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CHRONOAMPEROMETRIC AND CHRONOPOTENTIOMETRIC INVESTIGATIONS OF Ni-Mo CO-DEPOSITION

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Abstract: This work presents studies to study the nucleation and growth mechanism of electrochemical Ni-Mo films suitable for the decomposition reaction of water as an electrocatalyst, in which the value of the potential established from polarization measurements was maintained constant. Optimal conditions for the electrochemical synthesis of the Ni-Mo alloy on the surface of platinum and nickel electrodes were established using voltammetric studies examining the influence of many electrolysis parameters on the process of co-deposition of nickel with molybdenum. The chronoamperometric method provides more detailed information about the electrodeposition process by studying the nucleation and growth mechanism of electrochemical films in which the potential value determined from polarization measurements was kept constant. The curves were taken at various potential values -0.1, -0.2, -0.3, -0.4, -0.5, -0.6, -0.7, -0.8 and -0.9 V at room temperature. The study of these curves can be carried out by comparing the experimental calculated data with the equations of the Scharifker-Hills model. It was found that in the early stage, at potentials of -0.1 and -0.2 V, the experimental data fit into the curve of the progressive nucleation model, according to which Ni-Mo nucleation occurs on many active areas of the substrate surface, but at other potentials (from -0.3 to -0.9 V) deposition deviates towards instantaneous nucleation and growth. Keywords: potentiometry, chronoamperometry, chronopotentiometry, nucleation, nickel, molybdenum. DOI: 10.32737/2221-8688-2024-3-303-311

Introduction

It is known that hydrogen is considered a promising energy carrier to replace fossil fuels as it is versatile and can be used in a wide range of applications such as new energy chemical transportation, industry [1]. Electrolysis of water is one of the most convenient ways to effectively produce highpurity hydrogen [2-4]. Combined with renewable energy sources such as wind and solar power, this provides a smart solution for achieving clean hydrogen production. However, the technology cannot be used on a large scale

due to high energy consumption [5]. To reduce costs and improve the efficiency of hydrogen production by electrolysis of water, it is necessary to develop cathode materials with high electrocatalytic activity and long-term stability to reduce the hydrogen evolution reaction (HER) overvoltage [6]. Nickel and its alloys are the most studied materials as electrodes in the HER reaction [7]. The electrocatalytic activity of nickel is lower than that of steel, but it has excellent corrosion resistance in hot concentrated alkaline solutions

[8]. To increase the catalytic activity of nickel, it is used in the form of alloys. The most convenient way to obtain new electrode materials for the electrolysis of water is the which makes electrochemical method, it possible obtain nanoparticles with to selected purposefully physicochemical properties, provided that the influence of the potential and concentration of metal ions on the morphology of the resulting alloys [9]. Electrodeposition involves the formation of nuclei at active sites and the growth of nuclei through the incorporation of precursor metal ions [10]. Thus, in [11], when preparing catalysts based on transition metals (Ni-P-Se, Fe-P-Se and Ni-Fe-P-Se), the prepared solutions were applied to Ni foam and the resulting Ni-Fe-P-Se electrodes as a cathode material showed better HER performance compared to other materials studied. Previously, we studied the co-precipitation of nickel with molybdenum from an alkaline electrolyte by recording linear and cyclic polarization curves, and determined the influence of such factors on the process of coprecipitation as temperature, the concentration of the main components of the electrolyte, and the rate of potential sweep [12].

Currently, there are several competing models applicable to the case of diffusionlimited three-dimensional growth, in particular the models of Scharifker and Mostani [13], Sluijters-Rebach et al. [14], Heerman and Tarallo [15], and Mirkin and Nilov [16], based on slightly different assumptions. One common feature of all these models is that they use Cottrell diffusion to describe the total flux onto the electrode surface. In research. electrochemical phase transformations have attracted much attention at the early stage of electrodeposition [17,18]. Most authors believe that phase transformations usually occur during three-dimensional nucleation processes. In many cases of electrodeposition, the charge transfer stage is controlled by the mass transfer of electrodepositing ions to the growing centers [19]. It is obvious that the rate of phase transformation and the number of nuclei formed depend on stimulating signals, in this case potentiostatic or galvanostatic. The nucleation and early stages of layer growth are of both fundamental and theoretical interest, and the nucleation and further growth of layers is influenced by the deposition potential and the composition of the electrolyte [20]. The need for models predicting the current transient resulting from nucleation and growth under the control of mixed kinetic diffusion has been raised by several authors [21-23].

