

# DEFINITION OF THE INFLUENCE OF PULSED DEPOSITION MODES ON THE ELECTROCHROMIC PROPERTIES OF Ni(OH)<sub>2</sub>-POLYVINYL ALCOHOL FILMS

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*In this work, the influence of some types of the pulsed deposition mode of electrochromic films from aqueous solutions of nickel nitrate with the addition of polyvinyl alcohol was investigated. Glass coated with a fluorine-doped tin oxide film was used as the basis for deposition. The deposition of nickel (II) hydroxide – polyvinyl alcohol electrochromic films was carried out in three pulsed modes:  $-0.2 \text{ mA/cm}^2 \times 5 \text{ s}$ ,  $0 \text{ mA/cm}^2 \times 5 \text{ s}$  (10 minutes);  $-0.5 \text{ mA/cm}^2 \times 2 \text{ s}$ ,  $0 \text{ mA/cm}^2 \times 8 \text{ s}$  (10 minutes);  $-1 \text{ mA/cm}^2 \times 1 \text{ s}$ ,  $0 \text{ mA/cm}^2 \times 9 \text{ s}$ . In this case, the amount of electricity used for the formation of thin-film electrodes was the same for all samples.*

*The resulting films showed dramatic differences in electrochemical, optical, and quality characteristics. The sample obtained in the mode of the highest cathode current density and the duration of the no-current condition ( $1 \text{ mA/cm}^2 \times 1 \text{ s}$ ,  $0 \text{ mA/cm}^2 \times 9 \text{ s}$ ) had the worst specific capacity and optical characteristics. This sample was characterized by the highest number of coating defects and color non-uniformity as well.*

*The sample, which was obtained at average current densities ( $-0.5 \text{ mA/cm}^2 \times 2 \text{ s}$ ,  $0 \text{ mA/cm}^2 \times 8 \text{ s}$ ), had the highest specific characteristics among the electrodes in the series. The coating was uniform and solid. Also, this sample had the greatest stability of the coloration depth value, which varied from 79.1 to 78.1 % (first to fifth cycles).*

*The sample obtained in the mode  $-0.2 \text{ mA/cm}^2 \times 5 \text{ s}$ ,  $0 \text{ mA/cm}^2 \times 5 \text{ s}$  showed moderate specific indicators, however, there were some coating defects.*

*According to the results obtained, a mechanism was proposed that explained the differences in the characteristics of thin-film electrodes formed in different modes. This mechanism consists of changing the time of non-stationary processes and the distribution of the current density with a change in the value of the deposition current density, the duration of the cathode period, and the no-current condition*

*Keywords: pulsed mode, electrodeposition, electrochromism, composite coating, nickel hydroxide, polyvinyl alcohol*

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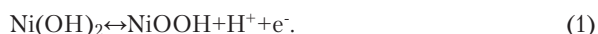
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## 1. Introduction

Nickel oxides and hydroxides are widely used in various devices. The key component of Ni-Cd, Ni-Fe Ni-MH, Ni-Zn, Ni-H<sub>2</sub> alkaline batteries [1, 2] is nickel (II) hydroxide, which is associated with the high reversibility of the electrochemical reaction (1):



Nickel oxides and hydroxides are used in asymmetric hybrid supercapacitors as active substances of one of the electrodes [3, 4]. Nickel oxide and hydroxide are also applicable as an active layer of sensors for acetaminophen [5], folic acid [6], acetone, toluene, n-butyl acetate [7], and other compounds [8].

In addition to the listed areas, oxide [9] and hydroxide [10] compounds have found application in electrochromic executive elements of so-called "smart" windows. Electrochromic cells contain thin films of nickel oxides or hydroxides deposited on electrically conductive transparent substrates. Depending on the polarity and the amount of

electricity, a reversible change in the colors of the electrochromic coating and the element as a whole occurs between the transparent and colored states.

"Smart" windows control the level of illumination in premises and significantly save electricity needed for air conditioning [11].

At the same time, the range of applications is not limited to those indicated above. It is proposed to use electrochromic coatings in the advertising and design industries, in medical institutions, as well as for the preservation of important artifacts (historical documents, books) that can deteriorate under intense light.

