

A STUDY OF PHYSICO-CHEMICAL CHARACTERISTICS OF ELECTROCHROMIC $\text{Ni}(\text{OH})_2$ -PVA FILMS ON FTO GLASS WITH DIFFERENT DEPOSITION DURATION

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The use of electrochromic elements in "smart" windows leads to significant savings in electricity required for cooling premises. However, the high cost of these devices does not allow the technology to be widely used. Since the cost is determined by costly vacuum deposition methods, the development of other cheaper methods of deposition of electrochromic element layers is urgent.

Aspects of alternative to vacuum formation methods – cathode template electrochemical deposition of composite electrochromic $\text{Ni}(\text{OH})_2$ -PVA films were investigated.

The study is devoted to determining the effect of the duration of deposition of the electrochromic layer on their physicochemical characteristics, in particular, on the optical and electrochemical properties. The deposition was carried out on fluorine-doped tin oxide glasses (FTO glasses). The time of deposition was chosen equal to 5, 10, 20, 40, 60, and 80 minutes.

As a result of the experiments, it was shown that the optimal duration of deposition under the selected conditions of the electrochromic layer formation was the interval from 5 to 20 minutes, inclusive. The deposition time of 40 minutes did not improve the optical characteristics of the film. At the same time, with the deposition duration of 60 and 80 minutes, the electrochemical and optical parameters sharply decreased, the coloration depth and irreversibility during bleaching, as well as the specific capacitances of the processes decreased.

In the course of data processing, the film thickness was calculated depending on the duration of deposition in several ways. Comparison of the graphs obtained made it possible to determine the approximate amount of polyvinyl alcohol in the electrochromic composite coating, as well as to estimate the current efficiency of the electrodeposition and oxidation-reduction process of the electrochromic material. In this case, the volume of polyvinyl alcohol in the composite was approximately equal to the volume of nickel hydroxide, and the efficiency of $\text{Ni}(\text{OH})_2$ deposition and coloration-bleaching processes was approximately 100 %

Keywords: electrochromism, electrodeposition, nickel hydroxide, polyvinyl alcohol, deposition duration, coating thickness, adhesion

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1. Introduction

Electrochromism, the phenomenon of color change of substances under the influence of electric current, has been studied

for a long time [1, 2]. However, only relatively recently, electrochromic devices began to be widely used for the manufacture of "smart windows" for buildings [3], as an adjustable car tinting [4], for shading windows in aviation [5]. At the same time, de-

spite the obvious benefits in improving visual comfort, the main advantage of the technology is the possibility of significant savings in resources for cooling premises. So, in [6] it is noted that the use of electrochromic “smart windows” can lead to energy savings during air conditioning up to 49 %, reduce the load on the power grid during “peak” periods of consumption up to 16 % (morning and evening). There is also an opportunity to reduce lighting costs by up to 51 %. Considering the sharp increase in average temperatures on the planet over the past decades, the need for this technology has increased significantly.

However, the high cost of the electrochromic elements produced does not allow extensive use of available technologies. So, with a price per square meter within 400–800 \$ [7], the use of ready-made solutions is economically unjustified.

One of the main reasons for the high cost of final devices is the use of vacuum deposition methods [8, 9]. An alternative can be different variations of the electrochemical deposition method, which are relatively simple, do not require expensive equipment, and are easily automated [10, 11].

Thus, studies aimed at reducing the cost of electrochromic technologies, including the development of new and improving classical methods for forming electrochromic coatings, are relevant and in demand.

2. Literature review and problem statement

Nickel oxide and hydroxide compounds are among the promising electrochromic materials. These films can be formed by vacuum methods, electrochemical, and some other methods. For example, in [12], NiO films, and in [13], non-stoichiometric NiO_x films were obtained by vacuum methods. The electrochemical production of nickel hydroxides was shown for films with Zn, Co, and Al additives in [14] and without additives in [15]. The sol-gel method was used in [16], and chemical deposition in the bulk of a solution was used by researchers in [17] to obtain electrochromic nickel oxide films.

