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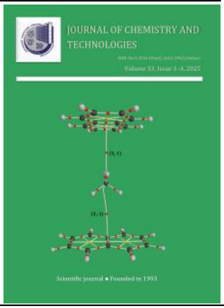

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ELECTROSYNTHESIS OF NI-FE FILMS WITH ENHANCED ELECTROCATALYTIC ACTIVITY FOR HYDROGEN EVOLUTION FROM ALKALINE ELECTROLYTE

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Abstract

The work is devoted to the topical issue of creating electrocatalysts for hydrogen evolution from alkaline electrolyte. The rapid development of hydrogen energy using the electrolytic method of hydrogen requires the use of cheap, resource-intensive and highly catalytic cathode materials. The most variable and flexible in control is the electrochemical method of synthesizing electrocatalysts of this type. A very promising material for cathodic hydrogen release is the Ni-Fe alloy. The limiting factor for its use is the high internal stresses that arise during its electrosynthesis. To reduce the internal stresses of Ni-Fe electrocatalyst films, sulfur-containing modifiers sodium allyl sulfonate and sodium saccharinate were used in this work, which were introduced into the methanesulfonate electrolyte of electrodeposition. It was established that increasing the concentration of sodium allyl sulfonate from 30 to 100 mmol/l leads to a decrease in internal stresses from 300 MPa to 100 MPa. The use of sodium saccharinate in a concentration of 0.5 mmol/l to 6 mmol/l contributes to a decrease in internal stresses from 300 MPa to 0 MPa. This effect of modifiers on internal stresses is associated with a change in the alloy structure due to the incorporation of sulfur and hydrocarbon residues of modifiers. It was found that the Ni-Fe alloy obtained in the presence of allyl sulfonate demonstrates high electroactivity, approaching the electroactivity of platinum. This effect is explained by an increase in defects in the crystal lattice and the presence of nickel and iron sulfide particles in the structure of the cathode material. It is recommended to carry out the electrosynthesis of Ni-Fe, as electrocatalysts for hydrogen evolution, from a methanesulfonate electrolyte containing 80–100 mmol/l of sodium allyl sulfonate in the range of current densities from 3 to 7 A/dm^2.

Keywords: hydrogen evolution electrocatalyst; Ni-Fe films; sulfur-containing organic substances; structure; internal stresses.

ЕЛЕКТРОСИНТЕЗ ПЛІВОК Ni-Fe З ПІДВИЩЕНОЮ ЕЛЕКТРОКАТАЛІТИЧНОЮ АКТИВНІСТЮ ЩОДО ВИДІЛЕННЯ ВОДНЮ З ЛУЖНОГО ЕЛЕКТРОЛІТУ

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Анотація

Робота присвячена актуальній темі створення електрокаталізаторів виділення водню з лужного електроліту. Найбільш варіабельним і гнучким в керуванні є електрохімічний спосіб синтезу електрокаталізаторів такого типу. Вельми перспективним матеріалом для катодного виділення водню є сплав Ni-Fe. Обмежуючим фактором для його використання є високі внутрішні напруги, що виникають під час його електросинтезу. Для зниження внутрішніх напруг плівок електрокаталізаторів Ni-Fe в даній роботі використано сульфурвмісні модифікатори аллілсульфонат натрію і сахаринат натрію, які вводили в метансульфонатний електроліт електроосадження. Встановлено, що збільшення концентрації аллілсульфонату натрію з 30 до 100 ммоль/л приводить до зменшення внутрішніх напруг з 300 МПа до 100 МПа. Застосування сахаринату натрію в концентрації від 0.5 ммоль/л до 6 ммоль/л сприяє зменшенню внутрішніх напруг з 300 МПа до 0 МПа. Такий вплив модифікаторів на внутрішні напруги пов'язаний з зміною структури сплаву за рахунок інкорпорації сульфуру і вуглеводневих залишків модифікаторів. Встановлено, що сплав Ni-Fe, отриманий у присутності аллілсульфонату, демонструє високу електроактивність, що наближається до електроактивності платини. Такий ефект пояснено збільшенням дефектів у кристалічній гратці і наявністю в структурі катодного матеріалу сульфідних частинок нікелю і заліза. Рекомендовано проводити електросинтез Ni-Fe як електрокаталізатору виділення водню з метансульфонатного електроліту, що містить 80–100 ммоль/л аллілсульфонату натрію в діапазоні густин струму від 3 до 7 А/дм^2.

