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INVESTIGATION OF THE CHARACTERISTICS OF SULFURIZED ELECTROCHROMIC NI(OH)$_2$-PVA FILMS DEPOSITED ON TRANSPARENT SUBSTRATES

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Obtaining new types of composite coatings for various purposes is the most important direction in chemistry. The synthesis of composite hydroxide-sulfide compounds can be useful in various areas of applied electrochemistry.

Using a simple two-step method, thin films composed of nickel hydroxide sulfide-polyvinyl alcohol were formed. The production of nickel hydroxide sulfide-polyvinyl alcohol composite was carried out by holding the nickel hydroxide-polyvinyl alcohol composite coating formed on the glass electrode with an electrically conductive substrate in a solution of 0.03 M Na_2S for 10 minutes. The formation of nickel sulfide on the surface of nickel hydroxide was shown indirectly. It was shown that there were no changes in morphology after the treatment of the electrode in sodium sulfide solution.

As a result of the treatment, the electrochemical and electrochromic characteristics changed. There was some deterioration in the average coloration depth from 55 % to 49 % for the electrode containing nickel sulfide. The electrochemical characteristics also deteriorated slightly after the formation of the sulfide film, namely, the specific capacitance, which went to the passage of anodic and cathodic processes. However, the capacitance efficiency increased from 83 % to 87 % for the sulfide-treated film. Despite this, this simple method is considered to be potentially promising for the formation of electrodes for use in other electrochemical devices.

In addition, due to the treatment in a sodium sulfide solution, it became possible to roughly determine the size of the nickel hydroxide clusters in the nickel hydroxide-polyvinyl alcohol composite coating. These clusters did not exceed 430 nm in size, which was almost equal to the lower limit of the wavelength of the visible spectrum

Keywords: electrochromism, nickel hydroxide, nickel sulfide, film, composite coating, polyvinyl alcohol

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1. Introduction

Metal oxides and hydroxides have taken a strong place among the materials used in electronic components [1, 2], solar cells [3], chemical current sources [4, 5], sensors [6], and other devices [7, 8]. Nanostructured oxide and hydroxide materials expand the capabilities of existing systems by improving their performance.

Examples of such materials are nickel oxide-hydroxide compounds [9]. The latter, due to the unique properties of the forms in which nickel is in the oxidation states 2^+ and 3^+, as well as the high reversibility of their transition between themselves, are used in electrodes of batteries [10] and supercapacitors [11]. A promising direction for using these compounds is the electrocatalytic deactivation of substances [12], electrooxidation of compounds in fuel cells [13], active elements of sensors [14], etc.

The elements of the so-called "smart windows" are the relevant direction of using nickel-based oxide-hydroxide compounds. Films of nickel oxides and hydroxides obtained

by special methods are transparent, and upon electrochemical oxidation, they acquire a dark brown color [15]. At the same time, by supplying a strictly metered amount of electricity, it is possible to obtain different degrees of color from a barely noticeable tint to a completely opaque state.

According to the National Renewable Energy Laboratory (USA) [16], the use of such windows can reduce the consumption of electricity needed for cooling premises by half, as well as reduce the load on the power grid during peak periods of consumption. The use of such devices, subject to a constant increase in ambient temperatures to save energy, as well as to increase comfort, is an important issue. Thus, the development of new materials, treatment methods, and technologies for creating electrochromic devices remains an urgent task of modern applied sciences.

2. Literature review and problem statement

Nickel hydroxide in the electrodes of alkaline batteries, supercapacitors, as well as in electrochromic elements of "smart windows" works quite reversibly. Despite the high reversibility of the reaction and, as a result, the possibility of using electrochemical devices in these groups, researchers are constantly making attempts to improve the specific characteristics of electrodes based on $Ni(OH)_2$. At the same time, there are several approaches to improving the characteristics: the use of nanostructured $Ni(OH)_2$ [17], the use of additives [18], doping [19], or the manufacture of nickel hydroxide composites [20], and a combined approach [21].

One of the methods used to improve the specific characteristics of active substances based on nickel hydroxide is the production of combined hydroxide sulfide-containing composites.