Films deposited in different ways have different, and often very different characteristics. It should be noted that as a result of using electrochemical deposition methods, especially from aqueous solutions, nickel hydroxide is mainly formed. If researchers process the obtained Ni(OH)_2 films after deposition at high temperatures (above 240–300 °C), this leads to the formation of stoichiometric and non-stoichiometric NiO_x oxides. The latter also have electrochromic properties.

In terms of theory and practice, it is necessary to have data on the operating parameters and limitations for films that are applied by electrochemical methods. It is important to know how thick the films can be deposited, how the depth of penetration of the electrochemical process and the optical properties of the films change depending on the duration of deposition. It should be noted that under Faraday's law, the process duration will be proportional to the amount of new substance that is formed on the electrode. On a flat electrode, this will mean that generally the duration of electrochemical deposition will be proportional to the film thickness.

For example, in [18], nickel oxide films were obtained electrochemically in a potentiodynamic mode from aqueous solutions of nickel chloride for 20, 30, 40, 50 minutes. The researchers showed that with an increase in deposition duration above 30 minutes, stresses appeared that led to the formation of cracks. In this case, the electrode formed within 20 minutes (thickness 104 nm) showed high stability, no cracks, and the number of bleaching-coloring cycles on it was 10.000.

In [19], the researchers formed electrochromic NiO films by spray pyrolysis on the surface of ITO and FTO glass using a nickel chloride solution for 20–140 s. As a result, it was shown that the thickness of the films corresponded to the thickness range of 50–400 nm. In this case, the film thickness significantly affected the optical characteristics. For example, films with a thickness of less than 120 nm were highly transparent, but could not be completely colored. Films with a thickness of more than 200 nm showed a deeper color but were less transparent in the discolored state. At the same time, the optimal thickness value was in the range 140–150 nm, since films of such thicknesses gave the highest optical modulation in both the visible and solar spectra.

A group of researchers in [20] obtained electrochromic Ni(OH)_2 films during cathodic polarization from solutions containing 0.01 M $\text{Ni(NO}_3)_2$ at a current density of 0.04 mA/cm² and a deposition time of 2–8 min. One of the conclusions of the researchers was that the absorption of light by an oxidized film was proportional to the deposition time (thickness) of the film.

In [21], NiO films deposited by the chemical method from a nickel sulfate solution with additives were studied. The time of deposition was: 10, 20, 30, 40, 50, and 60 minutes. The researchers found that the energy of the optical band gap decreased from 3.22 to 2.80 eV with increasing film thickness, and also confirmed that the modulation of light by films increased with increasing film thickness.

Similar conclusions as in [21] regarding the effect of coating thickness on optical characteristics were made in [22]. The experiments used 170–540 nm NiO films, deposited by electron beam physical vapor deposition.

In this work, it was shown that thicker films had a well-developed crystal structure and crystallite size, which provided greater light modulation and durability. At the same time, the deposition of thinner films with a thickness of 170 nm led to a shorter response time with a change in color and a coloring efficiency of 53.1 C/cm². It was also found that thicker films had a greater amount of intercalated charge than thinner ones, but the electrochromic properties did not increase proportionally. The change in the thickness of electrochromic NiO films depends on the conditions and methods of film preparation, as well as on the electrolytes used.

In [23], a study of the dependence of the properties of electrochromic films obtained by the cathodic template method on deposition time was described. However, the study used substrates of another type – opaque polished nickel, while the concentration of nickel nitrate was 1 M.

In other studies, for a transparent substrate (fluorine-doped tin oxide glass, FTO glass), it was proposed to use a 0.01 M nickel nitrate solution with the addition of polyvinyl alcohol and a cathode current density of 0.1 mA/cm² [24].

