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OPTIMIZATION OF THE DEPOSITION CONDITIONS FOR $Ni(OH)_2$ FILMS FOR ELECTROCHROMIC ELEMENTS OF “SMART” WINDOWS

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Дослідження було проведено для визначення впливу двох факторів на електроосадження плівок $Ni(OH)_2$: концентрації нітрату нікелю в електроліті і наявністю операції сушіння між отриманням плівки і її електрохімічними і оптичними випробуваннями. Для осадження використовували чисті розчини нітрату нікелю без добавок, щоб наявність останніх не ускладнювала аналіз одержуваних даних.

В результаті були отримані дві серії плівок з електролітів, які містять різні концентрації нітрату нікелю 0,01, 0,1 та 1 М. Відмінністю двох серій була відсутність або наявність операції сушіння після отримання. Сушіння плівок проводили в м'яких умовах при кімнатній температурі.

Електрохімічні та оптичні властивості визначали методом циклічної вольтамперометрії з паралельним записом зміни прозорості плівок.

В результаті аналізу отриманих даних було з'ясовано, що рівномірні плівки з приблизно однаковою товщиною можуть бути одержані в інтервалі концентрацій нітрату нікелю від 0,01 до 0,1 М. У розчинах, які містять 1 М нітрат нікелю, осадження відбувалося вкрай нерівномірно. Автори запропонували гіпотезу, згідно з якої така поведінка системи пояснюється перерозподілом густини струму по площі електрода через високу електропровідності концентрованого розчину нітрату нікелю. У свою чергу перерозподіл густини струму призводило до багаторазового підвищення густини струму у поверхні електроліт – повітря. Таке підвищення могло впливати на фронт реакції, який віддалявся від електрода або осадженням Ni $(OH)_2$ з високою товщиною. Останній втрачав контакт з поверхнею електрода і осипався.

У свою чергу було встановлено, що процес сушіння значно впливає на структуру і властивості плівок. Також було показано, що процес сушіння негативно впливає на візуальний вигляд плівок гідроксиду нікелю через утворення сітки тріщин на електроді. На додачу до цього, було припущено, що гідроксид нікелю отриманий електрохімічним шляхом містить велику кількість кристалізаційної води

Ключові слова: $Ni(OH)_2$, гідроксид нікелю, електрохромізм, NiOOH, плівка, електроосадження, циклічна вольтамперометрія, концентрація, нітрат нікелю

1. Introduction

Electrochromism is a phenomenon characterized by a reversible change in light absorbance (transmittance) of material under applied electrical current. The phenomenon was discovered more than 50 years ago [1, 2]. Nevertheless, real working prototypes of devices utilizing electrochromism only now started to appear [3, 4]. This is related to difficulties in implementing such systems, which in turn are determined by necessary requirements to them. These requirements are more strict than for other systems. Electrochromic materials that are based on electrochemical reactions are required to have a large working life cycle, stability over time and under different temperatures, while also having high transparency in bleached state and uniformed coloration in the colored state. This is the reason why development of new electrochromic materials that are suitable for application is such a difficult problem.

One of the devices that utilize electrochromic elements are smart windows. In turn, there is already a high demand for smart windows. There is a broad range of application for smart windows: glassing of buildings, automobiles, airplanes, creation of dynamic designs, etc. Additionally, the use of smart windows with dynamic transparency allows reducing power consumption for air conditioning [5].

2. Literature review and problem statement

Nickel (II) hydroxide is anodic electrochromic material that changes color from transparent to dark-brown upon oxidation [6, 7]. Anodic oxidation of nickel hydroxide occurs according to the solid-state reaction (1), which results in a transition from $Ni(OH)_2$ into nickel oxyhydroxide – NiOOH [7]:

$$Ni(OH)_2 \leftrightarrow NiOOH+H^{+}+1\bar{e}. \quad (1)$$

High reversibility of the electrochemical reaction (1) is the reason why this material is widely used in electrochemical accumulators [8, 9] and supercapacitors [10, 11]. This is also the reason why $Ni(OH)_2$ films are viewed as promising electrochromic material [12, 13].

Nickel hydroxide films possess high specific characteristics, including coloration efficiency reaching to 70 cm^2/C and number of coloration-bleaching cycles of up to 10^7 and more. The color of this material changes to dark-brown. At the same time, the majority of other materials change their color in the light-blue to dark-blue range. As such, the use of transparent counter-electrode allows a device to have dark-brown color in the colored state. However, nickel hydroxide films have poor adhesion to conductive transparent substrates (FTO, ITO) used for smart windows [12, 16].

Electrochemical methods favorably stand out among methods for deposition of electrochromic $Ni(OH)_2$ films [14, 15]. This is due to their relative simplicity, ease of control over final products, ease of automation, lack of high-tech equipment and relatively low power consumption [16]. However, despite existing works related to electrochemical deposition from solutions [17, 18], not all aspects of $Ni(OH)_2$ film deposition are studied to this day. Thus, deposition of $Ni(OH)_2$ from nickel nitrate solutions with different concentration onto low-conductivity substrates, which are oxide films used as electrodes, has not been studied in full detail. Influence of intermediate drying stage on cycling characteristics is also not studied. It should be noted that synthesis and cycling are conducted in different solutions, and drying stage can significantly affect the properties of resulting electrochromic films [19, 20]. Another thing to note is that manufacturing cost is largely determined by the deposition method. This is because the thickness of different electrochromic materials rarely exceeds 500–600 nm [21, 22], and more usually it ranges from a few to 100 nm [6]. As such, the specific consumption of rather costly reagents is rather small.

