Були синтезовані плівки гідроксиду нікелю катодним темплатним методом з електроліту, що містить добавки кобальту, цинку і алюмінію. Було показано, що тільки додавання кобальту призводить до поліпшення оптичних і електрохімічних характеристик. Крім того, додавання кобальту в електроліт осадження веде до утворення нікель-кобальтового СДГ, який при затемненні і освітленні має двокольоровий перехід забарвлення: безбарвний – бронзовий – темно-синій. Добавка алюмінію веде до повного отруєння електрода, в свою чергу додавання цинку погіршує електрохромні властивості електрода

Ключові слова: гідроксид нікелю, $Ni(OH)_2$, електрохромізм, електроосадження, катодний темплатний синтез, полівініловий спирт, слоистый двойной гидроксид

Были синтезированы пленки гидроксида никеля катодным темплатным методом из электролита, содержащего добавки кобальта, цинка и алюминия. Было показано, что только добавление кобальта приводит к улучшению оптических и электрохимических характеристик. Кроме того, добавка кобальта в электролит осаждения ведет к образованию никель-кобальтового СДГ, который при затемнении и осветлении имеет двухцветный переход окраски: бесцветный – бронзовый – темно-синий. Добавка алюминия ведет к полному отравлению электрода, в свою очередь добавление цинка ухудшает электрохромные свойства электрода

Ключевые слова: гидроксид никеля, $Ni(OH)_2$, электрохромизм, электроосаждение, катодный темплатный синтез, поливиниловый спирт, слоистый двойной гидроксид

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ELECTROCHROMISM OF $Ni(OH)_2$ FILMS OBTAINED BY CATHODE TEMPLATE METHOD WITH ADDITION OF AL, ZN, CO IONS

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1. Introduction

Nickel (II) hydroxide is used in power sources as an active material for a positive electrode [1, 2]. This compound also finds application in other fields: oxidation of organic compounds [3], sensors [4], electrochromic films [5].

Electrochromic films can be employed in so-called "smart windows". With the help of electrochromic films deposited onto glass panes with transparent conductors, these devices can alter their optical properties – color, transparency, haziness. Thus, these devices can be used as controlled wind blinds. They can create suitable illumination and as a result – suitable temperature in the room. These devices also have potential application in medical establishments, advertisement and automobile industry, etc.

The electrochromic devices can be based on different principles: use of liquid crystals [6], devices with suspended particles [7], electrochemical electrochromic materials [8], etc.

Electrochemical electrochromic materials change their color under oxidizing (anodic electrochromic materials) or reducing (cathodic electrochromic materials) current. Under the current of opposite polarity, they return to the initial state.

Nickel (II) hydroxide is amongst anodic electrochromic materials. Nickel hydroxide thin films are transparent in the reduced state and are dark-brown in the oxidized state. This material is viewed as promising, owing to potentially long life cycle, relatively low cost and high coloration degree [9].

Despite that $Ni(OH)_2$ has been known for a long time, as an electrochromic material, it still continues to attract researchers. This leads to new papers regarding the development of deposition methods, search of additives and study of coloration mechanism.

One of the promising methods for deposition of $Ni(OH)_2$ thin films is developing the cathodic template method [10].

In this method, the template – polyvinyl alcohol forms a matrix, in which $Ni(OH)_2$ is deposited according to reactions 1 and 2:

$$NO_3^- + H_2O + 2\bar{e} \rightarrow NO_2^- + 2OH^-, \quad (1)$$

$$2OH^- + Ni^{2+} \rightarrow Ni(OH)_2. \quad (2)$$

As a result, a composite of nickel hydroxide film and polyvinyl alcohol is formed. Such a film has good electro-

chromic properties and excellent physicochemical characteristics: high adhesion to the substrate and uniformity.

2. Literature review and problem statement

There are many laboratory methods for preparing oxide and hydroxide nickel materials for electrochromic applications. There are many known methods for the $NiO/Ni(OH)_2$ film deposition: magnetron sputtering [11], vacuum deposition [12], chemical bath deposition [13], sol-gel [14], pulse laser deposition [15], spray pyrolysis [16] and electrochemical deposition [17].