It should be noted that substrate type [25], reagent concentration, and electrodeposition current density [26] play a critical role in the production of deposits. However, no studies have been found in the literature for the deposition conditions indicated in [24] regarding the deposition duration and its effect on the optical and electrochemical properties.

3. The aim and objectives of the study

The study aimed to determine the effect of the deposition duration of electrochromic Ni(OH)_2 -PVA films on their electrochromic and electrochemical characteristics. The

solution of the aim will allow optimizing the thickness and, accordingly, quality characteristics of the resulting films.

Within the framework of this aim, the following research objective was formulated:

– to determine the dependence of the electrochromic (optical) and electrochemical characteristics of films obtained by the cathodic template method on substrates with a finished electrically conductive layer – F (FTO glass)-doped SnO_2 on the duration of deposition in the range of 5–80 minutes.

4. Research materials and methods

4.1. Deposition of $\text{Ni}(\text{OH})_2$ -PVA electrochromic films

All films were obtained under the same conditions. The only difference was the deposition duration of electrochromic coatings. $\text{Ni}(\text{OH})_2$ -PVA composite coatings were obtained on 0.9 mm thick FTO glass. The surface resistance of the FTO glass was $\Omega_{\text{sp}} \leq 10 \text{ Ohm/sq.}$ (China, Zhuhai Kaivo Optoelectronic Technology Co. Ltd.).

The preparation of the substrate for coating included the removal of contaminants from the electrically conductive surface in several stages. First, degreasing and wiping with a paste of soda ash and water was carried out. This was followed by sequential rinsing with running and distilled water, as well as treatment in 96 % ethanol under ultrasound for 10 minutes (60 W, $f=41,500 \text{ kHz}$). Then the substrate was dried and, before the coating formation, it was wiped with a lint-free cloth soaked in ethanol. The coated area of the electrodes was 4 cm^2 .

The $\text{Ni}(\text{OH})_2$ -PVA deposits were formed by the cathodic template method in the following mode:

- 0.01 M $\text{Ni}(\text{NO}_3)_2$, 5 % PVA solution;
- deposition on the cathode with a current density of 0.1 mA/cm^2 ;
- duration of deposition 5, 10, 20, 40, 60, 80 minutes.

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

4.2. Electrochromic and electrochemical characteristics

Electrochromic and electrochemical characteristics were determined on the setup. The setup consisted of a light source (5,500 K, China), a transparent cell with an electrolyte (0.1 M KOH), recording photoresistors, an electronic potentiostat (Elins R-8, Russia), an ADC (E-154, Russia), and a stabilized power supply unit. The setup worked as follows. When the potential set using a potentiostat was changed, the color of the electrode changed. At the same time, the light passed through it, which fell on the photoresistor. The change in the electrode transparency affected the light flux falling on the photoresistors and, as a consequence, changed its resistance, as well as the voltage drop across it. In turn, the voltage drop across the photoresistor was recorded by the program supplied with the ADC [27].

The electrochemical characteristics were evaluated using cyclic volt-ampere curves using a three-electrode scheme in the $[+201; +751 \text{ mV}]$ mode relative to the NHE at $v=1 \text{ mV/s}$. 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. During cycling, in parallel with the

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

The specific capacity of the anodic and cathodic processes was determined by numerically integrating the cyclic volt-ampere curves over time by the trapezoidal method. Average specific capacities were calculated as arithmetic mean values over five cycles.

4.3. Visual assessment of electrodes

To compare the quality of the films after all tests, the electrodes were colored electrochemically, sweeping the electrode potential from +201 to +751 mV relative to the NHE with a sweep rate of 1 mV/s . Then, the photographs were taken from different angles.