Ключові слова: електрокаталізатор виділення водню; плівки Ni-Fe; сульфурвмісні органічні речовини; структура; внутрішні напруги.

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Introduction

A marker of the development of modern society is the amount of energy consumed, which is necessary to create material goods and increase the comfort of people's lives [1]. The continuous increase in energy consumption has prompted humanity to search for energy resources that are alternative to fossil fuels [2,3]. A major breakthrough in solving the energy problem occurred with the creation of nuclear power, which allowed us to significantly reduce the cost of electricity. The next step in reducing the cost of electricity and introducing mobility in its generation was the industrial production of solar batteries [4]. Direct conversion of solar energy into electricity has become widely available and this direction of electricity generation is rapidly developing. With the advent of cheap electricity, the issue of developing hydrogen energy through the production of high-purity hydrogen by electrolysis has become relevant. This method of obtaining an energy-intensive resource is indispensable for fuel cells, the use of which in the near future is expected not only in aerospace and other high-tech industries, but also in conventional industrial facilities and in the transport sector [5].

The key issue in the development of a water electrolyzer is to ensure minimal energy consumption, which is mostly solved by reducing the overvoltage of hydrogen and oxygen evolution at the electrodes. The lowest overvoltage of hydrogen evolution is characteristic of platinum, rhodium, ruthenium and iridium [6]. However, the high cost of these rare metals has led to an extensive search for alternative materials with similar electrochemical characteristics. The electrochemical method of applying thin-film electrocatalysts for hydrogen evolution should be singled out as the most rational in terms of process controllability and extremely wide possibilities of varying the composition and structure of the resulting films.

Recently, a large number of studies of the electrocatalytic properties of hydrogen release of electrodeposited coatings from alloys and composites based on transition metals, mainly iron group, have been conducted [7]. The vast majority of synthesized materials contain nickel as a d-metal, which has a relatively small overvoltage of hydrogen evolution in an alkaline medium. To increase the electrocatalytic activity and wear resistance of cathode materials, it was proposed to modify nickel with molybdenum [8,9], tungsten [10; 11], chromium [12; 13], etc.

It has been shown that in nickel-based electrocatalysts, the synergistic effect between Ni and neighboring heteroatoms such as Fe, O, N, and P leads to an increase in surface adsorption due to changes in electronic properties and, possibly, an increase in surface area [14]. The authors [15; 16] reported that the Ni-Fe alloy is characterized by high rates of hydrogen evolution from alkaline solutions due to the fact that the rate of hydrogen adsorption and desorption on the nickel surface is significantly accelerated by iron, since unfilled d-orbitals (d6-orbitals of Fe) are able to facilitate hydrogen desorption according to the Geyrovsky mechanism. The high electrocatalytic and anti-corrosion properties of the Ni-Fe alloy were noted in [17]. In [18], a Ni-Fe alloy with a nickel content of 40 % was subjected to anodic treatment in hydrochloric acid for surface development. The obtained cathode showed half the overvoltage values of hydrogen evolution compared to smooth nickel. The electrocatalytic activity of the Ni-Fe alloy depends on its composition. The highest catalytic activity for the electrochemical reaction of hydrogen evolution from a one-molar KOH solution was demonstrated by the cathode obtained from an electrolyte containing nickel ions and iron (III) ions in a ratio of 4 to 6 [19]. In [20] it was found that an increase in the iron content in the Ni-Fe alloy leads to a decrease in the hydrogen evolution overvoltage in a 25 % KOH solution at 80 °C. However, in parallel with the saturation of the alloy with iron (III), the pitting corrosion of the electrodeposited films increases, which requires optimization of the conditions for their production. It should be noted that it is possible to obtain a Ni–Fe alloy electroplating with acceptable physical and mechanical properties only in the presence of organic sulfo compounds, which contribute to a decrease in the internal stresses of the deposits. Given the active use of methanesulfonate electrolytes in modern electroplating [21–24], it is relevant to establish the nature of the influence of the most common organic sulfo compounds, which reduce internal stresses in electroplated nickel and its alloys, on the structure, physical, mechanical, and electrocatalytic properties of the Ni–Fe alloy electrodeposited from methanesulfonate electrolyte.

Research methods

Electrodeposition of Ni–Fe alloy films was carried out(conducted) from a methanesulfonate electrolyte of the following composition: 1.00 M $Ni(CH_3SO_3)_2$+0.30 M NaCl+0.70 M H_3BO_3+0.08 M

$Fe(CH_3SO_3)_2$. As special electrocrystallization modifiers, sulfur-containing organic compounds sodium allyl sulfonate and sodium saccharinate were used. The reagents used in the experiments had a qualification of not lower than (p.f.a), distilled water. All experiments were carried out at an electrolysis temperature of 333 K and pH 3.