For example, in [22], a nanocomposite of nickel hydroxide and sulfide was synthesized for electrodes of asymmetric supercapacitors. The researchers showed that the electrodes gave 108 C/g at a current density of 3 A/g. The researchers also noted that the electrodes worked even at a current density of 50 A/g and a potential window of 0.65 V.

In another work [23], also concerning a Ni-Zn supercapacitor battery with a water-based electrolyte, thin sheets of nickel sulfide with the addition of molybdenum with layers of Ni-Co double hydroxide (Mo–NiS_2@NiCo-LDH) were synthesized. The synthesized material showed a high capacity of 325.6 mAh/g (2604.8 F/g) at 1 A/g.

Two-dimensional nickel hydroxide-sulfide nanosheets deposited on CdS nanospheres were studied as a cocatalyst for photocatalytic hydrogen evolution [24]. The study showed that the rate of photocatalytic release of H_2 for the synthesized material is 46 times higher than that of CdS. In addition, the photocatalytic stability of CdS in such a material is significantly improved under visible light irradiation.

Also in [25], a significant increase in the catalytic activity of oxygen evolution on Ni-Fe double hydroxide with a film coating, on a substrate with arrays of Ni_3S_2 nanosheets on foam nickel, was shown.

Many examples of hydroxide-sulfide or oxide-sulfide systems are known for metals other than nickel.

The authors in [26] showed that a material consisting of arrays of cobalt sulfide nanosheets based on aluminum hydroxide had a high capacity and stability. At the same time, the researchers showed that the capacitance reached 1,503 F/g at a current density of 2 A/g, and the specific capacity was 91 % of the theoretical one at a current density of 50 A/g.

A group of researchers synthesized WS_2–WO_3 heterostructures for lithium-sulfur batteries [27]. The latter, during electrochemical tests, showed high stability of parameters during cycling – a decrease in capacity by 0.06 % per cycle for 500 cycles at a current load of 0.5 of the full capacity.

As can be seen from the above examples, mixed metal hydroxides (oxides) and sulfides exhibit improved electrochemical characteristics. However, no attempts have been made so far to use such materials in electrochromic cells.

3. The aim and objectives of the study

The study aimed to determine the possibility of increasing the specific characteristics of an electrochromic coating based on $Ni(OH)_2$ using partial sulfurizing.

To achieve the aim, the following objectives were set:

– to carry out sulfurizing and evaluate changes in morphology and optical properties after the process;

– to determine the electrochemical and electrochromic characteristics of sulfurizing deposits of a composite electrochromic coating based on nickel hydroxide (II).

4. Research materials and methods

The production of a composite electrochromic coating was carried out on glass substrates coated with fluorine-doped tin oxide (FTO glass). The surface resistivity of the substrate was ≤10 Ohm/sq (China, Zhuhai Kaivo Optoelectronic Technology Co. Ltd).

Before coating, the substrates were treated in several steps. First, the substrates were rubbed with Na_2SO_4 paste. This was followed by rinsing with running and distilled water, as well as treatment in 96 % ethanol under sonication (60 W, 41,500 Hz).

$Ni(OH)_2$-polyvinyl alcohol (PVA) deposits were formed by the cathodic template method in the following mode:

– solution of 0.01 M $Ni(NO_3)_2$, 5 % PVA;

– deposition on the cathode with a current density of 0.1 mA/cm^2;

– duration of deposition – 10 min.

After electrodeposition, the samples were rinsed from water-soluble salts in distilled water at 30 °C for 10 minutes and dried for 1 day at room temperature.

Modification of the finished electrode was carried out through sulfurizing by holding the electrode with an electrochromic coating in a solution of sodium sulfide for 10 minutes. A 0.03 M sodium sulfide solution was used in the experiments. $Na_2S{\cdot}9H_2O$ crystalline hydrate of chemical purity grade was used for preparation.

4. 1. Electrochromic and electrochemical characteristics

The electrochemical characteristics were evaluated by the potentiodynamic method. The electrode was connected according to the three-electrode scheme. The potential was turned from +201 to +751 mV relative to a normal hydrogen electrode at a rate of 1 mV/s. A silver chloride electrode in saturated potassium chloride was used as a reference electrode. The cell was made of transparent polymethyl methacrylate. Nickel foil served as a counter electrode. In parallel with the determi-

nation of the electrochemical characteristics of the electrodes, changes in the transparency of the film were also recorded.