Nevertheless, only electrochemical methods are the simplest, cheapest and easily realized through technological lines of a galvanic production, while not requiring powerful power sources or sustained vacuum. It should also be taken into account that all technological expenses of other deposition methods would be included in the price of the final product, increasing its cost.

Electrochemical deposition methods are simple to use, do not require powerful power sources, easy to automate. In addition, the resulting properties of electrochromic films can be modified by changing the electrolyte composition, electrolysis conditions – current density, deposition duration and electrolyte temperature.

The cathodic template deposition method is one of the various variants of electrochemical hydroxide film deposition [18]. Some amount of water-soluble polymer (polyvinyl alcohol) is added to a solution of nickel nitrate, which forms a matrix in a solution and is co-deposited with nickel hydroxide film as a result of electrochemical reaction. The advantage of this method is high quantitative characteristics of electrochemical films obtained.

In order to improve specific characteristics of $Ni(OH)_2$ as an active material of accumulators, supercapacitors and electrochromic films, mixed hydroxides are prepared in laboratory conditions. These hydroxides form a structure different from initial nickel hydroxide and are called Layered Double Hydroxides (LDH). The coprecipitation is carried out at a specific ratio of nickel and second metal. Use of the following metals is known for coprecipitation: Al, Zn, Mn, Co, Fe, Cr and Cd [19]. Additionally, the prepared LDHs often possess higher specific characteristics. In the paper [20], it is mentioned that the strongest influence on the resulting properties of $Ni(OH)_2$ electrode is exerted by additives of cobalt and zinc. While presence of Al^{3+} ions in electrolyte poisons the electrode, but when coprecipitated with nickel, aluminum ions don't cause a poisoning effect. On the contrary, in the paper [21], it has been demonstrated that LDH precipitated with nickel has significantly higher specific electrochemical characteristics.

In order to evaluate the possibility of preparing an LDH and to evaluate its electrochromic properties, a decision has been made to conduct the deposition of nickel LDH with cobalt, zinc and aluminum. The deposition was carried out using the cathodic template method.

3. The aim and objectives of the study

The aim of the work is to evaluate the possibility of depositing nickel-aluminum, nickel-cobalt and nickel-zinc LDH using the cathodic template method and to determine the influence of adding the corresponding metal ions to the deposition electrolyte on electrochromic and electrochemical properties of prepared films.

In order to achieve the set-out aim, the following objectives were set:

– to prepare the electrochromic films from a mixed solution of nickel and the second metal (Al, Zn and Co) using the cathodic template synthesis and study their properties;

– to compare the structure of the obtained films and also their electrochromic and electrochemical properties, depending on the composition of the deposition electrolyte.

4. Research methods used to study the influence of the metal cation addition on deposition of PVA-$Ni(OH)_2$ films

For deposition, the electrolytes with the addition of zinc, cobalt or aluminum salts with a predefined molar ratio of nickel to metal (4:1) were used, according to the literature [21]. In case of cobalt, two molar ratios were used – previously defined molar ratio (4:1) and two times lower (8:1).

All regimes for film deposition of electrochromic films are listed in Table 1. The cell used for deposition of nickel hydroxide films was split into anodic and cathodic space with low permeability diaphragm.

Table 1

Electrolyte composition and regimes for the cathodic template synthesis of LDH

Sample* (conventional label)	Ni:Me	C (Ni^{2+}), mol/L	C (Me), mol/L	PVA content, %	Deposition time, min	Drying temperature, °C
Ni	–	1	–	5	80	20
Ni-Al 4:1	4:1	1	0.25 (Al^{3+})	5	80	20
Ni-Zn 4:1	4:1	1	0.25 (Zn^{2+})	5	80	20
Ni-Co 4:1	4:1	1	0.25 (Co^{2+})	5	80	20
Ni-Co 8:1	8:1	1	0.125 (Co^{2+})	5	80	20

*Note: * – for all samples, the drying time was 24 hours, the deposition was carried at 0.625 mA/cm², 1M KNO_3 solution was used in anodic space*

Electropolished nickel foil (Fig. 1, *a*) was used as a substrate for deposition of electrochromic films. Electrode working area was 4 cm^2. The electropolishing was conducted right before deposition of the electrochromic film. After electropolishing, the electrode was washed with large amounts of warm distilled water.