5. Results of studying the characteristics of thin-film electrodes deposited with different durations

Fig. 1–6 show the results of electrochemical tests and the obtained dependences of transparency (T) on time. All curves consist of two or three characteristic zones; two peaks (anodic and cathodic), as well as a rise in current after the anodic peak at more positive potentials. The anodic peak characterizes the oxidation of $\text{Ni}(\text{OH})_2$ to NiOOH , where the latter is colored dark brown. The rise in current more positively than the anodic peak (Fig. 1, *a*) characterizes the anodic release of oxygen at the electrode. This process is generally undesirable and depends on the electrode material, potential sweep rate, and other parameters. The cathodic peak characterizes the reverse process to the anodic one, i.e., the reduction of NiOOH to $\text{Ni}(\text{OH})_2$, and manifests itself as electrode bleaching, since $\text{Ni}(\text{OH})_2$ is transparent in thin films.

In some cases, a clear cathodic peak was not observed, as well as the rise associated with oxygen release. This is due to the increased polarization of the oxidation process of $\text{Ni}(\text{OH})_2$ to NiOOH , which led to a shift of the peak to a more positive region (a part of the peak was observed in the form of a rise). In such cases, the peak current is summed up with the current used for oxygen evolution. At the same time, the curve looks like a steep rise (sometimes with a stop in the middle of the rise).

During the potentiodynamic cycling of the electrode (cyclic voltammogram, CVA), the optical characteristic of the electrode was also recorded – transparency ($T, \%$). To assess the optical properties of the electrode, the shape of the transparency change curve was evaluated, as well as two characteristics determined from it. First of all, the coloration depth ($D, \%$) was assessed, which characterized the maximum change in transparency on a given coloring-bleaching cycle (Fig. 1, *b*). Also important for the assessment was the characteristic of the electrode irreversibility during bleaching ($I, \%$), which was defined as the difference between 100 % transparency and the bleached state of the electrode (Fig. 1, *b*). This characteristic shows how much the electrode loses its ability to return to the original transparency.

An analysis of the obtained curves qualitatively made it possible to distinguish several effects that manifested themselves with an increase in the duration of the film deposition process.

One of the effects was an increase in the current densities on the CVA with an increase in deposition duration. This is because with large electrodeposition durations, the thickness of the deposited film increases according to Faraday's

law. Accordingly, with a potentiodynamic potential sweep, high currents can be obtained on thicker films.

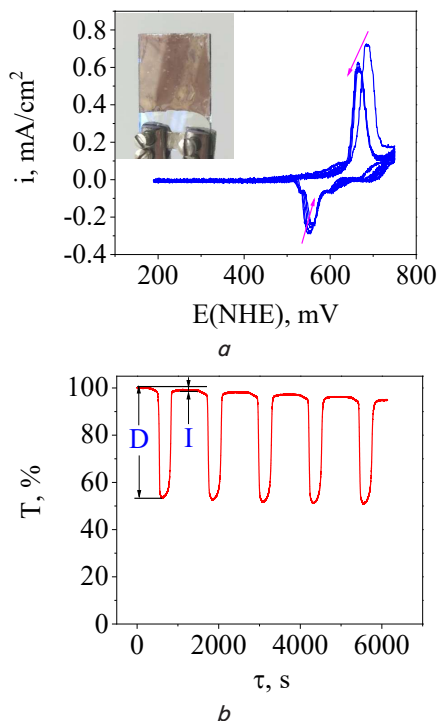


Fig. 1. Characteristics of a thin-film electrochromic electrode formed within 5 minutes: *a* – cyclic voltammogram with an inserted photograph of the electrode in a colored state; *b* – curve of transparency change during cyclin