When studying the dependence of the nucleation rate on the overvoltage and the concentration of deposited ions, it was found that the density of active nucleation centers depends not on the concentration of ions in the solution, but on the overvoltage [24, 25].

The potential nucleation and growth mechanism was studied using the Scharifker-Hills nucleation model [26]. Analysis of experimental current transients shows that nucleation is in good agreement with instantaneous nucleation and three-dimensional (3D) diffusion-limited growth.

In this work, our goal was to study the nucleation process during the synthesis of Ni-Mo alloys using chronopotentimetric and chronogalvanometric methods.

Experimental part

When synthesizing thin films of nickel with molybdenum, $NiSO_4 \cdot 7H_2O$ from Indian Central Drug House (p) Ltd was used as a source of nickel ions, and $Na_2MoO_4 \cdot 2H_2O$ from Indian company Qualikems Fine Chem Pvt was used as a source of molybdenum ions. Both salts were dissolved in NH₄OH from the Indian company Qualikems Fine Chem Pvt. Ltd., in addition, boric acid (CDH Ltd.) was added to the electrolyte as a buffering additive, and to reduce the formation of metal oxides, NiCl₂ \cdot 6H₂O was introduced into the solution, and the pH of all solutions was 11.2. Chronopotentiometric and chronoamperometric studies were carried out on the IVIUMSTAT interface potentiostat using a classic 100 ml three-electrode cell equipped with a water jacket. A platinum wire with an area of 0.2 cm² and 0.4 cm² was used as a working electrode, and a platinum plate with an area of 4 cm² was used as an auxiliary electrode. A silver chloride electrode (Ag/AgCl) was used as a reference

electrode, and all potentials in the article are given relative to this electrode.

Results and discussion

To clarify the mechanism of the process of joint deposition of two elements, first of all, polarization curves were recorded. It was found that the deposition potential depends on the concentration of the main components of the alloy, pH of the electrolyte, temperature and substrate material. By recording cyclic voltammetric curves, the potential at which coprecipitation of nickel with molybdenum and the mechanism of co-precipitation occurs was determined [7].

The optimal conditions for the electrochemical synthesis of Ni-Mo alloy on the

electrode surface of platinum have been detected using voltammetric studies and from further successive experiments via the variation of many parameters as mentioned above.

The chronoamperometric method provides more details about the electrodeposition process at which the potential was held (detected from CV measurements), such as by studying the nucleation and growth mechanisms for the electrochemical deposits. The curves were observed at different applied potentials of -0.1, -0.2, -0.3, -0.4, -0.5, -0.6, -0.7, -0.8, and -0.9 V at room temperature, as shown in Fig. 1.



Fig. 1. Chronoamperometric plots of the electrochemical synthesis of Ni-Mo alloy on Pt substrates in an electrolyte of 0.107 M NiSO₄ · 6H₂O, 0.13 M NiCl₂ · 6H₂O, 0.124 M Na₂MoO₄ · 2H₂O, 0.1 M H₃BO₃, 7 M NH₄OH, at T = 295 K (22 °C) and pH = 11.2, at different deposition potentials and room temperature; -0.1, -0.2, -0.3, -0.4, -0.5, -0.6, -0.7 and -0.8 V vs. Ag/AgCl.

According to the chart, the early regime of the current-time curve is characterized by an abrupt shift in current caused by applying a constant potential. This is due to the combination of a double layer between the negatively charged surface of substrate material and the ions of the test solution, which results in the instantaneous nucleation of Ni-Mo in all circumstances, as seen in Fig. 1. Following this, the resultant current rises slightly. This is because of an increase in the electroactive area caused by crystal formation on the electrode surface. Furthermore, raising the deposition potential implies a high density throughout this electrochemical deposition (throughout all cases).

Analysis of the resulting current-time curves can reveal the crystal nucleation and growth mechanism using the chronoamperometric approach. Examining these curves can be performed by comparing the experimental calculated data to the equations of the Scharifker-Hills model [26]. According to the proposed model, there are two nucleation processes: immediate and gradual. The term instantaneous nucleation refers to the simultaneous growth of nuclei when the deposition potential is applied, whereas progressive nucleation refers to the formation of nuclei during the electrodeposition process. Equations (1) and (2) describe the theoretical transient models for instantaneous and progressive 3D nucleation, respectively:

$$\left[\frac{I(t)}{I_{max}}\right]^{2} = \frac{1.9542}{\frac{t}{t_{max}}} \left\{ 1 - \exp\left[-1.2564\left[\frac{t}{t_{max}}\right]\right] \right\}^{2}$$
(1)

$$\left[\frac{I(t)}{I_{max}}\right]^{2} = \frac{1.2254}{\frac{t}{t_{max}}} \left\{ 1 - \exp\left[-2.3367 \left[\frac{t}{t_{max}}\right]^{2}\right] \right\}^{2}$$
(2)

where, t_{max} and I_{max} are the maximum value of time in second (s) and the maximum current in Amper (A).