The setup for determining the electrochromic and electrochemical characteristics consisted of a light source (5,500 K, China), a transparent cell with an electrolyte (0.1 M KOH), a recording photoresistor, an electronic potentiostat (Elins R-8, Russia), an ADC (E-154, Russia) and a stabilized power supply. The operating principle of the setup was considered in [28].

The specific capacitance of the anode and cathode processes was determined by integrating the cyclic current-voltage curves over time using the trapezoidal method.

4. 2. Morphology and visual evaluation of electrodes

The morphology of the films was studied using a scanning electron microscope (REM-106I, Ukraine), as well as an optical microscope (OSEELANG, China) and a digital eyepiece (Belona, China) with software that made it possible to take photographs and determine particle sizes. Measurements were made at maximum magnification using immersion oil for optical measurements (Turkey).

In addition, photographs of the electrodes with films were taken to evaluate changes during sulfurizing.

5. Results of the electrochemical and electrochromic characteristics of electrochromic electrode deposits before and after sulfurizing

5. 1. Results of sulfurizing, study of changes in the morphology and optical properties of the sulfurized electrode

Fig. 1 shows photographs of two electrodes – the first one with the film obtained without sulfide treatment, the second one with the film that was treated in a sodium sulfide solution. As can be seen from the given photographs (Fig. 1), treatment affected the color of the electrode. Most likely, the color change is associated with the passage of the following solid-phase reaction (1):

$$Ni(OH)_2+S^{2-}\rightarrow NiS+2OH^-. \quad (1)$$

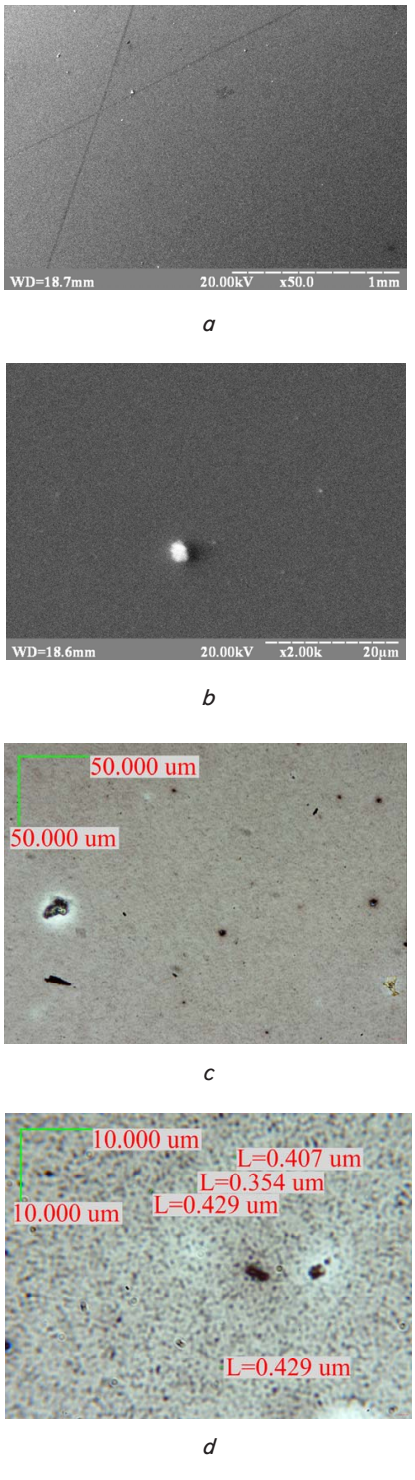
a *b*

Fig. 1. Photographs of the electrodes: *a* – after obtaining the electrochromic coating; *b* – after sulfurizing the resulting electrochromic coating

This assumption is because the film on the electrode acquired a gray color, which is most likely due to the formation of a thin layer of nickel sulfide on the nickel hydroxide surface. It should be noted that pure NiS forms black crystals.

To understand the effect of such treatment on the morphology of the sample treated with sulfide, photographs were taken with an electron and optical microscope (Fig. 2, *a*, *b*).

