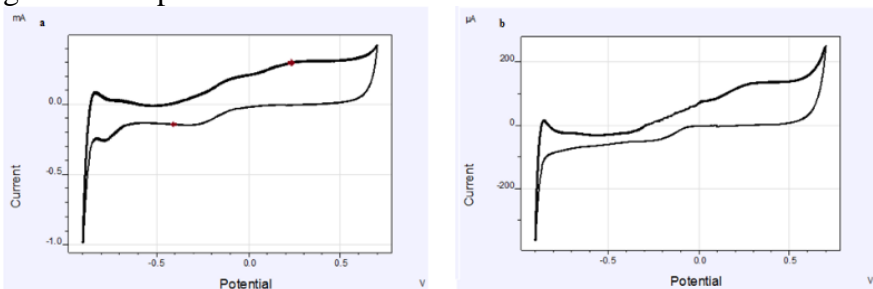
Figure 2. Cyclic voltammetry curves in alkaline electrolyte on Pt electrode. Electrolyte (M): a) $NiSO_4 - 0.107$, $NiCl_2 - 0.13$, $H_3BO_3 - 0.1$, $NH_4OH - 7$; b) $H_3BO_3 - 0.1$, $NH_4OH - 7$. $E_\upsilon = 80$ mV/s, T = 298 К.

It can be seen from the polarization curve in Figure 2 (a) that the oxidation of nickel ions at the anode occurs in two stages at the potentials of -0.12 V and +0.21 V. It is known that the oxidation of nickel as an anode in alkaline electrolytes leads to the passivation of the nickel electrode.

The temperature-kinetic method was used to more accurately determine the nature of cathodic polarization during the reduction of nickel ions (Fig. 3). For this purpose, linear cathodic polarization curves were plotted depending on the temperature of the electrolyte

on the nickel electrode. During the study, the electrolyte temperature varied in the 298 ÷ 333 K [7].

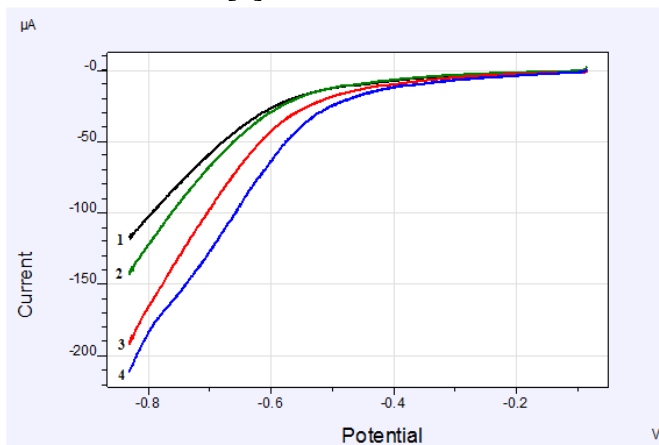**Figure 3. Cathodic polarization curves at different temperatures on the Ni electrode. Electrolyte (M): NiSO$_4$ – 0.107, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7. E$_\upsilon$ = 20 mV/s, T (K): 1 – 298; 2 – 308; 3 – 323; 4 – 333.**

The nature of the polarization to determine a lgi$_k$ - 1/T dependence was constructed based on the polarization curves in Fig. 3. The adequate activation energy for the electroreduction reaction of nickel ions was calculated based on the lgi$_k$ - 1/T dependence. The electrochemical precipitation of nickel ions from alkaline electrolytes is accompanied by electrochemical polarization (41 kC/mol) at potential values of -0.6 ÷ -0.725 V. At the values of -0.725 ÷ -0.8 V of the potential, the process gradually changes to solid polarization. The effect of the concentration of molybdate ions on the electroreduction process was studied by recording potentiodynamic polarization curves (21.6 kC/mol).

Figure 4 shows the cathodic polarization curves of the electrochemical reduction of molybdate ions at various concentrations of sodium molybdate (in the range of 0.05 ÷ 0.8 M). A "plateau" corresponding to the formation of a MoO$_2$ layer on the cathode is observed at all concentrations with an electrode potential in the range of -0.26 ÷ -0.28 V. At potential values of -0.74 ÷ -0.76 V, a peak is observed in the polarization curves, corresponding to the reduction of molybdenum dioxide (MoO$_2$) to molybdenum. At potential values of -

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0.91÷-0.92 V, the evolution of hydrogen accelerates, which compli-cates the electrodeposition of Mo on the cathode surface.
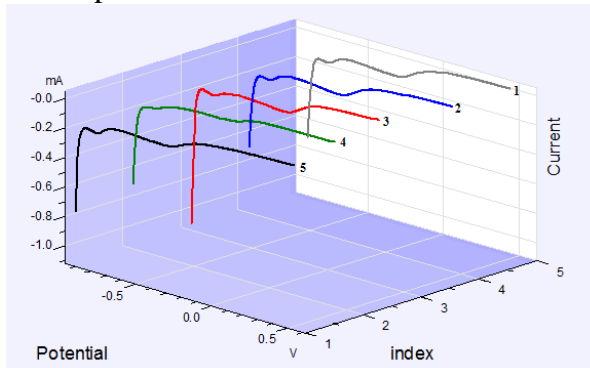

Figure 4. Voltammetry curves of electroreduction of molybdate ions at different concentrations in alkaline electrolyte on Pt electrode. Electrolyte (M): Na_2MoO_4: 1 – 0.05; 2 – 0.1; 3 – 0.2; 4 – 0.4; 5 – 0.8, H_3BO_3 – 0.1, NH_4OH – 7, E_υ = 40 mV/s, pH = 11.2, T = 298 K.

In Figure 5, the peak layer of molybdenum in the anodic part of the curve is oxidized at a potential of -0.86 V. The deposit of molybdenum dioxide (MoO_2) on the surface of the electrode is oxidized at a potential of -0.15 V. A large number of hydrogen ions are involved in cathodic reactions; the potentials of these reactions depend on the pH of the solution.
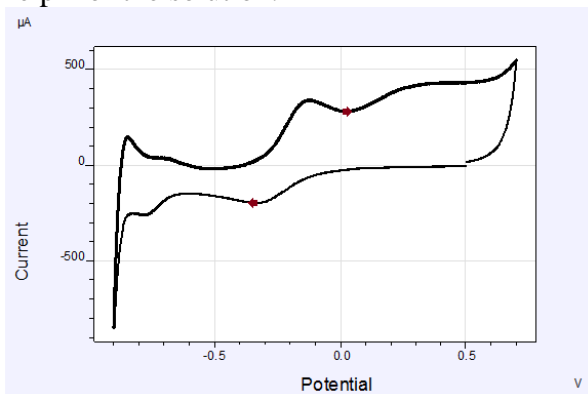

Figure 5. Cyclic voltammetry curve of molybdate ions in alkaline electrolyte on Pt electrode. Electrolyte (M): Na_2MoO_4 – 0.124, NH_4OH – 7, H_3BO_3 – 0.1. E_υ = 80 mV/s, T = 298 K.