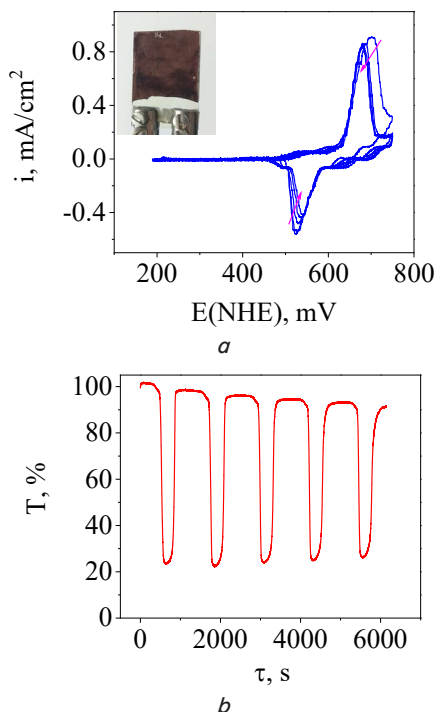


Fig. 2. Characteristics of a thin-film electrochromic electrode formed within 10 minutes: *a* – cyclic voltammogram with an inserted photograph of the electrode in a colored state; *b* – curve of transparency change during cycling

An interesting effect of increasing the duration of deposition was the change in the shape of the curves, from sharp

high peaks to “softer” shapes with diffuse peaks. Since other electrolysis parameters did not change, such a change may indicate diffusion restrictions associated with the movement of H^+ within the solid phase.

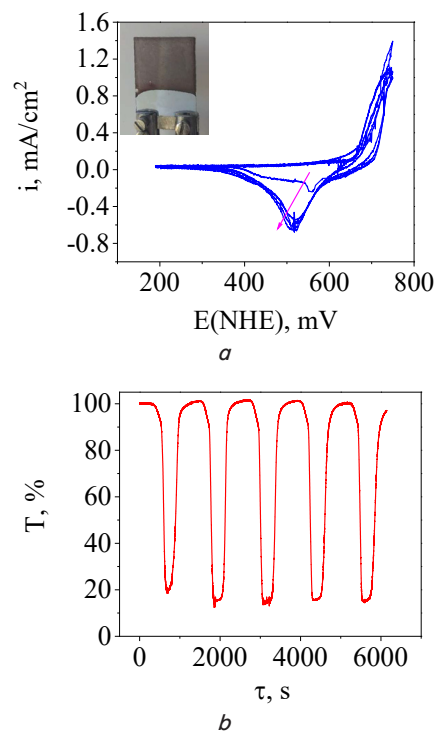


Fig. 3. Characteristics of a thin-film electrochromic electrode formed within 20 minutes: *a* – cyclic voltammogram with an inserted photograph of the electrode in a colored state; *b* – curve of transparency change during cycling

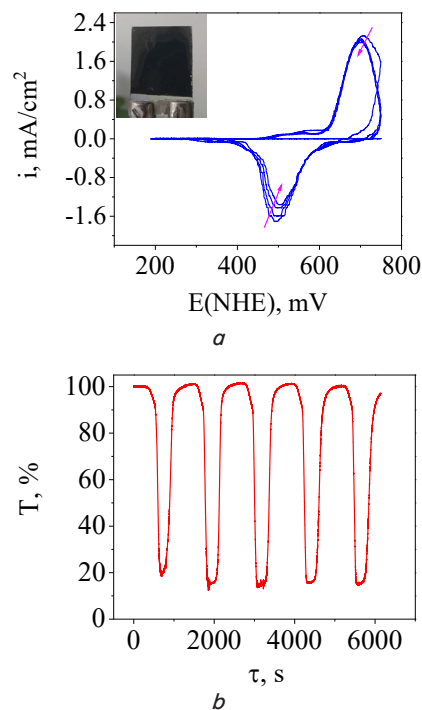


Fig. 4. Characteristics of a thin-film electrochromic electrode formed within 40 minutes: *a* – cyclic voltammogram with an inserted photograph of the electrode in a colored state; *b* – curve of transparency change during cycling

Since the diffusion rate is limited by the diffusion coefficient of the proton in the solid phase, a “smearing” of the reaction front can be observed in thick films, which can lead to rounding of the peak shapes.

An interesting effect was also the shift of the anodic peak in the cyclic voltammogram towards more positive potentials with an increase in the duration of film formation. This can also result from diffusion restrictions in thick films.