An analysis of the electron microscopy of the film surface showed that the films were homogeneous, relatively flat, and differed little from similar untreated films [29].



a

b

c

d

Fig. 2. Micrographs of an electrode with an electrochromic film after sulfurizing: *a*, *b* – electron microscopy; *c*, *d* – optical microscopy

Optical microscopy, in turn, showed the presence of more intensely colored clusters. In this case, the size of these clusters was less than 430 nm (0.43 μm) (Fig. 2, *b*, *c*).

5. 2. Study of electrochemical and electrochromic properties of electrodes

To adequately compare the effect of sulfide treatment, the same electrode after cycling in the described mode was treated in a sulfide solution, rinsed in distilled water, and cycled again. Cyclic voltammetry curves (CVA) for the electrode before and after treatment are shown in Fig. 3. For the untreated electrode, the anodic peak corresponded to the passage of the direct reaction (2) and (3), and the cathodic peak corresponded to the passage of the same reaction in the opposite direction.

$$Ni(OH)_2 \rightarrow NiOOH + H^+ + e^-. \quad (2)$$

$$Ni(OH)_2 + OH^- \rightarrow NiOOH + H_2O + e^-. \quad (3)$$

Since, in parallel with the electrochemical cycling of the films, changes in their transparency (*T*, %) were recorded, then the characteristics shown in Fig. 4 can be used to judge changes in the electrochromic properties of the film. Two important features that distinguish the behavior of the films before and after treatment should be noted. The first feature is that the sulfide-treated electrode had less than 100 % transparency and was first bleached before coloring (Fig. 4, *d*), which did not occur for the untreated electrode (Fig. 4, *b*). It is interesting that the indicated behavior of the sulfide-treated electrode was observed only once and exactly corresponded in time to the appearance of a small anodic peak in the first CVA cycle, which is marked with a red square (Fig. 3, *b*).

The second difference in the behavior of the electrodes is the lower degree of coloring of the electrode after treatment (Fig. 4, *c*) compared with the same electrode after treatment (Fig. 4, *a*). Here, the coloration depth means the difference between the transparencies in the fully bleached and colored states of the electrode at each bleaching-coloring cycle [29].

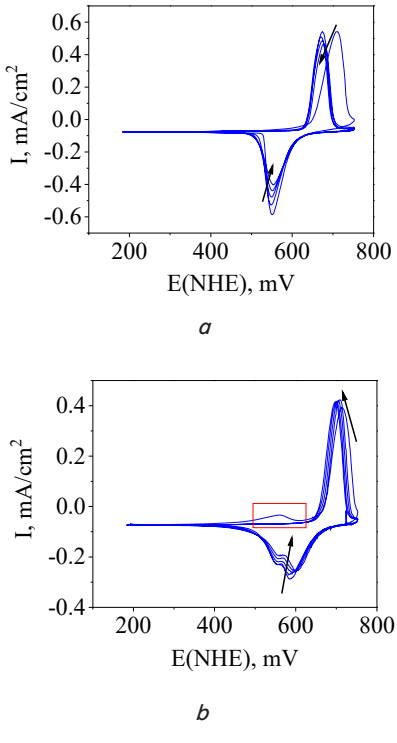


Fig. 3. Cyclic voltammetry curves of the electrodes (arrows indicate changes in the position and height of the peaks during cycling): *a* — untreated electrochromic coating; *b* — after sulfurizing the resulting electrochromic coating