The relevance of work within the framework of this topic is connected, on the one hand, with the possibility of significant savings in electrical energy during air conditioning of premises when using "smart" windows. On the other hand, the use of windows with electrochromic elements will reduce the load on the electrical grid during peak periods of consumption (early morning and late evening). An important and urgent task, which is solved when using this class of "smart" devices, is to create a comfortable level of illumination in premises.

**2. Literature review and problem statement**

One of the most problematic issues regarding the widespread introduction of “smart” windows based on electrochromic coatings is their high price. As a rule, it is in the range of 100–1,000 USD/m<sup>2</sup> [12]. This is due to two factors – a small volume of production and a high cost of vacuum deposition methods of the device layers in comparison with other deposition methods [13]. Therefore, a significant reduction in the cost of technology is a key factor for the widespread use of “smart” windows.

In [10], a cathode template method of Ni(OH)<sub>2</sub> deposition was proposed, which is cheap, simple to implement, does not require high-tech equipment and can be easily automated. For this method, special treatment of the substrate [14], post-treatment [15], as well as an approach to the selection of modes [16] were proposed. It is shown in [17] that the modes of electric current supply significantly affect the specific characteristics of the film. However, the question of the influence of current supply modes during deposition remains completely unexplored. For example, pulsed deposition modes of various coatings are widely used. Such modes can significantly change the kinetics of the deposition reaction, and, thus, affect the final quality characteristics of the coatings.

For example, the authors of [18] used direct and pulsed currents to obtain hydroxyapatite (Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>) on stainless steel. It was shown that when using a pulsed current, the adhesion and crystallinity of the resulting coatings for medical steel increased significantly.

The Cu-Ni alloy was deposited electrochemically using pulsed modes [19]. The researchers found that the film composition, the lattice constant and the magnetic properties of the films can be controlled by changing deposition modes and current density.

In [20], the process of forming transparent conducting electrodes based on copper nanowires coated with nickel in pulsed modes was carried out. The group showed that different modes of pulsed deposition of nickel over a copper nanowire mesh significantly changed the characteristics of the resulting coating.

Researchers in [21] noted that iridium oxide, which was deposited in a pulsed mode for use in a polymer electrolyte electrolyzer, had high catalytic characteristics.

It was also shown that nickel hydroxide formed on electrodes for alkaline batteries in a pulsed mode showed higher specific characteristics than those of hydroxide deposited in a DC mode [22].

In addition, the study [23] showed that pulsed deposition of polythiophene films as electrochromic coatings gave better results compared to films deposited at constant potentials. The films obtained in pulsed modes had a greater coloration depth, a faster rate of changes in color characteristics, and a better coloring efficiency.

Analysis of the above works shows that the use of pulsed modes can potentially improve the characteristics of electrochromic deposition.

**3. The aim and objectives of the study**

The study aimed to determine the effect of deposition of composite Ni(OH)<sub>2</sub>-PVA films in pulsed modes on their

electrochemical and electrochromic characteristics. The information obtained in this study will optimize the process of obtaining films, as well as improve their quality.

To achieve the formulated aim, it was necessary to accomplish the following objectives:

- to deposit Ni(OH)<sub>2</sub>-PVA films by the cathode template method in pulse modes;
- to carry out a comparative analysis of the electrochemical, electrochromic, and qualitative properties of the films.

**4. Research materials and methods**

*Obtaining composite Ni(OH)<sub>2</sub>-PVA films.*

All composite electrochromic Ni(OH)<sub>2</sub>-PVA coatings were obtained on 0.9 mm thick glass coated with an electrically conductive transparent SnO<sub>2</sub> coating doped with F (FTO glass): R<10 Ohm/sq., Zhuhai Kaivo Optoelectronic Technology Co. Ltd. (China).

The preparation of the substrate for the coating included degreasing and wiping with a mixture of Na<sub>2</sub>CO<sub>3</sub> (technical grade) with the addition of distilled water. This was followed by sequential rinsing with running and distilled water, as well as with ultrasonic radiation treatment in 96 % ethanol: duration – 10 minutes, power 60 W, radiation frequency 41,500 Hz. After that, the glass was dried, and immediately before electrodeposition was wiped with a lint-free cloth soaked in C<sub>2</sub>H<sub>5</sub>OH. The coating area was 4 cm<sup>2</sup> (2 by 2 cm square).