The internal stresses of the electrodeposited films were determined by the flexible cathode method. A copper plate measuring 50×20 was insulated on one side and fixedly fixed on top. When forming a film during electrolysis, internal stresses of the deposit arise, which lead to bending of the cathode. The equation for calculating internal stresses $\sigma_{вн}$ has the form [25]:

$$\sigma_{is} = \frac{E_c \cdot d_c \cdot (d_c + d_{dep}) \cdot z}{3 \cdot l^2 \cdot d_{dep}}, \qquad (1)$$

where E_c is the modulus of elasticity of the cathode plate; d_c is the thickness of the cathode; d_{dep} is the thickness of the deposit; l is the length of the working part of the cathode; z is the deviation of the cathode end from the initial position.

The thickness of the cathode deposit with the Ni–Fe alloy was 25 µm. Each experiment was repeated at least 5 times. The confidence interval limits for the values of the internal stresses of the deposits were found using the Student's t-test for a confidence probability of 95 %.

The structure of nickel-iron films was studied using the X-ray diffractometer DRON–3 in monochromatized CuK_α-radiation. Measurement of the sulfur content in Ni–Fe alloy films was performed by the fluorescent X-ray method using a portable X-ray universal technical spectrometer "Sprut" X100 with a Si(Li) detector [26].

The current-voltage dependences of hydrogen evolution were obtained using an MTech SPG-500 fast potentiostat. Platinum, nickel, iron and samples with electrodeposited Ni–Fe alloy films were used as working electrodes. Platinum served as an auxiliary electrode, and a chloride-silver electrode of the EVL – 1M1 brand, immersed in a saturated solution of potassium chloride, was used as a reference electrode. The experiments were carried out in an electrochemical cell made of heat-resistant glass with a volume of 50 cm^3. The temperature of the solutions was maintained at 50 °C using the thermostat UT – 15 with an accuracy of 0.5 °C.

Results and discussion

Electrolytic films of Ni–Fe alloy, obtained from methanesulfonate electrolyte without special organic compounds, at a thickness of 25 µm are characterized by significant internal tensile stresses, which leads to cracking of such films and loss of their adhesion to the base. The study of the influence of sulfur-containing organic compounds selected in this work on the internal stresses of galvanic deposits with Ni–Fe alloy showed that when sodium allyl sulfonate is introduced into the electrolyte, the internal tensile stresses decrease (Fig. 1).

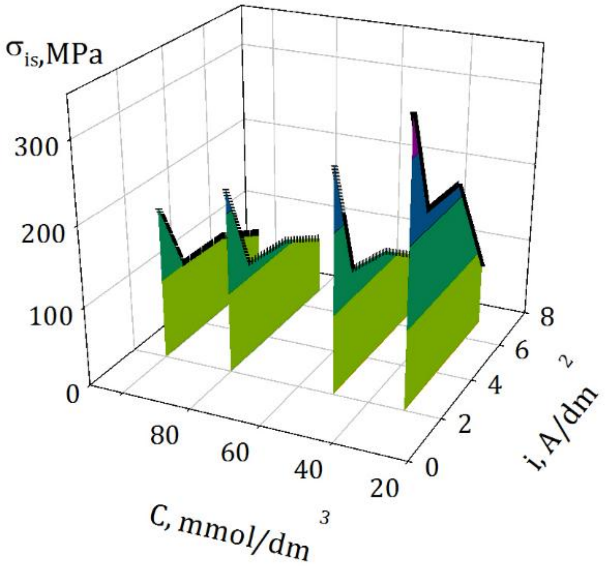

Fig. 1. Dependence of internal stresses of Ni-Fe films on electrodeposition current density and sodium allyl sulfonate concentration in the electrolyte

Moreover, when the concentration of sodium allyl sulfonate is increased from 30 mmol/l to 50 mmol/l, a sharp decrease in internal stresses is observed, with practically constant values upon further addition of this compound to the electrolyte. It should be noted that the effect of allyl sulfonate on the internal stresses of Ni–Fe films increases with increasing electrodeposition current density. In particular, at a current density of 2 A/dm^2, this effect is minimal, and it is characteristic that an increase in the concentration of sodium allyl sulfonate in the electrolyte at this current density leads to a significant difference in the values of internal stresses. Therefore, it can be stated that an increase in current density contributes to a more effective effect of sodium allyl sulfonate on the structure of Ni–Fe films in the context of their internal stresses. Probably, the adsorption of this organic substance on the cathode surface increases in the region of more negative potentials, which may be associated with its anionic nature. Unlike sodium allyl sulfonate, the use of sodium saccharinate allows obtaining Ni-Fe alloy films without internal stresses. The concentration of this substance required for complete elimination of internal stresses is 6 mmol/L (Fig. 2).