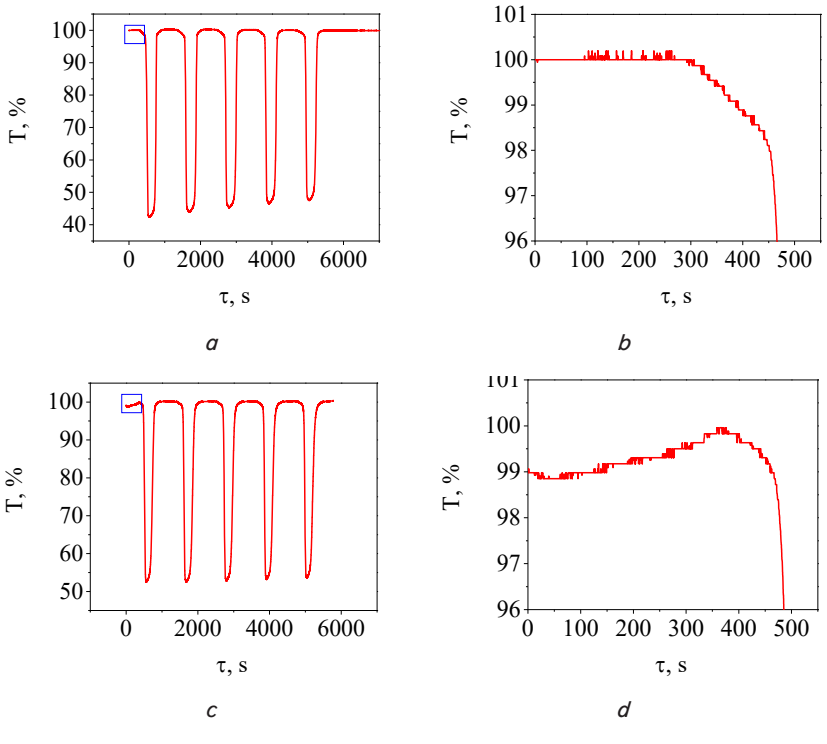


Fig. 4. Curves of changes in the transparency of the electrodes:
a — untreated electrochromic coating;
b — enlarged initial region of the curve for the untreated electrochromic coating;
c — after sulfurizing of the resulting electrochromic coating;
d — enlarged initial region of the curve for electrochromic coating after sulfurizing

In this case, there was a slight decrease in height and position during cycling, which is shown by arrows.

For the electrode after treatment, the picture changed dramatically – a small anodic peak appeared in the first cycle, which disappeared in subsequent cycles. At the same time, two peaks of different intensities appeared on the cathode part of the curve. The given signs for the electrode after sulfide treatment unambiguously indicate a change in the composition, and thus, the electrochemical properties of the film.

It should be noted that, in addition to changes in the coloration depth, the shape of the coloring-bleaching curve of the electrode before and after treatment somewhat changed.

6. Discussion of research results of characteristics of untreated and sulfide-treated electrochromic electrodes

Analyzing the data of optical microscopy, we could draw a preliminary conclusion about the sizes of nickel hydroxide

clusters in the composite electrochromic film polyvinyl alcohol – nickel hydroxide. Approximately, these clusters were about 400 nm in size, which corresponded to the lower limit of the wavelength of visible light. It was possible to determine these dimensions after the film treatment since the nickel sulfide deposit on the surface of nickel hydroxide was indicated by the color where the boundaries of the clusters were located.

The color change, according to our assumptions, is associated with the formation of nickel (II) sulfide. According to the authors, the $Ni(OH)_2 \rightarrow NiS$ solid-state transition became possible due to the difference in the solubility product (SP) of the nickel hydroxide deposit $2 \cdot 10^{-15} - 6.3 \cdot 10^{-18}$ [30] and α, β, and γ of modifications of NiS $3.2\ 10^{-19}$, $1 \cdot 10^{-24}$, $2.0 \cdot 10^{-26}$ respectively. In this case, any of the modifications can be formed since the SP of the hydroxide is at least an order of magnitude greater than any of the sulfide forms.

It should be noted that an electrode with a nickel hydroxide film and treatment in sulfide greater than that used in this series of experiments (10 min) did not lead to a greater coloration of the electrode. This may mean that a very thin and dense sulfide film was immediately formed on the surface of the hydroxide, which prevented the further process of the transition of $Ni(OH)_2$ to NiS. At the same time, the formation of sulfide was indirectly confirmed by both electrochemical and optical characteristics.