The metal substrate was chosen because of two reasons:

– higher conductivity of nickel, in comparison to indium-tin oxide coat on glass results in better distribution of current density along the electrode's height;

– better sensitivity during recording a change of the film coloration-bleaching because of light passing two times through the $Ni(OH)_2$ film: light source → $Ni(OH)_2$ film → mirror-like nickel substrate → $Ni(OH)_2$ film → photoresistor.

For measuring optical and electrochemical properties, cyclic voltamperometry (CVA) with simultaneous recording of the coloration-bleaching process were used. The cell shown in Fig. 1, *a*, *b* was used for this purpose. Ag/AgCl (KCl sat.) was used as a reference electrode. Nickel foil was used as a counter electrode. As an electrolyte, 0.1 M KOH

solution was used as an electrolyte. Optical characteristics were recorded using ADC E-154 (Russian Federation), electrochemical characteristics were recorded using digital potentiostat-galvanostat Ellins P-8 (Russian Federation). The regime of electrochemical and optical measurements: potential range from +200 to +750 mV, scan rate 1 mV/s, number of cycles – 5. The coloration degree, calculated as a difference between transparency in the colored and bleached states averaged from 5 cycles, was used as one of the characteristics that characterize electrochromic properties.

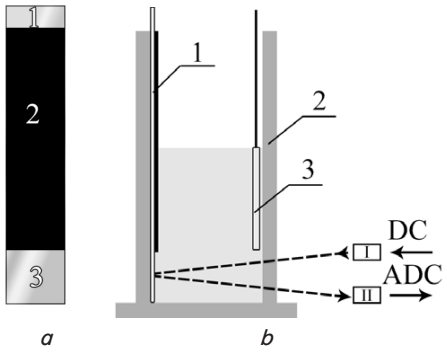


Fig. 1. Electrode and cell for electrochemical and optical measurements: *a* – working electrode: 1 – region for connecting a polarizing wire; 2 – black dielectric on the foil surface; 3 – mirror-like surface with electrochromic film; *b* – cell for recording optical characteristics: 1 – working electrode; 2 – cell frame made out of transparent plastic; 3 – counter electrode; I – source of white (5500 K) light; II – photoresistor; DC – source of stabilized current; ADC – analogue-to-digital converter

In order to determine the structure of the obtained hydroxide films, X-ray diffraction patterns were recorded in Co-Kα monochromated radiation without separating the electrochromic film from the substrate, using diffractometer DRON-3 (Russian Federation).

5. Experimental results of deposition of nickel hydroxide, prepared with Al, Co, Zn and from pure nickel nitrate

As a result, a few electrochromic films were prepared using the cathodic template synthesis: from pure nickel nitrate, from nickel nitrate with aluminum, from nickel nitrate with zinc and from nickel nitrate with cobalt (with two molar ratios of nickel to cobalt). It should be noted that during the deposition with zinc, some foam formation was observed on the electrode. It is obvious that the addition of zinc changes the deposition mechanism. Most likely, the zinc ions ease hydrogen evolution or increase overpotential of nitrate ion reduction (Reaction 1).

5. 1. Structural comparison of nickel hydroxide films prepared with Al, Co, Zn and from pure nickel nitrate

All experimentally prepared nickel hydroxide films were studied by means of X-ray diffraction. Analysis results are presented in Fig. 2.

Based on the presented Fig. 2, it can be said that all XRD patterns are similar except for sample Ni-Zn 4:1. High-intensity peaks at 52 and 62° correspond to metallic nickel used as a substrate. All samples prepared using the cathodic template method; have low crystallinity, high quantity of defects and, likely, a large amount of structural water, however, the latter assumption requires verifications by other methods.