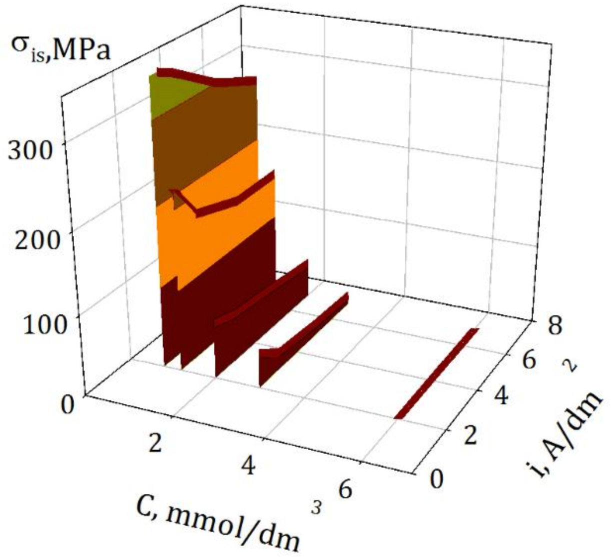

Fig. 2. Dependence of internal stresses of Ni-Fe films on electrodeposition current density and sodium saccharinate concentration in the electrolyte

Increasing the concentration of sodium saccharinate in the electrolyte for electrodeposition of the Ni-Fe alloy proportionally reduces the internal stresses of the deposits. The distribution of the values of internal stresses depending on the current density for sodium saccharinate is much more uniform compared to sodium allyl sulfonate. As can be seen from the obtained data, a decrease in the internal tensile stresses of nickel-iron films is achieved by introducing sodium saccharinate into the electrodeposition electrolyte, the concentration of which is two orders of magnitude lower than in the case of using sodium allyl sulfonate. The high efficiency of sodium saccharin is apparently associated with a more intensive incorporation of its degradation products into the cathode deposit, which is associated with a stronger adsorption of this substance on the electrode surface. As is well known, the electronic structure of nickel and iron contains an unfilled d-orbital, and organic molecules with excessive electron density, in addition to adsorption under the action of electrostatic forces and Van der Waals forces, are capable of forming a stronger bond with the metal. Apparently, the relatively high adsorption capacity of sodium saccharinate is due to the fact that the nitrogen atom present in the molecular structure has an unshared electron pair, which takes part in the creation of a bond with the surface atoms of nickel and iron.

It should be noted that the presence of the studied sulfur-containing organic compounds in the methanesulfonate electrolyte for electrodeposition of the Ni–Fe alloy affects the structure of the obtained precipitates. This is evidenced, in particular, by the change in the values of the dislocation density in the precipitates. As can be seen from Fig. 3, an increase in the concentration of sodium allylsulfonate in the electrolyte to 30 mmol/l causes a sharp increase in the dislocation density to $65 \cdot 10^{10}$ 1/cm^2. Further saturation of the electrolyte with sodium allylsulfonate has little effect on the value of the dislocation density. Sodium saccharinate is characterized by a slight increase in the dislocation density, which is about 40 % at a concentration of this substance in the electrolyte of 10 mmol/l (Fig. 4).

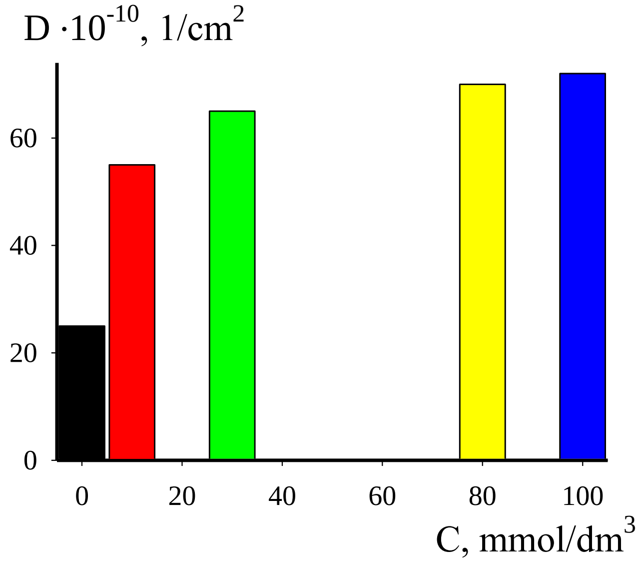

Fig. 3. Dependence of the dislocation density of Ni-Fe films on the concentration of sodium allyl sulfonate in the electrolyte