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Figure 6 shows the polarization curves of the temperature dependence of the reduction of molybdate ions. The study was conducted in the $298 \div 333$ K [9]. It is clear from the curves that an increase in the electrolyte temperature leads to an increase in the height of the "plateau," corresponding to molybdenum dioxide (MoO_2) formation.
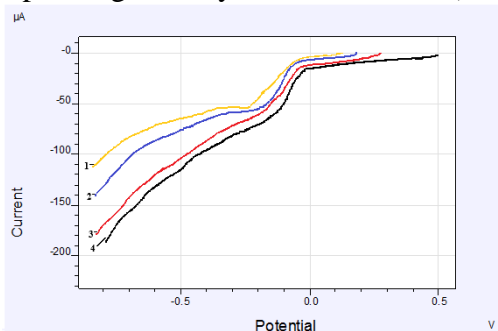
Şəkil 6. Pt elektrodda müxtəlif temperaturlarda katod polyarizasiya əyriləri. Elektrolit (M): Na_2MoO_4 – 0.124, H_3BO_3 – 0.1, NH_4OH – 7. E_ν =20 mV/s, T (K): 1 – 298; 2 – 308; 3 – 323; 4 – 333.

To clarify the nature of polarization based on the polarization curves presented in Figure 6, the dependence $\lg i_k$ -1/T was constructed. The slope of these curves gradually increases as the value of the cathode potential increases.

The adequate activation energy (17.2-20.8 kC/mol) for the electroreduction reaction of molybdate ions was calculated based on the data obtained from $\lg i_k$ - 1/T. The calculated activation energy shows that the diffusion of molybdenum ions controls the reaction rate.

Figure 7 shows the polarization curves of the electrochemical co-deposition of Ni, Mo, and Ni-Mo from an alkaline electrolyte on a platinum electrode.

$NiSO_4$, Na_2MoO_4, $NiCl_2$, H_3BO_3, and NH_4OH concentrations were stable in all electrolytes. It can be seen from Figure 7 that the co-precipitation of nickel with molybdenum by the electrochemical method occurs at values very close to the potential of both nickel and molybdenum deposition. The analysis of the polarization curves shows that the cathode polarization curve of the co-electrodeposition of nickel-molybdenum alloy (curve 3) is between the polarization

curves of nickel (curve 1) and molybdenum (curve 2) deposition. In this case, the cathode polarization curve of the co-precipitation process shows that a solid solution consisting of alloys is formed between two components (nickel and molybdenum). The presence of both metals in the deposited film was confirmed by the chemical analysis results and by data obtained using a scanning electron microscope (SEM).
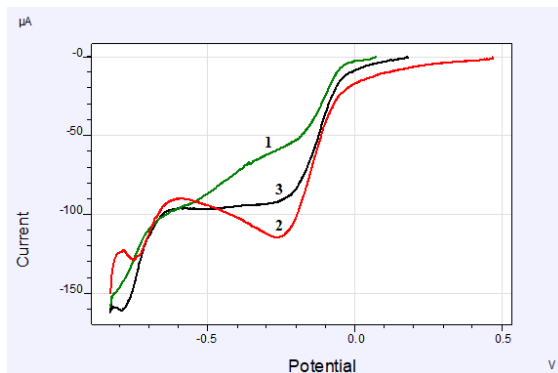

Figure 7. Potentiodynamic polarization curves of Ni^{2+} (1), Mo^{6+} (2) and co-electroreduction of Ni^{2+} and Mo^{6+} (3) from an alkaline electrolyte on a Pt electrode (M):
1) $NiSO_4 – 0.107$, $NiCl_2 – 0.13$, $H_3BO_3 – 0.1$, $NH_4OH – 7$;
2) $Na_2MoO_4 – 0.124$, $H_3BO_3 – 0.1$, $NH_4OH – 7$;
3) $NiSO_4 – 0.107$, $Na_2MoO_4 – 0.124$, $NiCl_2 – 0.13$, $H_3BO_3 – 0.1$, $NH_4OH – 7$. $E_v = 40$ mV/s, pH = 11.2, T = 298 K.

Figure 8 shows the cyclic polarization curve of nickel ions co-electrodeposition with molybdate ions. It can be seen from the picture that the polarization curve of the co-electrodeposition of nickel with molybdenum is divided into two main sections: The first section corresponds to the co-deposition of nickel with molybdenum, which occurs at a potential of -0.28 V. When the potential reaches -0.8÷-0.9 V, simultaneously with the deposition of the sediment, the evolution of hydrogen occurs, which occurs in stages, as indicated by the peaks on the cathode polarization curve at potentials of -0.81 V and -0.87 V. Two plateaus appear on the anodic curve at potentials of -0.11 V and +0.22 V, the first corresponds to the anodic dissolution of the deposited film, and the se-

cond at a potential of +0.22 V is due to the dissolution of nickel, which is part of the deposit as a separate phase.
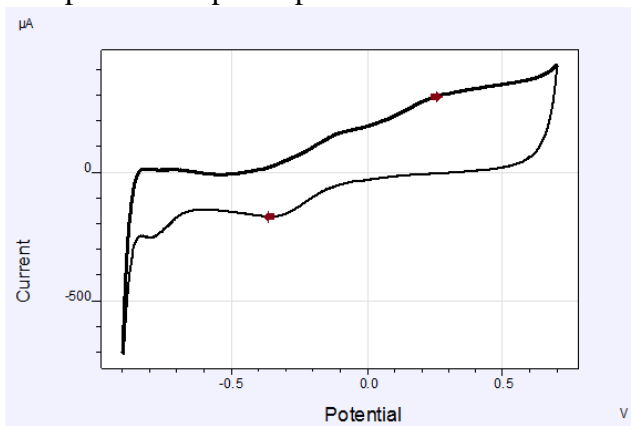

Figure 8. Cyclic polarization curve of co-electrodeposition of nickel with molybdenum in alkaline electrolyte on Pt electrode. Electrolyte (M): NiSO$_4$ – 0.107, Na$_2$MoO$_4$ – 0.124, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7. E$_v$ = 80 mV/s, pH = 11.2, T = 298 K.

In order to accurately determine the processes occurring on the surface of the cathode during the co-deposition of nickel and molybdenum by electrochemical method, cathodic polarization curves were taken to potentials of -0.28 V and -0.87 V. The process was stopped, and the anodic curve was taken after 5 minutes (figure 9).
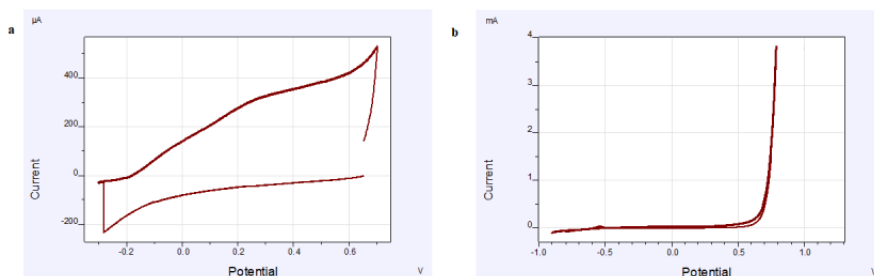

Figure 9. Anodic polarization curves obtained after keeping the Pt electrode at a potential of (a) -0.28 V, (b) -0.87 V for 5 minutes. Electrolyte (M): NiSO$_4$ – 0.107, Na$_2$MoO$_4$ – 0.124, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7. E$_v$ = 100 mV/s, pH = 11.2, T = 298 K.