It can also be added that in all films prepared at a nickel to metal ratio of 4:1, a first peak at (12–13°) can be observed, which corresponds to the α-$Ni(OH)_2$ structure, that is characteristic for all LDH [21].

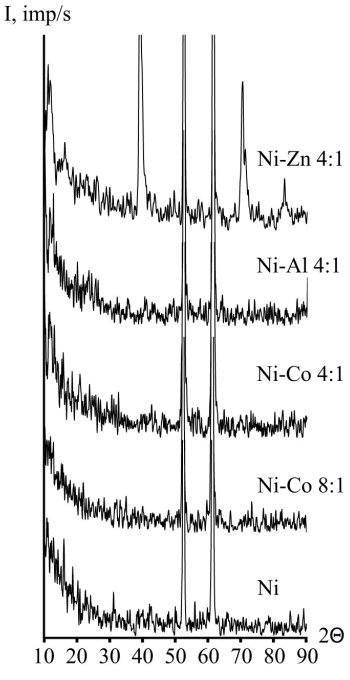


Fig. 2. XRD patterns of nickel hydroxide films prepared from pure nickel nitrate and with metal additives to the deposition electrolyte

In case of the Ni-Zn 4:1 sample, peaks that are not characteristic for nickel hydroxide are observed. Obviously, the deposition at the electrode resulted in a physical mixture of compounds – nickel hydroxide with zinc oxide.

5. 2. Comparison of properties of $Ni(OH)_2$ films prepared with and without additives

Electrochromic films were prepared using the cathodic template synthesis in presence of Zn, Al and Co and also from pure nickel nitrate solution.

Film analysis results – cyclic voltamperograms and coloration-bleaching curves are presented in Fig. 3–7.

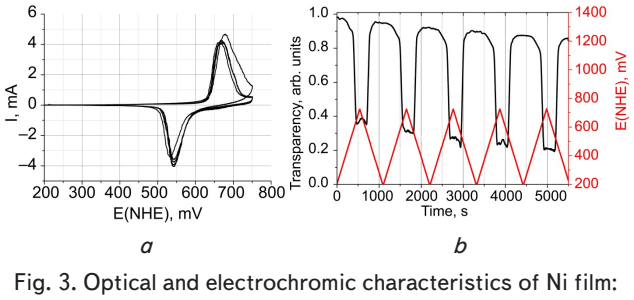
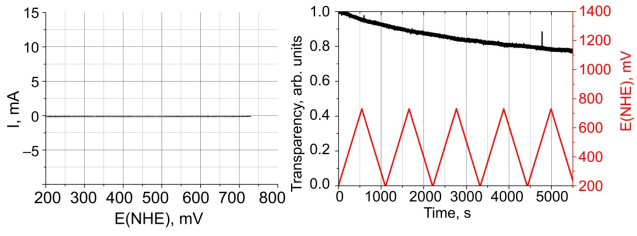


Fig. 3. Optical and electrochromic characteristics of Ni film: *a* – cyclic voltamperogram; *b* – coloration-bleaching curve

Fig. 3 shows the results obtained for the film prepared from pure nickel nitrate solution. Based on the cyclic voltammogram analysis, it can be said that the film quickly changes to the stationary regime, with the difference between potentials of the oxidation peak (660 mV) and

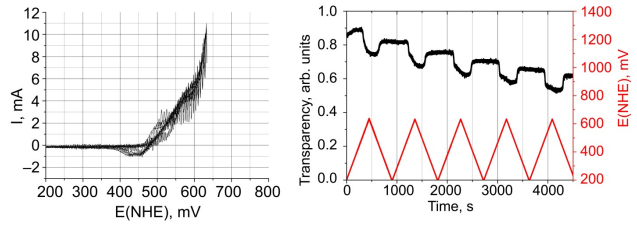
reduction peak (540 mV) being 120 mV. Current values of oxidation and reduction peaks are approximately 4 mA. The coloration degree is high and is 0.6 on average.