The Ni(OH)<sub>2</sub>-PVA deposits were formed by the cathode template method in three selected pulse modes. Application conditions are given in Table 1.

Table 1

Modes of electrochromic coating application

Sample designation	Pulses of current	No-current condition	Deposition time	Deposition capacity	Electrolyte composition
S	-0.2 mA/cm <sup>2</sup> ×5 s	5 s	600 s	60 mA·s/cm <sup>2</sup>	0.01 M Ni(NO <sub>3</sub> ) <sub>2</sub> , 4 % PVA*
M	-0.5 mA /cm <sup>2</sup> ×2 s	8 s			
L	-1 mA /cm <sup>2</sup> ×1 s	9 s			

Note:\* PVA – polyvinyl alcohol

After the coating was formed, the electrode was rinsed from the electrolyte in warm distilled water. The procedure of drying the obtained films was carried out at room temperature for one day.

*Electrochemical and optical tests.*

To assess the electrochemical and optical characteristics, we used the setup shown in Fig. 1.

The electrochemical characteristics were assessed by the method of potentiodynamic cycling according to the three-electrode scheme in the [+201; +751 mV] mode relative to the NHE at v=1 mV/s [24]. A silver chloride electrode in saturated potassium chloride was used as a reference electrode. The cell was made from transparent PMMA. During cycling, changes in film transparency were also recorded in parallel with the electrochemical characteristics of the electrodes.

*Visual assessment of electrodes.*

To compare the quality of the films after all tests, the electrodes were colored by turning the potential of the film electrode from +201 to +751 mV relative to the NHE at v=1 mV/s. Then the photographs were taken from different angles.

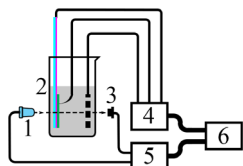


Fig. 1. Schematic of the setup for studying the electrochemical and optical characteristics of electrochromic films:  
 1 – LED (5,500 K); 2 – cell with electrolyte (0.1 M KOH);  
 3 – photoresistance; 4 – electronic potentiostat (Elins P-8);  
 5 – ADC (E-154) and power supply unit of the light source;  
 6 – computer; ■ – glass; ■ – electrically conductive coating; ■ – composite electrochromic Ni(OH)<sub>2</sub>-PVA coating

## 5. Results of studying the characteristics of thin-film electrodes formed in pulsed modes

### 5.1. Electrochemical and optical characteristics of electrodes

Fig. 2–4 show the characteristics of thin-film electrodes obtained in pulsed modes according to Table 1.

Analysis of the obtained characteristics revealed significant differences in electrochemical behavior, as well as in optical and quality characteristics.

The cyclic voltammogram (CVA) of sample *S* showed the presence of an oxidation peak in the first cycle, shifted to a more positive side as compared to the same peak at 2–4 cycles (Fig. 2, *a*). In this case, the height and position of the oxidation peak stabilized at 2–4 cycles. The position of the peak after stabilization was equal to 688 mV, which significantly differed from the position of the corresponding peak of sample *M* (727 mV) and *L* (702 mV) in Fig. 3, 4, *a*. It should be noted that the anode peak corresponded to the transition of Ni(OH)<sub>2</sub> to NiOOH.

In all CVAs of the samples, the current density of the cathode peak decreased from cycle to cycle. The sharpest decrease was observed for sample *L*. The position of the peaks for different samples was close to 570 (*S*), 586 (*M*), and 581 (*L*) mV.

The height of the peaks, which semi-quantitatively determines the capacity used for the oxidation and reduction processes, is different for all samples. Obviously, these differences reflect the differences in the films, which, in turn, are determined by the production modes.

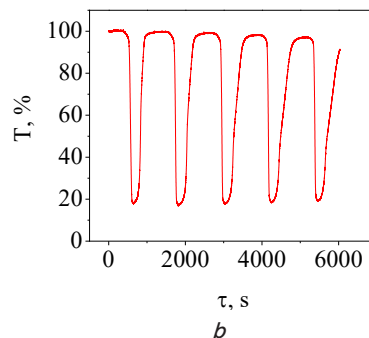
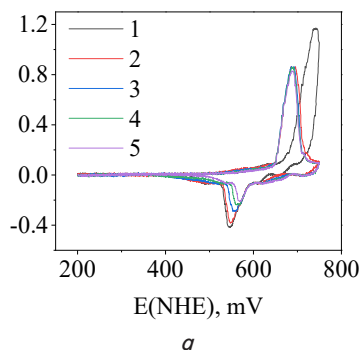