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Figure 9 (a) shows that a "plateau" is formed on the anodic curve at a potential of +0.26 V, corresponding to the dissolution of a thin layer of molybdate ion, partially reduced by nickel. In the cyclic voltammogram shown in Figure 9 (b), it is clear that no peaks are visible on the anodic curve before oxygen evolution occurs at a potential of ~ +0.7 V. Consequently, at potential values of -0.81 V and -0.87 V at the cathode, hydrogen evolution predominantly occurs in two stages.

Figure 10 shows the cathode potentiodynamic polarization curves of nickel and molybdenum co-electrodeposition at different concentrations of molybdate ions and constant concentrations of other ions. An increase in the concentration of sodium molybdate leads to an increase in the peak height corresponding to the formation of an alloy. As a result of the co-deposition of elements, the alloy is formed in the potential field of -0.65 ÷ -0.8 V [11].
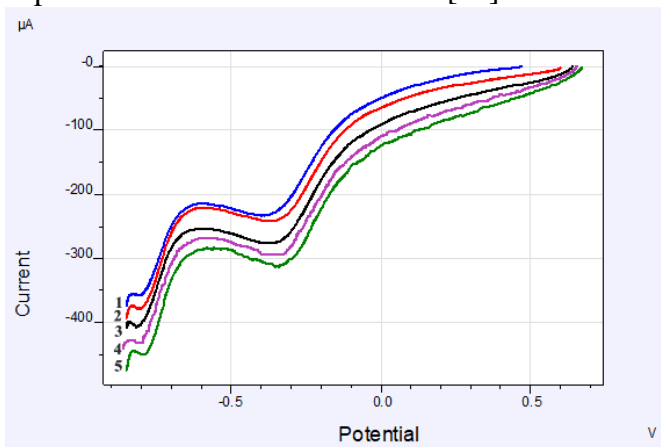

Figure 10. Cathodic polarization curves of nickel co-electrodeposition with molybdenum at different concentrations of molybdate ions. Electrolyte (M): NiSO$_4$ – 0.08, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7, Na$_2$MoO$_4$: 1 – 0.057; 2 – 0.069; 3 – 0.092; 4 – 0.124; 5 – 0.138. E$_v$ = 100 mV/s, pH = 11.2, T = 298 K.

Co-electrodeposition of molybdenum with nickel occurs in two stages. In the first stage (slow), molybdenum dioxide (MoO$_2$) is converted into mixed oxide (NiMoO$_2$) by the following reaction:

$$MoO_2 + Ni^{2+} + 2e^- = NiMoO_2 \qquad (3)$$

The rate of this reaction is low; a compound is formed on the surface of the electrode, leading to the release of hydrogen. The mixed oxide is then electrochemically reduced in the presence of nickel ions to form a Ni_3Mo alloy:

$$MoO_4^{2-} + 3Ni^{2+} + 8e^- + (4+n)H = Ni_3Mo + 4OH^- + nH_{ads} \qquad (4)$$
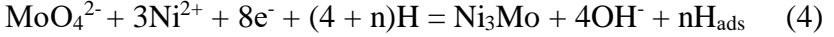
Since the rate of reaction (3) is lower than the rate of reaction (4), it may be the main reaction in the co-electrodeposition of nickel with molybdenum to form the Ni_3Mo alloy.

At the initial stage of obtaining thin layers, crystalline centers are formed. Around them, there is a competitive growth of crystallites (crystalline grains) of different directions and irregular shapes, leading to a polycrystalline structure.It is possible to learn more detailed information about the study of nucleation and growth mechanisms for electrochemical deposits detected from polarization measurements by chronoamperometric method (Fig. 11). The curves are at room temperature, as shown in figure 14, -0.1; -0.2; -0.3; -0.4; -0.5; -0.6; -0.7; -0.8; -0.9 V was studied at different potentials.
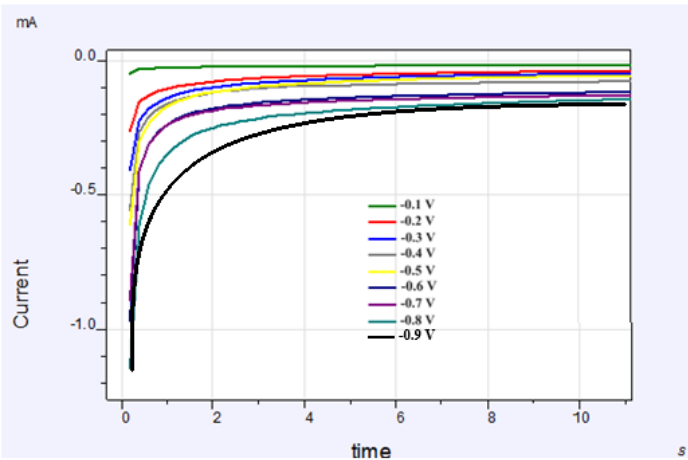

Figure 11. Chronoampermetric curves of co-deposition process of Ni and Mo in alkaline electrolyte on Pt electrode. Electrolyte (M): NiSO₄ – 0.107, Na₂MoO₄ – 0.124, NiCl₂ – 0.13, H₃BO₃ – 0.1, NH₄OH – 7. pH = 11.2, T = 298 K.

According to the graph, the early mode of the current-time curve is characterized by a sharp current shift caused by applying a constant potential. Figure 12 (a-i) shows the growth processes of Ni - Mo crystals. At the initial stage, according to experimental data, the nucleation formed in many active areas of the electrode surface at potentials of -0.1 and -0.2 V corresponds to the curve of the developing nucleation model of Ni-Mo. However, the deposition changes to instantaneous nucleation and growth for other potentials (from -0.3 to - 0.9 V), as shown in Figure 12 (c–i).
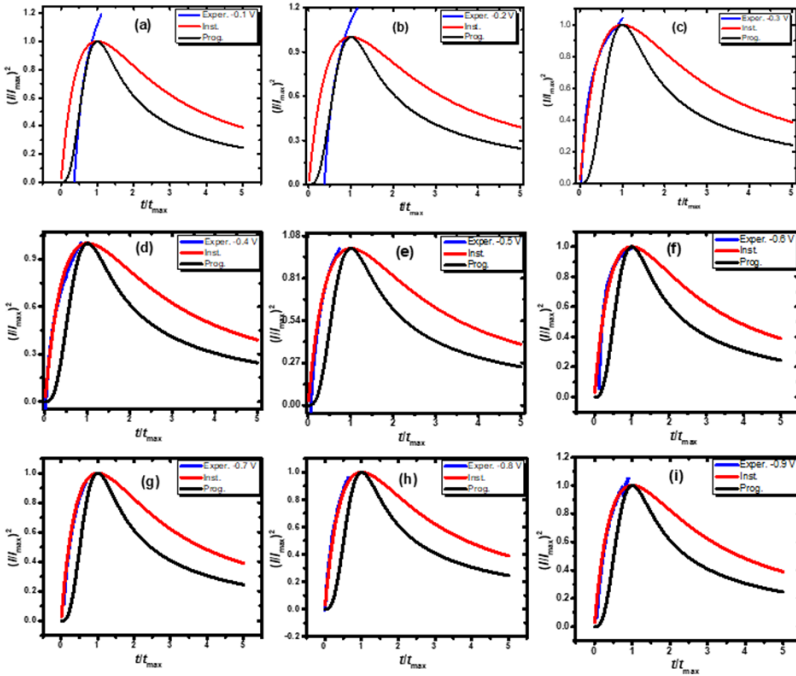

Figure 12. Plots of theoretical comparison of the dimensionless fields of $(I/I_{max})^2$ and (t/t_{max}) of instantaneous and ascending nucleation based on experimental data obtained from chronoamperometric co-deposition curves.