At the same time, photographs of super-thick electrodes obtained within 60 and 80 minutes indicated the presence of cracks and shedding of part of the electrochromic coatings. This may be due to the increasing stresses that occurred in thick films.

The optical characteristics also showed a clear dependence on the duration of deposition of the composite $\text{Ni}(\text{OH})_2$ -PVA electrochromic coating. For films obtained in the interval of deposition durations of 5–40 minutes, the coloration depth increased and then fell sharply for the deposition duration of 60 and 80 minutes (Fig. 4–6, *b*).

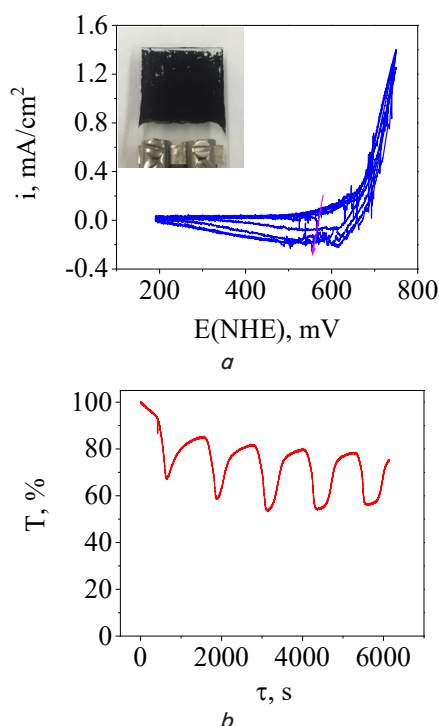


Fig. 5. Characteristics of a thin-film electrochromic electrode formed within 60 minutes: *a* – cyclic voltammogram with an inserted photograph of the electrode in a colored state; *b* – curve of transparency change during cycling

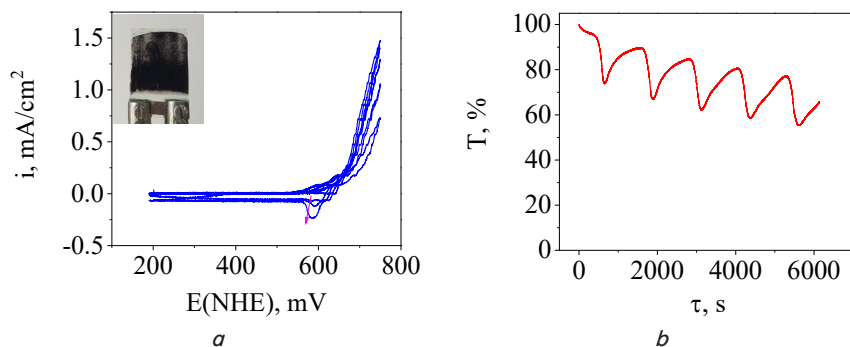


Fig. 6. Characteristics of a thin-film electrochromic electrode formed within 80 minutes: *a* – cyclic voltammogram with an inserted photograph of the electrode in a colored state; *b* – curve of transparency change during cycling

It was significant that in thick films obtained within 60 and 80 minutes, the irreversibility of bleaching also increased sharply.

Thus, a preliminary conclusion can be made that the deposition of very thick films leads to a significant deterioration in the electrochemical and optical characteristics of electrochromic electrodes.

6. Discussion of the results of studies of the characteristics of thin-film electrodes deposited with different durations

The curves in Fig. 7, 8 show a quantitative analysis of the characteristics of the films. The amount of electricity of the anodic and cathodic processes was determined by integrating the CVA by the trapezoidal method (Fig. 1–6, *a*). The analysis of this dependence suggests that with an increase in the deposition duration to 40 minutes, the amount of electricity (Q) used for the processes increased almost linearly and proportionally to the deposition duration. This is followed by a sharp drop in the Q value – durations of 60 and 80 minutes. Thick films may lose contact with the substrate and begin to work less efficiently. This assumption is proven by the insertions in Fig. 5, 6, *a*, since cracks and a partial absence of coating are visible on them.