The established effects of the influence of sulfur-containing organic substances on the physical and mechanical properties and structure of electrodeposited films of Ni-Fe alloy obtained from methanesulfonate electrolyte can be explained on the basis of the following considerations. The studied modifiers of the structure of the electrolytic alloy contribute to the reduction of internal tensile stresses due to the introduction of sulfur into the crystal lattice of the electrolytic deposit and the weakening of the effect of dislocations emerging to grain boundaries. In addition, hydrocarbon decomposition products of organic substances can enter the deposits, resulting in internal compressive stresses.

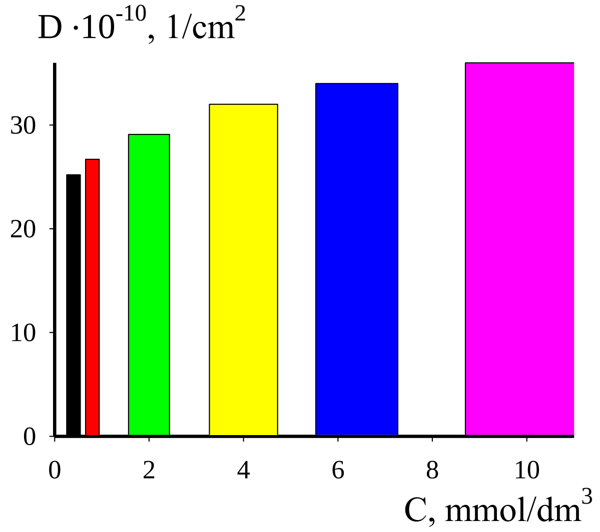

Fig. 4. Dependence of the dislocation density of Ni-Fe films on the concentration of sodium saccharinate in the electrolyte

As can be seen from Table 1, the sulfur content in the films increases with increasing concentration of modifiers in the electrolyte.

Table 1

Sulfur content in Ni-Fe films obtained from methanesulfonate electrolyte

Modifier	**Modifier concentration, mmol/**	**Sulfur conten wt.%**
Without modifier	–	0.00
Sodium allyl sulfonate	10	0.02
	80	0.4
Sodium saccharinate	2	0.02
	6	0.03

It should be noted that the effect of reducing the internal tensile stresses of electroplated films with Ni-Fe alloy due to the introduction of sulfur into the deposit is not sufficient to obtain stress-free deposits. This is evident from the data obtained using sodium allyl sulfonate, where an increase in the concentration of the modifier achieves a maximum value for dislocation density, but the internal tensile stresses are not completely eliminated. The dependencies obtained when using sodium saccharinate are indicative in this regard. With a significantly lower sulfur content and lower values of the dislocation density in the deposits compared to films deposited in the presence of sodium allyl sulfonate, electroplated deposits with Ni-Fe alloy are obtained without internal stresses. This is due to the fact that the products of sodium saccharinate transformations at the cathode are intensively introduced into the film and provoke the occurrence of internal compressive stresses, which compensate for the internal tensile stresses. As a result, it becomes possible to electrodeposit Ni-Fe films, the internal stresses of which are not detected.

Studies of the electrochemical behavior of the obtained Ni-Fe films in relation to the reaction of hydrogen evolution from a solution containing 0.5 mol/l KOH showed the presence of electrocatalytic activity of these films. As can be seen from Fig. 5, the hydrogen evolution overvoltage on Ni-Fe alloys is lower than on individual alloy components.