Additional information about the crystal growth mechanism can be obtained by calculating the active particles node density (N_0). The growth mechanism can also be obtained by determining the dif-

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fusion coefficient D using the chronoamperometric method. According to the instantaneous nucleation model, the diffusion coefficient D is related to Imax and tmax.

Table 1 shows the values of D and N_0 at various deposition potentials and temperatures. These results indicate that increasing the deposition potential decreases the diffusion coefficient D values of electroactive nuclei under these conditions, consistent with the Randles-Sevcik equation. The table shows that the density of nuclei N_0 increases sharply with increasing deposition potential.

<div align="right">**Table 1**</div>

Experimental data on chronoampermetric deposition of a nickel-molybdenum alloy

Applied potential, V	D, $sm^2 \cdot s^{-1}$	N_0, sm^{-2}
-0.10	$0.89 \cdot 10^{-10}$	$0.90 \cdot 10^7$
-0.20	$6.12 \cdot 10^{-10}$	$12.96 \cdot 10^7$
-0.30	$4.74 \cdot 10^{-10}$	$16.74 \cdot 10^7$
-0.40	$3.91 \cdot 10^{-10}$	$20.25 \cdot 10^7$
-0.50	$1.00 \cdot 10^{-10}$	$81.02 \cdot 10^7$
-0.60	$1.74 \cdot 10^{-10}$	$45.57 \cdot 10^7$
-0.70	$0.68 \cdot 10^{-10}$	$11.67 \cdot 10^8$
-0.80	$0.48 \cdot 10^{-10}$	$16.53 \cdot 10^8$
-0.90	$0.11 \cdot 10^{-10}$	$72.92 \cdot 10^8$

Research on co-precipitation of nickel with molybdenum by electrochemical method shows that the composition of cathode deposits and their quality (corrosion resistance, relief, hardness, adhesion to the electrode surface) depends on the composition of the electrolyte, the density of the main components in the electrolyte, current density, mixing speed and temperature. In this regard, it is essential to study the influence of the mentioned factors on the composition and quality of the deposited layers. Figure 13 shows the dependence of the composition of thin films on a platinum electrode at a temperature of 298 K on the current density. The studies were carried out in the sodium molybdate concentration range of $0.05 \div 0.13$ M.

Figure 13 shows that with increasing current density it leads to an increase in the molybdenum content in a thin film [8]. The adhesion of Ni-Mo thin films to the substrate is high, but at current densities above 2.5 A/dm^2, the adhesion of the layer to the substrate decreases and part of the resulting Ni-Mo alloy settles to the bottom of the electrolyzer. High-quality Ni-Mo thin films with high catalytic activity and satisfactory adhesion to the substrate were obtained at an optimal current density of 1.0 - 2.5 A/dm^2.
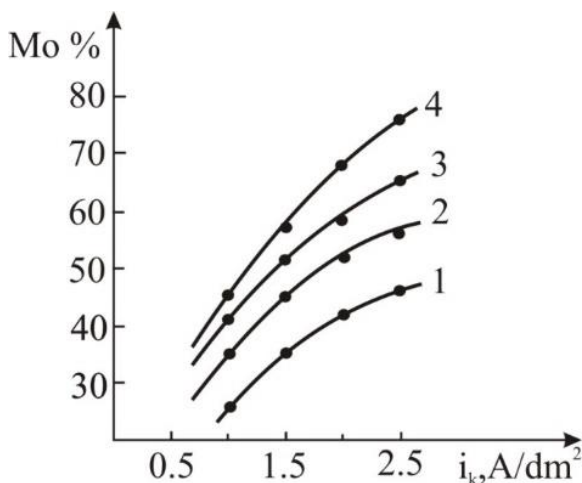

Figure 13. Dependence of the composition of Ni - Mo thin layers on the Pt electrode on the current density at different concentrations of molybdate ions. Electrolyte (M): NiSO$_4$ – 0.107, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7, Na$_2$MoO$_4$: 1 – 0.05; 2 – 0.08; 3 – 0.124; 4 – 0.13. T = 298 K.

Figure 14 shows the dependence of the composition of Ni - Mo thin layers on the Ni electrode at different current densities on the concentration of molybdenum. It has been shown that the amount of molybdenum in the alloy is up to 80% with the increase in the concentration of molybdate ions in the electrolyte. Increasing the concentration of molybdate ions from 0.03 M to 0.124 M at a current density of 1.5 A/dm^2 leads to an increase in the amount of molybdenum in the thin layer from 35% to 68%.
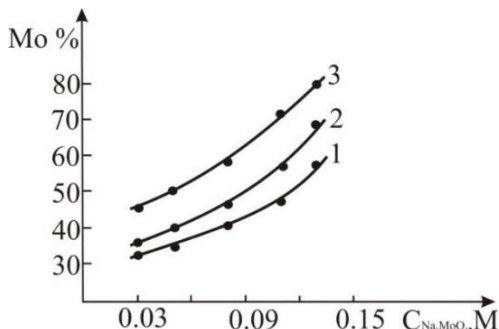
Figure 14. Dependence of the composition of Ni - Mo thin layers in the electrolyte on the concentration of molybdate ions, cathode - Ni, anode - Pt. Electrolyte (M): NiSO$_4$ – 0.107, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7. pH = 11.2, T = 298 K, conditions of electrolysis i$_k$ (A/dm^2): 1 – 1.0, 2 – 1.5, 3 – 2.5.

Figure 15 shows the effect of the concentration of nickel ions on the composition of thin layers. An increase in the concentration of nickel ions in the electrolyte leads to a decrease in molybdenum in the thin layer. Increasing the concentration of nickel sulfate from 0.02 M to 0.06 M reduces the amount of molybdenum in the thin film by 10% at a current density of 2.5 A/dm^2. Increasing the concentration of nickel sulfate from 0.06 M to 0.14 M almost does not change the amount of molybdenum in the thin layer.
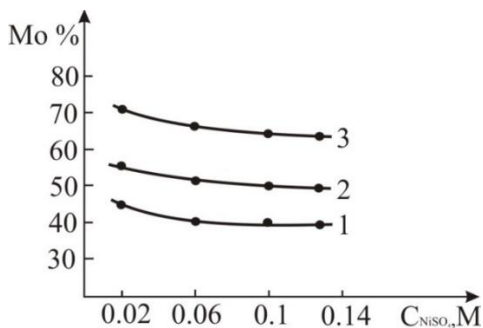

Figure 15. The influence of the concentration of nickel ions in the electrolyte on the composition of the Ni - Mo thin layer, cathode - Ni, anode - Pt. Electrolyte (M): Na$_2$MoO$_4$ – 0.124, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7. pH = 11.2, T = 298 K, conditions of electrolysis i$_k$ (A/dm^2): 1 – 1.0, 2 – 1.5, 3 – 2.5.