a *b*

Fig. 4. Optical and electrochromic characteristics of the Ni-Al 4:1 film: *a* — cyclic voltamperogram; *b* — coloration-bleaching curve

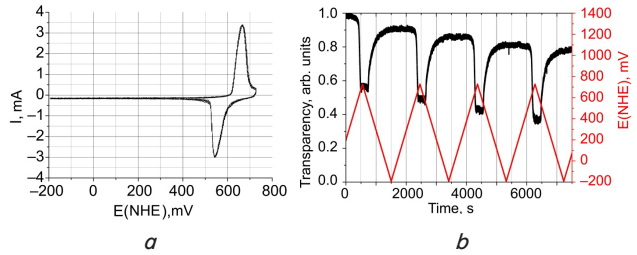
Fig. 4 shows the results for the Ni-Al 4:1 film. It can be seen from both graphs (Fig. 4, *a*, *b*), that this film shows no electrochemical or electrochromic activity. Such behavior is likely related to poisoning with aluminum ions that are present in the deposition electrolyte.



a *b*

Fig. 5. Optical and electrochromic characteristics of the Ni-Zn 4:1 film: *a* — cyclic voltamperogram; *b* — coloration-bleaching curve.

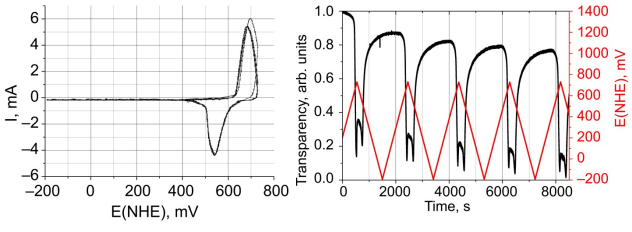
In turn, the Ni-Zn 4:1 film has demonstrated low electrochemical and electrochromic characteristics – Fig. 5. On the cyclic voltamperogram, the potential shift of oxidation and reduction peaks to more positive values – 450 mV and 500 mV correspondingly, is observed. In addition, the peak currents have reduced significantly in comparison to the film deposited from nickel nitrate solution without additives (Fig. 3) and are 1 mA for reduction and 2 mA for oxidation. The small difference between the colored and bleached states (0.15) further supports worsening of the film properties. It should also be noted that because of significant oxygen evolution, the cycling potential range has been limited to 640 mV in order to avoid film destruction by evolving oxygen.



a *b*

Fig. 6. Optical and electrochromic characteristics of the Ni-Co 4:1 film: *a* — cyclic voltamperogram; *b* — coloration-bleaching curve

Use of cobalt as an additive to the deposition electrolyte has led to significant improvement of electrochemical properties for both samples with cobalt; and electrochromic properties for the Ni-Co 8:1 sample, as can be seen after comparing Fig. 3, 6, 7. Cyclic voltamperogram of the Ni-Co 4:1 film demonstrated the peak values somewhat lower than those of the sample prepared from pure nickel nitrate solution. Additionally, the peaks were located at the same position, which indicated a high rate of film activation. The potentials of both peaks for the samples (Ni-Co 4:1 and Ni-Co 8:1) matched with the potentials of oxidation and reduction of the samples prepared from pure nickel nitrate and are 660 mV and 540 mV.



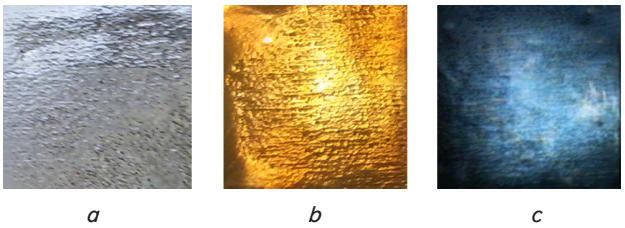
a *b*

Fig. 7. Optical and electrochromic characteristics of the Ni-Co 8:1: film: *a* — cyclic voltamperogram; *b* — coloration-bleaching curve

The difference between the colored and bleached states for the Ni-Co 4:1 sample was somewhat lower than for the Ni sample. While the Ni-Co 8:1 film had a higher coloration degree than the sample prepared from pure nickel nitrate solution and was 0.8 on average. Unlike other films, the film did not reduce completely to the initial state with each cycle.