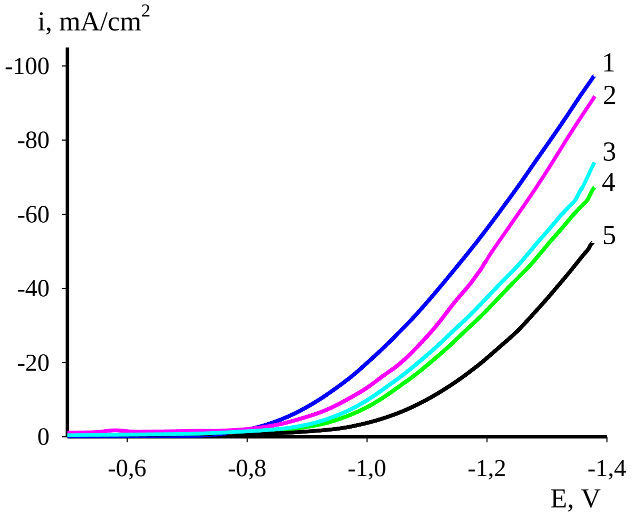

Fig. 5. Current-voltage dependences of hydrogen evolution from 0.5 M KOH at a temperature of 50°C on the surface of: 1 – platinum; 2 – Ni-Fe, obtained in the presence of 100 mmol/l sodium allyl sulfonate; 3 – Ni-Fe, obtained in the presence of 6 mmol/l sodium saccharinate; 4 – Ni; 5 – Fe

The evolution of hydrogen on the Ni-Fe alloy obtained from a methanesulfonate electrolyte containing sodium allyl sulfonate occurs with less kinetic limitations compared to the cathode obtained in the presence of sodium saccharinate. Such an electrocatalytic effect can be associated with two circumstances. The electrocatalytic properties of metals increase with increasing crystalline heterogeneity of the cathode surface. As can be seen from the obtained experimental data, the dislocation density of the Ni-Fe alloy obtained in the presence of allyl sulfonate is twice as high as the values corresponding to the film electrodeposited in the presence of sodium saccharinate. Therefore, the difference in the structure of these cathode materials with the presence of a larger number of defects in the electrocatalyst, which demonstrates lower values of the hydrogen evolution overvoltage, is obvious. The second important point is that the electrodeposition of the Ni-Fe alloy carried out in the study is accompanied by the incorporation of sulfur into the precipitate. As is known [27; 28], nickel sulfides and Ni-Fe-S structures are characterized by a synergistic effect in the release of hydrogen due to the peculiarities of the electronic configuration, the increased number of active centers and good dissipation of gaseous products. The amount of sulfur incorporated into the electroplating films obtained in the presence of 80 mmol/L sodium allyl sulfonate is several times higher than when using 6 mmol/L sodium saccharinate. This may indicate the influence of sulfur on the electrocatalytic activity for hydrogen evolution.

Therefore, the conditions for the electrosynthesis of Ni-Fe alloy films as electrocatalysts for hydrogen evolution from an alkaline electrolyte must meet two main requirements. Such films must have high physical and mechanical properties, in particular, have low internal stresses. And, obviously, they must have high electrocatalytic activity. The optimal choice in this case is the electrodeposition of a Ni-Fe electrocatalyst from a methanesulfonate electrolyte containing 80–100 mmol/L of sodium allyl sulfonate.

Conclusions

1. The work investigated the effect of sulfur-containing organic substances sodium allyl sulfonate and sodium saccharinate on the internal stresses and electrocatalytic activity for hydrogen evolution from alkaline solutions of Ni-Fe films electrodeposited from methanesulfonate electrolyte. It was found that the internal stresses of the Ni-Fe alloy obtained in the presence of allyl sulfonate are reduced to 100 MPa, which is more than three times less than in the absence of a modifier of the structure of electroplated deposits. The use of sodium saccharinate as a modifier of the structure of the Ni-Fe alloy in an amount of 6 mmol/l allows obtaining films with zero internal stresses. The effect of the studied modifiers on the value of the internal stresses of the obtained films is due to the blocking of the release of dislocations due to the incorporation of sulfur into the precipitates and the increase in the intercrystalline volume due to the ingress of hydrocarbon residues into the modifier.

2. The electrocatalytic activity of Ni-Fe films obtained from methanesulfonate electrolyte in the presence of sulfur-containing modifiers of the precipitate structure was determined. It was shown that the use of sodium allyl sulfonate allows obtaining Ni-Fe films with high electrocatalytic properties. The overvoltage of hydrogen evolution from an alkaline electrolyte on such electrodes is close to the overvoltage on platinum. To obtain Ni-Fe electrocatalysts with low internal voltages and high electrocatalytic activity for the reaction of hydrogen evolution from an alkaline electrolyte, a methanesulfonate electrolyte with a sodium allyl sulfonate content of 80–100 mmol/l is recommended. The range of electrodeposition current densities is 3–7 A/dm^2.

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