Electrochemical processes involve many variables that affect the composition and properties of the resulting deposits, and to select the optimal electrolysis conditions and electrolyte composition, it is necessary to carry out many experiments. These problems are easier to solve using mathematical modeling. Thus, by performing a minimal number of electrochemical deposition processes, it is possible to construct a mathematical model from the results obtained by determining the influence of various factors on the final product, i.e., the composition of the deposited alloy. A regression equation for the process under study has been compiled and has the following form. [12]:

$$\hat{y} = 58.1 + 143.1Z_1 - 337Z_2 + 10.6Z_3 - 4423Z_1Z_2 - 25Z_1Z_3 - 24Z_2Z_3 \quad (5)$$

As a result of calculations based on the obtained experimental data, a regression equation was obtained that quite fully describes the electrodeposition of nickel with molybdenum. Considering that nickel-molybdenum alloys form several solid solutions widely used in practice, the required composition of alloys obtained by the electrochemical method is determined by this equation. The regression equation shows that the ratio of the concentrations of the main components in the electrolyte has the most significant influence on the composition of the electrodeposited layers.

Summary of the discussion in Chapter Four

A study of the microstructure of Ni-Mo thin films obtained from an alkaline electrolyte shows that the appearance of the films significantly depends on the electrolysis conditions, heat treatment temperature, and duration.

Figure 16 (a) shows a Ni-Mo thin film deposited on the electrode surface at low current densities ($1.0 \div 2.5$ A/dm^2). The resulting thin layer consists of dark gray, almost black, small granular layers with high adhesion. With the increase of the current density (above 2.5 A/dm^2), the adhesion of the layers to the substrate decreased. The color of the alloys turns light gray at high current densities and is illustrated in Figure 16 (b).
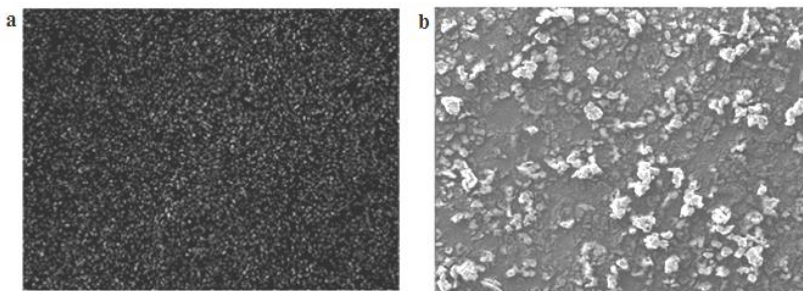
Figure 16. Microstructure of Ni – Mo alloys. Electrolyte (M): NiSO$_4$ – 0.107, Na$_2$MoO$_4$ – 0.124, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7. pH = 11.2, T = 298 K, a) i = 1.5 A/dm^2, b) i = 3.5 A/dm^2.

Figure 17 (a) shows the scanning electron microscope morphology of an unheated-treated thin film deposited electrochemically under optimal conditions. The resulting films were amorphous, the layer was relatively smooth, and its adhesion to the substrate was quite strong. After heat treatment, the alloy particles (Fig. 17 (b)) increased in size, their structure became crystalline, and these samples were subjected to X-ray phase analysis.
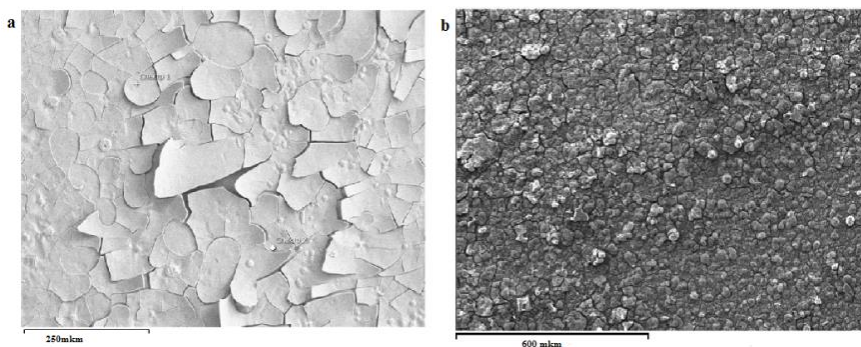

Figure 17. Microstructure of Ni – Mo alloys. Electrolyte (M): NiSO$_4$ – 0.107, Na$_2$MoO$_4$ – 0.124, NiCl$_2$ – 0.13, H$_3$BO$_3$ – 0.1, NH$_4$OH – 7. pH = 11.2, current density 1.0 A/dm^2, T = 298 K, a) before heat treatment, b) after heat treatment (heat treatment temperature: 773 K).

Figure 18 shows the microstructure of Ni-Mo thin films obtained from an alkaline electrolyte under optimal conditions. Ni-Mo

thin layers were heat treated at 773 K for 1 hour in an argon atmosphere. The figure shows that the amount of oxygen in the heat-treated alloy in an argon atmosphere is significantly reduced. The catalytic properties of this alloy after heat treatment were studied. Since the results obtained when using these alloys in water electrolysis were unsatisfactory, heat treatment was carried out in a muffle furnace in an air atmosphere.
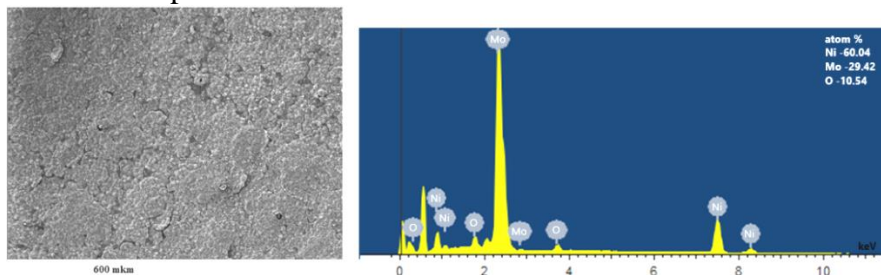