Fig. 2(a–i) shows the non-dimensional $I^2\!/I^2_{\ max}$ vs. t/t_{max} plots of the CA results at

different variant conditions. The solid lines (black and red) are the theoretical transients of the instantaneous and the progressive nuclear growth, respectively, as labeled in the figure, and blue lines for the experimental results.



Fig. 2. Theoretical non-dimensional plots of $(I/I_{max})^2$ vs (t/t_{max}) , for instantaneous and progressive nuclear growth and experimental data derived from the chronoamperometric deposition data.

The crystal growth processes of Ni-Mo under these conditions can be detected in Fig. 2(a-i). At the early stage, for potentials -0.1 and -0.2 V, the experimental data fit the curve of the progressive nucleation model by which the Ni-Mo nucleation occurred on many active sites of the substrate surface. But, for the rest potentials (from -0.3 to -0.9 V), the deposition deviates to the instantaneous nucleation and growth, as shown in Fig. 2(c-i).

The deviation from the ideal assumption of the Scharifker model may be attributed to the that the nuclei grow under diffusion control under these conditions. In fact, through time, the nuclei growth and the electrodeposition of Ni-Mo will be under mixed control (diffusion and charge transfer). Further information on the growth mechanism can be obtained by calculating the density of actives sites (N_0).

Also, the growth mechanism can be obtained by determining the diffusion coefficient D via the chronoamperometric method. According to the instantaneous nucleation model, the D is related to the I_{max} and the t_{max} . Moreover, the deviation can also be

interpreted as due to the hydrogen reduction during nuclei formation or the nucleus's morphology change. Table 1 shows the values of D and N₀ at various deposition potentials and temperatures. These results indicate that raising the deposition potential reduces the D values of electroactive species under the these circumstances, which is consistent with the Randles-Shevchic equation. As seen in Table 1, the polarization potential affects the D value. The table shows that the nuclei density N_0 increases dramatically when the deposition potential rises. This increase with applied deposition potential is often interpreted as higher activation of nucleation sites at higher potentials, which agrees with the traditional nucleation theories, as seen in Fig. 2.

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	$0.11 \cdot 10^{-10}$	$72.92 \cdot 10^8$

Table 1. The experimental data of the CA deposition of Ni-Mo.



Fig. 3. Chronopotentiometric curves of the deposition process show the variation of the electrode potential during the electrodeposition process. The deposition current density was 2 mA/cm².

Chronopotentiometric profiles obtained under the effect of different current densities are

compared in Figure 3. The behavior of potential recorded through the electrodeposition process

from the studied bath exhibit an initial short phase of higher overpotential due to the nucleation of Ni-Mo particles on the substrate surface and the following phase at constant potential due to the growth of Ni-Mo particles after their nucleation. The potential profile obtained during the electrodeposition with the studied bath shows three different sections: a short zone of high potential, a wide potential peak, and a gradual potential decrease. The reduction of Ni and Mo ions may explain the increased potential zone. The following wide peak underlines a strong inhibition at the beginning of the Ni-Mo growth, caused by the adsorption of these ions on the surface of the substrate metal, leading to the formation of micelle-like structures or hydrophobic double layers on the surface of the electrode. The adsorption of ions is due to the coulombic attraction between the studied ion and the electrode. It is focused on high current density areas on the electrode surface, such as crests and peaks. Deposition potential slowly increases in the final part of the deposition; this may be explained by an increase in the electroactive surface area of the electrode.

Conclusion

It was found that during co-deposition of nickel with molybdenum, a solid solution of these two metals is formed, and the deposited alloys are amorphous. Co-deposition passes through the stage of formation of oxides of these two metals. Annealing at 500 °C for one hour makes sample polycrystalline and NiMoO₄ compounds appear in the X-ray diffraction pattern. The diffusion of ions controls the co-

deposition process to the cathodic surface. Knowing the mechanism of co-deposition of nickel with molybdenum will make it possible to select optimal conditions for the deposition of alloys of strictly required composition with good electrocatalytic properties.