A feature characteristic for the films deposited in presence of cobalt should be noted. Complete bleaching was not achieved in the potential range of 0 mV – +750 mV. Thus, the potential range was broadened to the negative value of –200 mV.

Defining characteristic of such films was two-stage coloration (Fig. 8). During anodic film oxidation, the film color changed to bronze (in case of the Ni-Co 4:1 film, the color was copper-bronze) with further oxidation the color changed to dark blue.



a *b* *c*

Fig. 8. Coloration stage of electrochromic Ni-Co 8:1 and Ni-Co 4:1 films (first color transition bleached → bronze; second color transition bronze → dark blue): *a* — bleached electrode; *b* — partially colored electrode; *c* — completely colored electrode

The discovered two-stage color change is interesting in that it can be used for indicators and artistic shading of windows in premises and automobiles.

6. Discussion of the metal cation additive influence on the characteristics of electrochromic nickel hydroxide films

The cathodic template deposition resulted in hydroxide films that showed different electrochemical and electrochromic characteristics from those prepared from pure nickel nitrate solution.

The film deposited from a solution containing aluminum did not show any electrochemical activity or electrochromic properties. Nevertheless, the XRD pattern showed a peak characteristic of the α-$Ni(OH)_2$ like structure. This can mean that deposition of LDH occurred, however, the absence of electrochemical activity is related to poisoning with aluminum ions, because of their presence in the deposition electrolyte. Such behavior of nickel oxide electrode is known for accumulator electrodes and is described in the literature [20].

The film deposited from the electrolyte containing zinc ions showed significant changes in comparison to the film deposited from pure nickel nitrate solution. Analysis of the obtained coloration-bleaching curves allows concluding that the addition of zinc to the deposition electrolyte for the cathodic template synthesis leads to worsening of the properties of the resulting film. Additionally, the authors assume that Ni-Zn LDH have not been prepared because of the presence of a few crystal phases in the sample, which can indicate the presence of a physical mixture of nickel hydroxide and zinc oxide on the electrode surfaces.

In case of deposition from a mixture of nickel and cobalt nitrates, the obtained film had good electrochemical and electrochromic properties. In addition, when cobalt was added to the deposition electrolyte, the Ni-Co LDHs were obtained. This was expressed in presence of peak at 12–13° on the XRD pattern. Partial worsening of electrochromic properties of the Ni-Co 4:1 sample is likely related to lower nickel content in the LDH film. The latter is defined by the fact that a part of nickel is substituted by cobalt, which doesn't partake in the main redox reaction.

In case of lower cobalt concentration (Ni-Co 8:1 sample), significant improvement of electrochemical and electrochromic properties was observed, with the highest coloration degree among all samples of about 0.8.

Additionally, the two-stage color transition was discovered for the Ni-Co 4:1 and Ni-Co 8:1 samples (transparent → → bronze → dark blue), which the authors assume to be related to the properties of nickel cobalt LDH.

7. Conclusions

1. It has been demonstrated that the addition of aluminum and cobalt to the deposition electrolyte allows preparing the corresponding LDH, however, the addition of aluminum leads to poisoning of the active material. The addition of zinc leads to worsening of electrochemical and electrochromic properties of the film, which is composed of a physical mixture of nickel hydroxide and zinc oxide.

2. In case of deposition from solutions containing cobalt, the prepared films posses high electrochemical and electrochromic properties. It also has been shown that the film prepared at a molar ratio of nickel to cobalt of 8:1 possesses the best electrochromic properties. The coloration degree of this film is 0.8, which is more than that for the film prepared from nickel nitrate solution without any additives by 0.2. Additionally, for all films prepared in presence of cobalt, the two-stage color transition was observed.

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