Figure 18. Microstructure of Ni – Mo alloys. Electrolyte (M): NiSO₄ – 0.107, Na₂MoO₄ – 0.124, NiCl₂ – 0.13, H₃BO₃ – 0.1, NH₄OH – 7; pH = 11.2, i = 1.0 A/dm², T = 298 K (heat treatment temperature: 773 K).
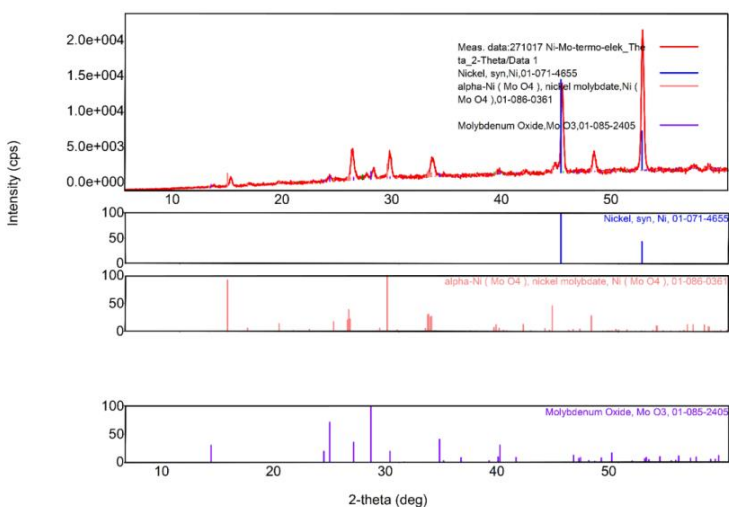
Figure 19. X-ray diffraction pattern of Ni – Mo alloys. Electrolyte (M): NiSO₄ – 0.107, Na₂MoO₄ – 0.124, NiCl₂ – 0.13, H₃BO₃ – 0.1, NH₄OH – 7; pH = 11.2, i = 1.0 A/dm², T = 298 K (heat treatment temperature: 773 K).

Nickel-molybdenum alloys were subjected to heat treatment at a temperature of 773 K for 1 hour in an air atmosphere, and the composition of the alloys was studied by X-ray phase analysis.

Figure 22 shows an X-ray diffraction analysis of a thin film containing 68.2% Ni, 11.5% Mo, and 20.3% O. The electrodeposited layer was initially amorphous and had a composition of 73.5% Ni, 13.3% Mo, and 13.2% O. After heat treatment of thin films in air, the molybdenum and nickel content is reduced, and the oxygen content is increased by 7.1%.

X-ray phase analysis of the resulting thin film after heat treatment was carried out. The analysis showed that after heat treatment, the structure of the thin film changed, and the amorphous films became polycrystalline. As mentioned above, co-precipitation of nickel with molybdenum probably occurs through the formation stage of these metals' oxides. As a result, a solid solution is formed. After thermal treatment of the layers, a solid-phase reaction occurs between the oxides of these metals, resulting in the formation of $NiMoO_4$ appearing on the diffraction pattern. In addition, the MoO_3 and Ni phases were detected in the diffraction pattern. Most likely, the MoO_3 phase is formed due to the oxidation of free molybdenum or low-valent molybdenum due to annealing under atmospheric conditions. Thus, due to heat treatment, thin layers of the alloy with an amorphous structure switched to a polycrystalline structure; X-ray phase analysis revealed the presence of three phases in the layers - Ni, MoO_3, and $NiMoO_4$.

Figure 20 shows the catalytic activity of water electrolysis in 0.5 M Na_2SO_4 electrolyte for various electrodes (cathode). Pt, Ni, and Steel-3 electrodes were also used to characterize the catalytic activity of nickel-molybdenum alloys synthesized during the research. The figure shows that the highest catalytic activity was observed on thin Ni-Mo films containing 73.5% Ni, 13.3% Mo, and 13.2% O and not subjected to heat treatment. The catalytic activity of these layers is ~1.5 times higher than the catalytic activity of the platinum electrode.
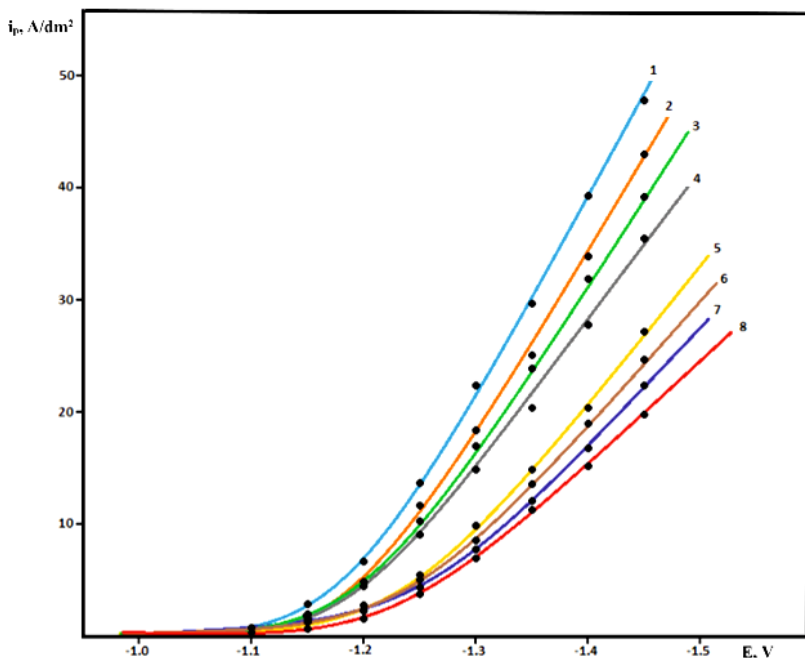
Figure 20. Cathodic polarization curves at different electrodes in 0.5 M Na_2SO_4 electrolyte: 1 – $Ni_{73.5}Mo_{13.3}O_{13.2}$ (Ni substrate without heat treatment); 2 – $Ni_{83.4}Mo_{2.1}O_{14.5}$ (on a Pt substrate); 3 – $Ni_{68.2}Mo_{11.5}O_{20.3}$ (after heat treatment on a Ni substrate); 4 – Ni; 5 – Pt; 6 – $Ni_{70.12}Mo_{14.59}O_{15.29}$ (Steel-3 substrate), 7 – Polad–3, 8 – $Ni_{13.79}Mo_{64.85}O_{21.36}$ (Ni substrate).

The catalytic activity of Ni - Mo thin films was studied depending on their composition in a neutral environment (0.5 M Na_2SO_4). The data obtained are presented in Table 2. The lowest slope value was observed for thin films not subjected to annealing with a nickel content of 73.5% (Fig. 23 - curve 1). After heat treatment, the Ni content in the sediment decreased to 68.2%, and the slope of the Tafel curve increased (Fig. 23 - curve 3).

Figure 21 shows the catalytic activity of various electrodes as a cathode in the electrolysis of water in 3.0 M NaOH.

Table 2

The values of the Tafel slope of the polarization curves for various electrodes during the electrolysis in the cathode process of water in a neutral environment

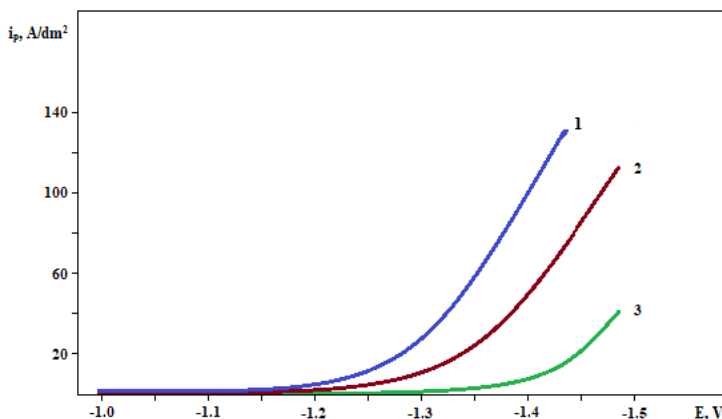| №	Selected substrates electrode	Elektrode	$\partial E/\partial lgi$ (mV)
1.	Pt	platinum	164
		$Ni_{83.4}Mo_{2.1}O_{14.5}$	110
2.	Ni	nickel	136
		not subject to heat treatment $Ni_{73.5}Mo_{13.3}O_{13.2}$	105
		after heat treatment $Ni_{68.2}Mo_{11.5}O_{20.3}$	123
		$Ni_{13.79}Mo_{64.85}O_{21.36}$	220
3.	Steel grade Steel - 3	Steel - 3	190
		$Ni_{70.12}Mo_{14.59}O_{15.29}$	183

Figure 21. Cathodic polarization curves at different electrodes in 3.0 M NaOH electrolyte: 1 – Pt; 2 – Ni$_{73.5}$Mo$_{13.3}$O$_{13.2}$ (without heat treatment on Ni substrate); 3 – Ni.