The catalytic activity of amorphous Ni-Mo film is higher than film subjected to annealing due to the real large surface of amorphous films.

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NİKELİN MOLİBDENLƏ BİRGƏ ÇÖKDÜRÜLMƏSİ PROSESİNİN XRONOAMPEROMETRİK VƏ XRONOPOTENSİOMETRİK TƏDQİQATLARI

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Xülasə: Bu işdə suyun parçalanması reaksiyası üçün elektrokimyəvi üsulla çökdürülmüş Ni-Mo nazik təbəqələrinin nüvələşmə və böyümə mexanizmi öyrənilmiş və burada polarizasiya əyrilərinin çəkilməsində müəyyən edilmiş potensialın qiyməti sabit saxlanılmışdır. Platin və nikel elektrodlarının səthində Ni-Mo ərintisinin elektrokimyəvi üsulla sintezi üçün optimal şərait, bir çox elektroliz parametrlərinin təsirini araşdıran voltampermetrik tədqiqatlardan istifadə etməklə yaradılmışdır. Xronoamperometrik üsul polarizasiya ölçmələri nəticəsində müəyyən edilmiş potensialın qiymətinin sabit saxlanıldığı elektrokimyəvi təbəqələrin nüvələşmə və böyümə mexanizmini öyrənməklə elektroçökmə prosesi haqqında daha ətraflı məlumat verir. Əyrilər otaq temperaturunda -0.1, -0.2, -0.3, -0.4, -0.5, -0.6, -0.7, -0.8 və -0.9 V müxtəlif potensiallarda götürülmüşdür. Bu əyrilərin eksperimental hesablanmış məlumatları Şarifker-Hills modelinin tənlikləri ilə müqayisə etməklə həyata keçirilmişdir. Müəyyən edilmişdir ki, ilkin mərhələdə -0.1 və -0.2 V potensiallarda eksperimental məlumatlar müvəqəti nüvələşmə modelinin əyrisinə uyğun gəlir, ona görə Ni-Mo nüvələşməsi substratın səthinin bir çox aktiv sahələrində baş verir, lakin digər potensiallarda (-0.3-dən -0.9 V-a qədər) çökmə ani nüvələşmə və böyüməyə uyğun gəlir.

Açar sözləri: potensiometriya, xronoamperometriya, xronopotentiometriya, nüvələşmə, nikel, molibden

ХРОНОАМПЕРОМЕТРИЧЕСКИЕ И ХРОНОПОТЕНЦИОМЕТРИЧЕСКИЕ ИССЛЕДОВАНИЯ ПРОЦЕССА СОВМЕСТНОГО ОСАЖДЕНИЯ НИКЕЛЯ С МОЛИБДЕНОМ

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Резюме: В данной работе приведены исследования по изучению зародышеобразования и механизма роста электрохимических пленок Ni-Mo, пригодных для реакции разложения воды в качестве электрокатализатора, при котором значение потенциала, установленного из измерений, поддерживался постоянным. Оптимальные поляризационных **VCЛОВИЯ** электрохимического синтеза сплава Ni-Mo на поверхности платинового и никелевого электродов были установлены с помощью вольтамперометрических исследований с изучением влияния многих параметров электролиза на процесс совместного осаждения никеля с молибденом. Хроноамперометрический метод дает более подробную информацию о процессе электроосаждения путем изучения зародышеобразования и механизма роста электрохимических пленок, при котором значение потенциала, установленного из поляризационных измерений, поддерживался постоянным. Кривые были сняты при различных значениях потенциала -0.1, -0.2, -0.3, -0.4, -0.5, -0.6, -0.7, -0.8 и -0.9 В при комнатной температуре. Исследование этих кривых можно проводились путем сравнения экспериментальных расчетных данных с уравнениями модели Шарифкера-Хиллса. Установлено, что в ранней стадии, при потенциалах -0.1 и -0.2 В экспериментальные данные укладываются в кривую прогрессивной модели зародышеобразования, согласно которой зародышеобразование Ni-Mo происходит на многих активных участках поверхности подложки, но при остальных потенциалах (от -0.3 до -0.9 В) осаждение отклоняется в сторону мгновенного зарождения и роста.

Ключевые слова: потенциометрия, хроноамперометрия, хронопотенциометрия, нуклеация, никель, молибден