Fig. 7, *b* shows the thicknesses of the films formed by the cathodic template method depending on deposition duration. When calculating the thickness (Faraday) using Faraday's law, some assumptions were made. The first assumption was that the current efficiency of the deposition process was taken to be 100 %. This is because the only possible process in dilute nickel nitrate solutions, the deposition of metallic nickel, was not observed [27]. The second assumption was that the $\text{Ni}(\text{OH})_2$ -PVA deposition was considered non-porous, solid, and homogeneous. This assumption is made because according to the results of scanning electron microscopy in [24], the deposited films were either non-porous or low-porous. In addition, the study [10] showed that polyvinyl alcohol was deposited together with $\text{Ni}(\text{OH})_2$, however, with an unknown ratio of components.

The film thickness δ (ref.) was obtained based on the experimental results for the deposition method used in this study and [24]. In this case, for a deposition duration of 10 minutes, the thickness was taken from [24], and the remaining thicknesses were recalculated based on the linear dependence of the deposition thickness on the deposition duration.

The last thickness δ (cathodic) was calculated from experimental data, namely, based on the specific cathode capacitances of the films shown in Fig. 7, *a*. The cathode capacitances were taken on the basis of minimizing the distortion of their values due to side processes. Cathode capacitances, unlike anode ones, did not contain an additional value arising from the release of oxygen during anodic polarization. The assumptions were the same as for the calculation of δ (Faraday). It should be noted that this method of synthesis produces the α -form of nickel hydroxide [10, 28]. Therefore, for calculations, we used the α - $\text{Ni}(\text{OH})_2$ density of 3.9 g/cm^3 [29].

Analysis of the dependencies in Fig. 7 leads to several important conclusions. The first conclusion is that the thickness of the films, calculated from the data of [24], is approximately two times greater than for flat, non-porous films, calculated by the other two methods. Obviously, the difference may be due to the presence of polyvinyl alcohol in the $\text{Ni}(\text{OH})_2$ -PVA composite. Thus, the volume of polyvinyl alcohol is approximately equal to the volume of nickel hydroxide. Interestingly, the δ calculation based only on the electrodeposition parameters δ (Faraday) and the calculation based on the partial use of experimental data δ (cathodic) in the deposition duration interval of 5–40 minutes practically coincide (Fig. 7, *b*). The coincidence of the curves in the interval of 5–40 minutes suggests several conclusions:

- the accepted assumptions are adequate to reality, but they do not take into account the co-deposition of polyvinyl alcohol, since the mechanism of polymer deposition is not directly related to the electrochemical process;
- the current efficiency of both the electrodeposition process and the oxidation process is equal or close to 100 %;
- electrochromic films deposited in the interval of 5–40 minutes electrochemically work over the entire thickness.

Further deviation of δ (cathodic) from δ (Faraday) may indicate the loss of contact of thick films with the conductive FTO glass substrate, which, as mentioned earlier, is most likely due to the appearance of stresses. The latter is also confirmed by the occurrence of irreversibility upon bleaching and a drop in the coloration depth starting with a deposition duration of 60 minutes (Fig. 8).