The figure shows that the platinum electrode has the most excellent catalytic activity as a cathode in an alkaline electrolyte. The

catalytic activity of Ni-Mo alloy in alkaline electrolyte is lower than that of Pt electrode and higher than that of pure Ni electrode.

The value of Tafel slope was calculated from E - lgi curves, that is, $\partial E/\partial lgi$ (mV) was calculated for the electrodes made of Pt, Ni, and $Ni_{73.5}Mo_{13.3}O_{13.2}$ alloy, respectively, the catalytic activity in the cathode. It was determined that the Tafel tendency of the Pt electrode in 3.0 M NaOH electrolyte is 70 mV, the Tafel slope of the $Ni_{73.5}Mo_{13.3}O_{13.2}$ alloy obtained on a Ni substrate and not subjected to heat treatment is 90 mV, and the Tafel slope of the Ni electrode is 110 mV.

Figure 22 shows the catalytic activity of different electrodes in the electrolysis process of water in 0.5 M Na_2SO_4 electrolyte as anode. Potentiodynamic polarization curves were drawn to determine the catalytic activity of the electrodes in the oxygen evolution reaction. Pt electrode was used as the cathode, and Ni, Pt, Steel-3, and Ni - Mo alloys (heat treated and not) were used as the anode.
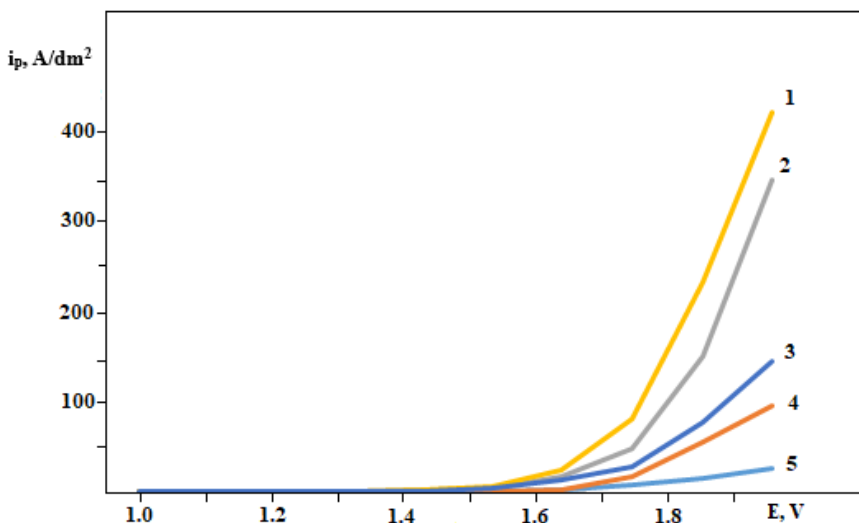

Figure 25. Anodic polarization curves at different electrodes in 0.5 M Na_2SO_4 electrolyte: 1 – Pt; 2 – $Ni_{73.5}Mo_{13.3}O_{13.2}$; 3 – $Ni_{68.2}Mo_{11.5}O_{20.3}$; 4 – Ni; 5 – Steel-3.

Table 3

The values of the Tafel slope of the polarization curves for various electrodes during the electrolysis in the anode process of water in a neutral environment

№	Selected substrates electrode	Elektrode	$\partial E/\partial lgi$ (mV)
1.	Pt	platinum	160
2.	Ni	nickel	230
		not subject to heat treatment $Ni_{73.5}Mo_{13.3}O_{13.2}$	170
		after heat treatment $Ni_{68.2}Mo_{11.5}O_{20.3}$	210
3.	Steel grade Steel - 3	Steel - 3	280

From the data given in Table 3, it can be concluded that platinum is the best anode in the oxygen evolution reaction, followed by $Ni_{73.5}Mo_{13.3}O_{13.2}$ alloy with a small difference, showing high catalytic activity.

Figure 23 shows the catalytic activities of water electrolysis in 3.0 M NaOH electrolyte as anode for different electrodes.
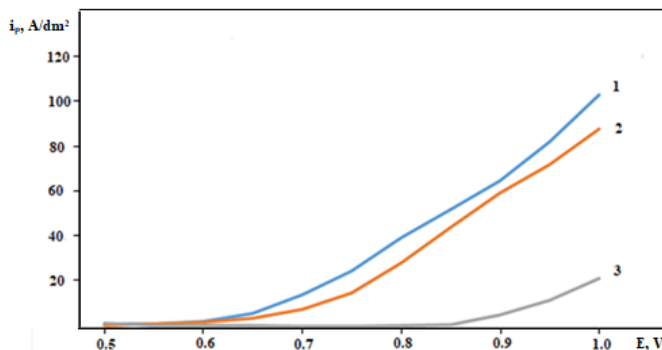

Figure 23. Anodic polarization curves at different electrodes in 3.0 M NaOH electrolyte: 1 – $Ni_{73.5}Mo_{13.3}O_{13.2}$; 2 – Ni; 3 – Pt.

From the above polarization curves, it is clear that the nickel-molybdenum alloy has the best catalytic activity. Hence, the tgα value, calculated from the E-lgi curves, is 70 mV. The Tafel slope of the nickel electrode is 100 mV, and the platinum electrode is 157 mV.

Thus, based on the above, it has been established that Ni-Mo thin films of the $Ni_{73.5}Mo_{13.3}O_{13.2}$ composition synthesized by the electrochemical method and not subjected to heat treatment exhibit high catalytic activity in a neutral environment as a cathode and in an alkaline environment as an anode.

The microhardness of electrochemically synthesized Ni-Mo thin films was determined using a PMT-3 microhardness tester. Table 4 shows the dependence of the microhardness of Ni - Mo thin films on the heat treatment temperature of the alloys. The table shows that the alloys subjected to heat treatment at 773 K for 1 hour have the highest microhardness value, equal to 1120.8 MPa. It should be noted that the microhardness of the alloys obtained under the same conditions but not subjected to heat treatment was 934.2 MPa. From the data obtained, we can conclude that the microhardness of alloys depends not only on the composition of thin layers but also on the heat treatment temperature; the microhardness of thin films increases with increasing annealing temperature, which is associated with a change in the crystal structure under the influence of temperature.