Fig. 2. Characteristics of sample *S*:  
*a* – cyclic voltammogram;  
*b* – coloration-bleaching curve;  
*c* – photo of the colored electrode after cycling

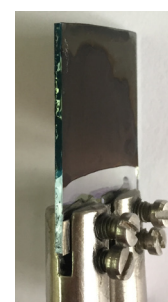
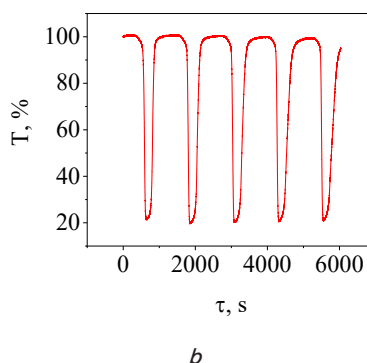
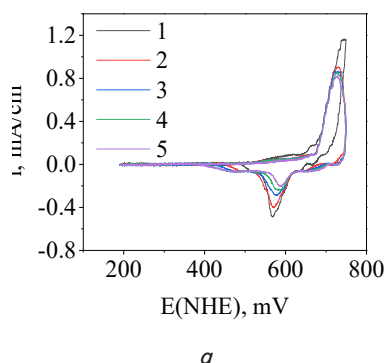


Fig. 3. Characteristics of sample *M*: *a* – cyclic voltammogram; *b* – coloration-bleaching curve;  
*c* – photo of the colored electrode after cycling

Changes in transparency ( $T$ ) during cycling in the potentiodynamic mode also differ significantly in the samples. Thus, the best shape of the curve is for sample  $M$ , which is closest to a rectangular shape. The coloration depth as the difference between  $T$  in the colored and bleached states in the first→fifth cycles for samples  $S, M, L$ , is 82.1→77.8 %, 79.1→78.1 %, and 80.9→2.55 %, respectively. In turn, the transparency at the fifth cycle in the maximum bleached state for samples  $S, M, L$  equals 97.1 %, 99.3 %, and 62.9 %. Analysis of the given values suggests that sample  $M$  demonstrates the most stable characteristics since it has the smallest change in the coloration depth over five cycles and the highest transparency in the bleached state during cycling.

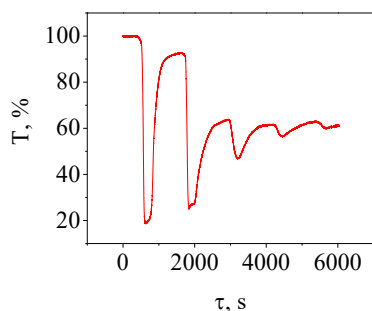
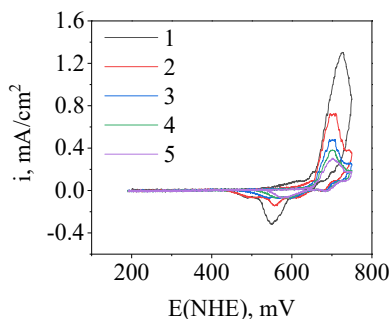


Fig. 4. Characteristics of sample  $L$ :  $a$  – cyclic voltammogram;  $b$  – coloration-bleaching curve;  $c$  – photo of the colored electrode after cycling

Analysis and comparison of photographs of the electrodes in the colored state suggest that the quality of the electrochromic coating critically depends on the mode of its formation. Thus, sample  $M$  has the best appearance – there are no cracks, there are no uncoated places, the entire surface has a uniform color. At the same time, samples  $S$  and  $L$  contain defects, and sample  $L$  has a non-uniform film color.

### 5.2. Comparison of the capacity characteristics of electrodes formed in pulsed modes

Fig. 5 shows the average specific capacities of the anode and cathode processes, determined from the cyclic current-voltage curves. The determination was carried out by the method of numerical integration of the areas of the anode and cathode processes.

The given capacity values illustrate that sample  $M$  has the highest capacities, in which both the anode and cathode capacities are the highest among the samples.