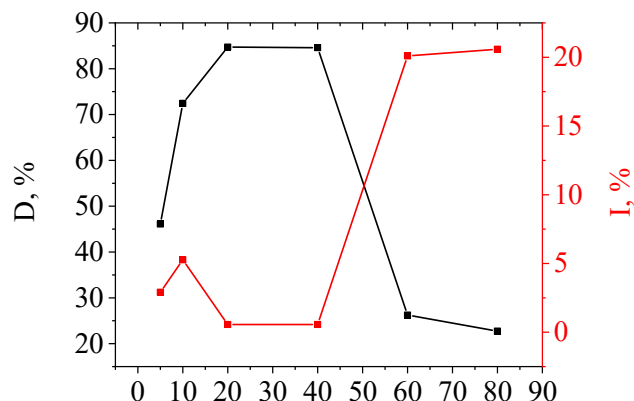


Fig. 8. Average characteristics of thin-film electrochromic electrodes formed with the deposition duration in the range of 5–80 minutes: D – coloration depth, I – irreversibility when bleaching electrochromic electrode

In terms of practical application, the data in Fig. 8 show that it makes no sense to increase the deposition time by more than 20 minutes under the chosen deposition conditions (current density, reagent concentration) [29–31] since a further increase in the deposition time does not lead to a significant increase in the coloration depth. At the same time, the long duration of deposition in production leads to additional consumption of reagents and electric current.

An important element in the further development of research in this direction should be the stage of determining the reasons for the deterioration in characteristics with an increase in deposition duration above 40 minutes.

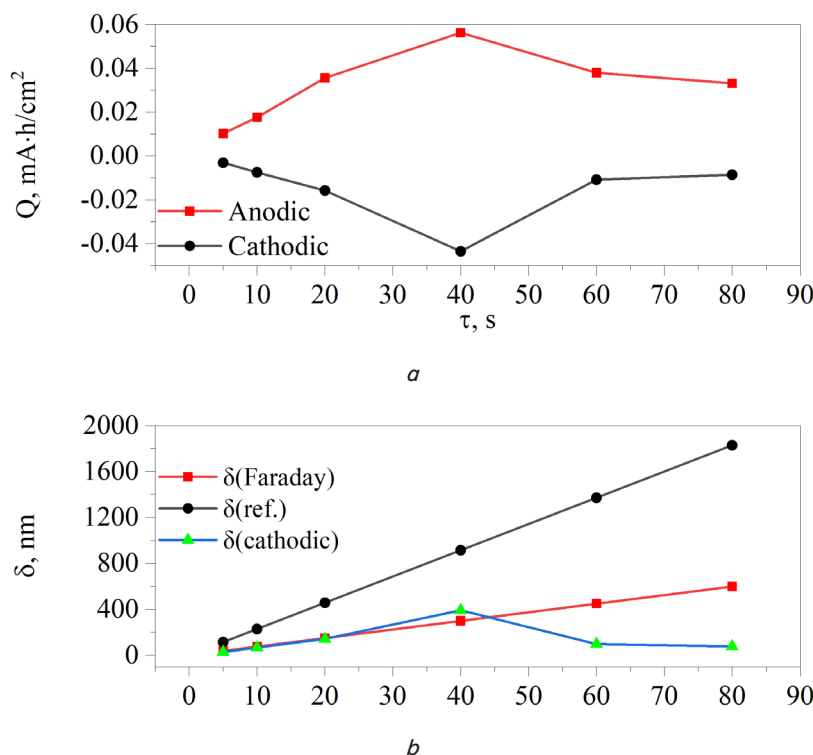


Fig. 7. Characteristics of a thin-film electrochromic electrode formed within 80 minutes: *a* – specific capacitances of the cathodic and anodic processes, calculated from CVA; *b* – thicknesses of the working $\text{Ni}(\text{OH})_2$ layer, calculated in different ways: δ (Faraday) – according to Faraday's law; δ (ref.) – according to literature data for this deposition method [24]; δ (cathodic) – based on specific capacitances for the cathodic process (Fig. 6, *a*)

7. Conclusions

As a result of the experimental work performed, it was shown that the optimal duration of deposition of electrochromic

composite films by the cathodic template method is the formation time of ≤ 20 min. The rest of the electrodeposition conditions is as follows: 0.01 M $\text{Ni}(\text{NO}_3)_2$, 5 % PVA solution; deposition on the cathode with a current density of 0.1 mA/cm².

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