Table 4

Dependence of the microhardness of the nickel-molybdenum layer on the heat treatment temperature

t^0C heat treatment	Microhardness MPa
–	934.2
100	985.3
200	1050.5
500	1120.8

It is known that electrodes used for the electrolysis of water and having high electrocatalytic properties must be cheap and resis- tant to corrosion. With prolonged use, even the surface of platinum group metals becomes poisoned and loses its activity. In this regard,

the corrosion resistance of two samples of nickel-molybdenum alloys of different compositions, $Ni_{13.79}Mo_{64.85}O_{21.36}$ and $Ni_{73.5}Mo_{13.3}O_{13.2}$, was studied. Corrosion resistance was studied in a 0.5 M Na_2SO_4 solution for 50 hours. The surface of the nickel cathode is $2 \cdot 10^{-2}$ dm^2; the mass of the sample was determined before and after the experiment. Based on the difference in sample weight before and after testing, the corrosion coefficients were calculated for the alloys $Ni_{73.5}Mo_{13.3}O_{13.2}$ and $Ni_{13.79}Mo_{64.85}O_{21.36}$, the values of which were 0.0005 $g/m^2 \cdot h$ and 0.0003 $g/m^2 \cdot h$ Accordingly, that is, the corrosion resistance of thin films with an increase in the mass fraction of molybdenum in them was higher than in films with a low molybdenum content.

MAIN RESULTS

1. *For the first time,* thin films of nickel-molybdenum were electrochemically deposited from an alkaline ammonia electrolyte at pH 11.2 (Electrolyte composition: 0.107 M $NiSO_4$, 0.124 M Na_2MoO_4, 0.13 M $NiCl_2$, 0.1 M H_3BO_3, 7 M NH_4OH; electrolysis conditions: current density 1.0 - 2.5 A/dm^2, electrolyte temperature 298 K, electrolyte pH 11.2). As a result of numerous studies, the presented electrolyte composition and electrolysis conditions were patented. Patent Az.No.I 2020065.

2. The process of deposition of nickel and molybdenum from an alkaline electrolyte was carried out separately and the nature of polarization was determined. The deposition of nickel ions from the ammonia electrolyte on a platinum electrode occurred in the potential range $-0.65 \div -0.78$ V, and at the beginning of the process, the deposition of nickel was accompanied by electrochemical polarization, then mixed polarization. The process of reduction of molybdate ions occurs in two stages: the MoO_4^{2-} ion is electrically reduced to MoO_2 in the potential range $-0.26 \div -0.28$ V, and in the second stage to molybdenum in the potential range $-0.74 \div -0.76$ V. The process of electroreduction of molybdate ions is accompanied by concentration polarization.

3. It has been established that co-electrodeposition of nickel and molybdate ions on a platinum electrode in an alkaline electrolyte

occurs at a potential of -0.28 V. The polarization curve of the process of co-electrodeposition is located between the polarization curves of the process of electroreduction of nickel and molybdate ions, which indicates the formation of a solid solution.

4. The effect of various factors on the composition and quality of Ni-Mo thin films - the concentration of the main components in the electrolyte, electrolysis conditions - current density, temperature, mixing speed and pH of the solution was studied. The increase of the current density in the electrode (1.0-2.5 A/dm^2), the concentration of molybdate ions in the electrolyte (0.05 ÷ 0.13 M) is accompanied by an increase in the content of molybdenum in the deposited layers (31÷52%).

5. The electrochemically obtained Ni-Mo thin films were amorphous, but after heat treatment at 773 K for 1 hour in air atmosphere, the films turned into polycrystalline form and clear lines of $NiMoO_4$, MoO_3 and Ni were visible in the X-ray image. It was determined that in the intermediate stage of co-precipitation of nickel with molybdenum, the oxides of these two metals are formed on the cathode surface. Three phases ($NiMoO_4$, MoO_3, Ni) are formed as a result of the solid-phase reaction of sulfur oxides at a temperature of 773 K in the air atmosphere.

6. A regression equation expressing the dependence of the composition of the thin layer obtained in the electrochemical co-deposition process on the composition of the electrolyte and the conditions of electrolysis was established:

$$\hat{y} = 58.1 + 143.1Z_1 - 337Z_2 + 10.6Z_3 - 4423Z_1Z_2 - 25Z_1Z_3 - 24Z_2Z_3$$

7. The microhardness of electrochemically deposited thin films was investigated depending on their composition and heat treatment temperature. It was found that increasing the amount of molybdenum in thin films and the thermal treatment temperature increased the microhardness of Ni-Mo thin films. The corrosion resistance of the studied layers in neutral (0.5 M Na_2SO_4) electrolytes increases with the increase of molybdenum in their content, but this leads to a decrease in their electrocatalytic activity. The corrosion

coefficient for the $Ni_{73.5}Mo_{13.3}O_{13.2}$ alloy not subjected to heat treatment was $5 \cdot 10^{-4}$ g/m^2·h when tested for 50 hours. By drawing polarization curves in neutral and alkaline electrolytes, the catalytic activity of Ni-Mo thin films with different compositions was studied, and a comparative evaluation of the catalytic activity of these electrodes with other electrodes such as cathode and anode in the water electrolysis process was carried out. It was determined that $Ni_{73.5}Mo_{13.3}O_{13.2}$ thin particles are a neutral medium in the electrolysis process of water. The Tafel slope of the cathode is 105 mV, and the platinum electrode is 160 mV. In the alkaline environment, the Tafel slope of these electrodes synthesized as anode is 70 mV, while the platinum electrode is 100 mV.

The main results of the dissertation has been published in the following scientific works:

1. Курбанова У.М., Гусейнова Р.Г., Тагиев Д.Б. Электрохимический синтез нанопленок Ni–Mo // Международная научно-практическая конференция и школа молодых ученых. «Химия, химические технологии и экология: наука, производство, образование». Сб.Тезисов, – Махачкала: – Октябрь, – 2018, – с.156

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8. Gurbanova, U.M. Electrodeposition of Ni–Mo alloys from ammonium electrolytes / U.M.Gurbanova, R.G.Huseynova, H.M.Tahirli [et al.] // Azerbaijan Chemical Journal, – 2019. (3), – p. 25–31.

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The defense will be held on ___07___ _December_ 2023 at ___10^{00}___ at the meeting of the Dissertation council BED 1.15 on specialties 2316.01 – "Chemical kinetics and catalysis" and 2308.01 – "Electrochemistry" established on the basis of ED 1.15 Dissertation Council of the Supreme Attestation Commission under the President of the Republic of Azerbaijan under the Institute of Catalysis and Inorganic Chemistry named after acad. M.Nagiyev of Ministry of Science and Education of the Republic of Azerbaijan.

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The dissertation is available in the library of the Institute of Catalysis and Inorganic Chemistry named after acad. M.Nagiyev of Ministry of Science and Education of the Republic of Azerbaijan.

Electronic versions of dissertation and its abstract are accessible on the official website of the Institute of Catalysis and Inorganic Chemistry named after acad. M.Nagiyev of Ministry of Science and Education of the Republic of Azerbaijan.

Abstract was sent to the required addresses on "_27_" _October_ 2023