The electrode after sulfide treatment had a small anodic oxidation peak only in the first cycle (Fig. 3, *b*) and, at the same time, the film was clarified (Fig. 4, *c*, *d*). It is assumed that at this moment the oxidation of NiS (S^{2-}) occurred and, at the same time, the gray color at the electrode disappeared (the electrode became more transparent due to the disappearance of black NiS):

$$NiS + 2OH^- \rightarrow Ni(OH)_2 + S\downarrow + 2e^-. \quad (4)$$

After a one-time transformation of NiS (black) into $Ni(OH)_2$ (transparent), the process did not repeat in subsequent cycles. The possibility of this process is because the sulfide ion can easily be oxidized – opposite process to reaction (5).

$$S\downarrow + 2e^- \rightarrow S^{2-} (E \equiv = -0.48\ V). \quad (5)$$

In turn, nickel hydroxide obtained as a result of reaction (4) seems to have characteristics different from the original one, which suggests different electrochemical properties. This may explain the appearance of the second cathodic peak at 600 mV (Fig. 3 *a*, *b*). This is because types of nickel hydroxides being different in composition, structure, and modification have different oxidation and reduction potentials [31].

Despite the formation of nickel sulfide on the surface of the hydroxide in the nickel hydroxide-polyvinyl alcohol composite, the characteristics of the treated electrode deteriorated (Fig. 5). The untreated film had a higher specific capacitance of the anode and cathode processes (Fig. 5, *a*) and a higher average coloration depth (*D*, %). Although, the ratio of anodic to cathodic capacity was higher for the sulfurized film (Fig. 5, *b*).

Obviously, the treatment in sulfide changes the characteristics of the electrochromic film.

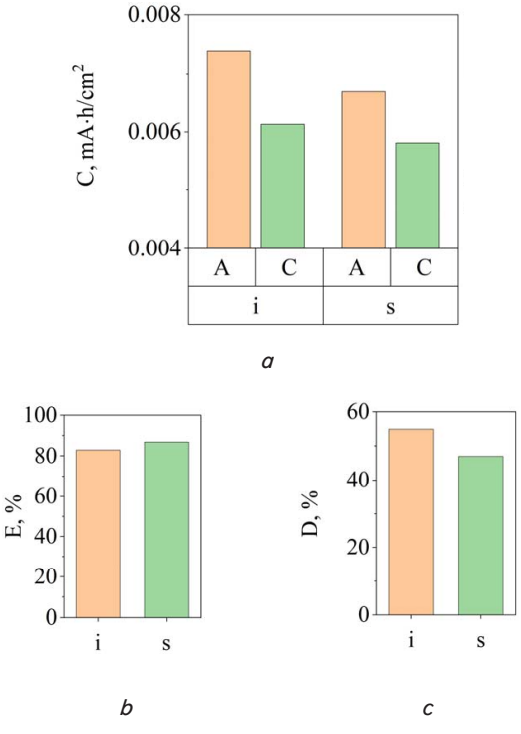


Fig. 5. Generalized characteristics for electrodes: i – without treatment, s – for sulfurized electrode: *a* – specific capacitance of anodic (A) and cathodic (C) processes; *b* – the ratio of anodic to cathodic capacitance of electrodes in the fifth cycle; *c* – average coloration depth for electrodes

Although the method shown does not lead to an improvement in the electrochromic characteristics of the electrode, nevertheless, it can potentially be useful. The presented method can be used to form electrodes effective for the catalytic oxidation of organic substances in fuel cells or the process of wastewater deactivation. Also, this method can be useful in the formation of various sensors [32].

In terms of the development of this research area, it is interesting to use this composite for the formation of water decomposition electrodes for hydrogen synthesis. To do this, the specified composite can be formed on a metal substrate and calcined at high temperatures to obtain mixed oxide-sulfide-metal films with the possible presence of elemental carbon. Such films have already found application in the electrochemical evolution of hydrogen. However, the presence of organic substances can lead to a partial reduction of oxides to metals, which will affect the morphology and structure of the resulting composite. Such features can have a positive effect on the qualitative characteristics of the catalytic properties of the final composite layer on the electrode.

This study has some limitations that can be eliminated in further experiments. These limitations include the lack of experiments related to checking the properties of the formed films for the presence and magnitude of electrocatalytic properties.

7. Conclusions

1. A simple and fast method for sulfurized electrodes containing nickel hydroxide by holding in a 0.03 M Na_2S

solution for 10 minutes has been proposed. This method can potentially be useful for the formation of electrodes for various electrochemical applications.

2. Using the method, the sizes of the nickel hydroxide clusters in the $Ni(OH)_2$-polyvinyl alcohol composite electrochromic film have been approximately determined to be nearly 400 nm.

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