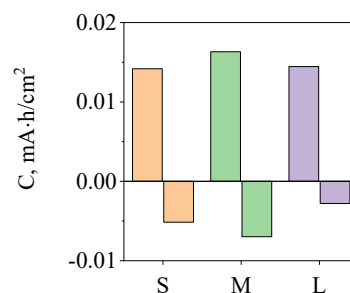


Fig. 5. Average specific capacities of anode and cathode processes for samples  $S, M, L$

The anode capacity of sample  $S$  is almost equal to the anode capacity of sample  $L$ , but the cathode capacity is almost twice as large.

Thus, we can say that these capacities correspond to the analysis of the optical characteristics of the films, which showed the best characteristics for sample  $M$ .

### 6. Discussion of the characteristics of film electrodes deposited in pulse modes

The data presented in Fig. 2–5 clearly illustrate the dependence of the characteristics of  $\text{Ni}(\text{OH})_2$ -PVA films on the conditions of electrodeposition. It is obvious that the applied modes affect the deposition kinetics, and, thus, the deposition structure. The latter, in turn, determines the final properties of electrochromic coatings.

For obtaining these films, the direct current (galvanostatic) production mode is traditional [25], which determines the establishment of a stationary mode after the start of deposition and the initial non-stationary process. An obvious advantage of direct current deposition modes is the constancy of deposition conditions during almost the entire process. Galvanostatic mode provides constant and uniform mass exchange in the reaction zone, supply of reagents, and removal of products. When the current supply mode is switched from direct current to pulsed, the total amount of time of non-stationary processes increases in proportion to the number of pulses, which radically changes the reaction kinetics. In this case, a change in the kinetics leads to a change in the properties of the resulting deposition.

However, high current densities lead to non-uniform distribution of current density over the sample area. Usually, this pattern is not typical for deposition on metal substrates, since their resistance is relatively low compared to that of the electrolyte. However, as it was shown in [16], on electrodes based on low-conductive materials, the non-uniformity of the current distribution is typical and increases with increasing current density. This fact explains the difference

in color for sample *L*, which was obtained at the maximum current density in the series (pulse at 1 mA/cm<sup>2</sup>).

The qualitative and quantitative differences between the films are associated with several processes:

- the rate of production of OH<sup>-</sup> ions on the electrode surface under the electrochemical reaction (2);
- diffusion of ions to (from) the electrode, which is determined by the concentration gradient at a given time;
- the rate of formation of Ni(OH)<sub>2</sub> by chemical reaction (3).



The three listed processes will depend on the deposition current density, the duration of the current pulse, and the no-current condition.

It should be noted that these studies did not consider a significant increase in pulse frequency, the use of pulses with opposite polarity to the main current pulses (i.e. positive). Also, within the framework of this study, the influence of the shape of the supplied pulses was not determined, which is a question of additional research.

An important task for further research is to determine the dependence of the structure on the pulsed deposition mode. However, taking into account the thickness of the deposited films (300 nm) and the thickness of the electrically conductive substrate (≈500 nm) [14], this task is laborious and requires special experiments.

In addition, we can say that the structure of samples *S* and *L*, determined by the deposition mode, most likely has stresses, which are expressed in the formation of visible cracks.

## 7. Conclusions

1. As a result of a series of experiments, three films have been deposited and investigated in the following modes:

–0.2 mA/cm<sup>2</sup>×5 s, 0 mA/cm<sup>2</sup>×5 s (10 minutes);

–0.5 mA/cm<sup>2</sup>×2 s, 0 mA/cm<sup>2</sup>×8 s (10 minutes);

–1 mA/cm<sup>2</sup>×1 s, 0 mA/cm<sup>2</sup>×9 s.

The resulting electrochromic films have demonstrated specific characteristics that significantly differed in specific capacities used for the cathode and anode processes: sample *S* 0.014 and –0.0052 mA•h/cm<sup>2</sup>; sample *M* 0.016 and –0.0070 mA•h/cm<sup>2</sup>; sample *L* 0.014 and –0.0028 mA•h/cm<sup>2</sup>.

2. The best characteristics have been shown by the film obtained in the pulsed mode –0.5 mA/cm<sup>2</sup>×2 s, 0 mA/cm<sup>2</sup>×8 s (10 minutes). At the same time, the film has the minimum change in the coloration depth among the samples from 79.1 to 78.1 %, the maximum specific capacities and uniform color in the colored state.

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