Nevertheless, the high price of smart windows (above 200 USD/m^2) limits their wide application. The cost is primarily determined by the deposition cost of electrochromic films. As such, the development of cheaper deposition methods and their optimization is a key problem in the technology of smart windows and mirrors.

As previously stated, the electrochemical deposition method is one of the cheapest methods, as it doesn't require high-tech equipment and special conditions. The present work is a dedication to optimization and finding of optimal conditions for deposition of nickel (II) hydroxide films.

3. The aim and objectives of the study

The aim of the work is to study the influence of a few factors on the deposition of electrochromic films onto glass substrates coated with flouring-doped tin oxide (FTO glass). The factors are: concentration of nickel nitrate solution used for deposition of $Ni(OH)_2$, and presence/absence of drying between deposition and cycling of the electrochromic film.

To achieve the set aim, the following objectives were formulated:

– to deposit 6 films (2 films per solution) from solutions with a concentration of 1, 0.1 and 0.01 M $Ni(NO_3)_2$, with three of prepared films not being subjected to drying between preparation and optical tests;

– to evaluate electrochemical and optical characteristics of films and visually compare and evaluate the quality of prepared electrochromic films.

4. Materials and methods used to study the influence of conditions on the deposition of electrochromic films

Analytical grade reagents were used in the study. All solutions were prepared using distilled water. FTO glass 7.5 Ω/□ (China, Zhuhai Kaivo Optoelectronic Technology Co.) was used as a substrate. Before the deposition, the FTO glass was treated as follows: washing with wet sodium bicarbonate, washing with distilled water, ultrasonic treatment in ethanol (50 W, 41.5 kHz), 400 s.

Electrodeposition was conducted in a single compartment acrylic cell – Fig. 1. The working surface of FTO glass was S=4 cm^2 (2×2 cm). All films were deposited cathodically at i=0.1 mA/cm^2 for 10 min. The following solutions were used for deposition: 1, 0.1 and 0.01 M $Ni(NO_3)_2$. It should be noted that unlike in previous works, the electrolyte did not contain any additives [6, 7, 11–13, 16]. This is necessary to isolate the influence of additives from the main processes. In all experiments, the electrode with the freshly deposited electrochromic film was washed by soaking it in a beaker with distilled water.

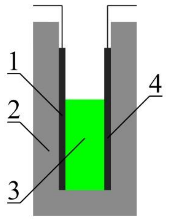


Fig. 1. Schematic of the cell for film deposition: 1 – cathode (FTO glass, secured in a holder); 2 – cell frame; 3 – electrolyte; 4 – anode (nickel plate)

Drying was conducted in air at room temperatures, for intended samples.

For secure contact between the substrate electrode and current supply and for ease of testing, an in-house built holder was employed (Fig. 2). FTO glass was placed into a slit of two cylindrical holders, with the conductive film oriented upwards. A soft nickel foil was placed between the conductive film and retention bolts to ensure uniform distribution of electrical current across the electrode and prevent damage to the conductive film.

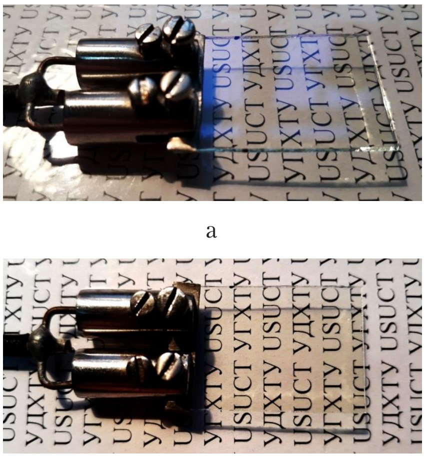


Fig. 2. Photogram of the electrode holder with FTO glass: *a* — side view, *b* — top view

Experimental data were recorded using the potentiostat Elins P8. The cell for simultaneous recording of cyclic voltammograms and coloration-bleaching curves is shown in Fig. 3.

FTO glass with deposited $Ni(OH)_2$ electrochromic film was used as a working electrode.

Nickel foil was used as a counter electrode and Ag/AgCl (KCl sat.) was used as a reference electrode.

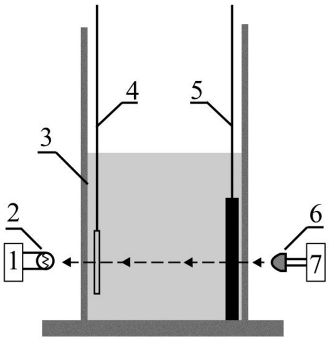


Fig. 3. Cell for recording of cyclic voltammograms: 1 — analog-to-digital converter; 2 — photoresistor; 3 — cell made of transparent polymer; 4 — working electrode; 5 — counter-electrode; 6 — source of why light (T_c=5500 K); 7 — source of stabilized voltage

All cyclic voltammograms for $Ni(OH)_2$ films were recorded using the following cycling parameters:

– potential window E_1=201 mV to E_2=+751 mV;

– scan rate 1 mV/s;

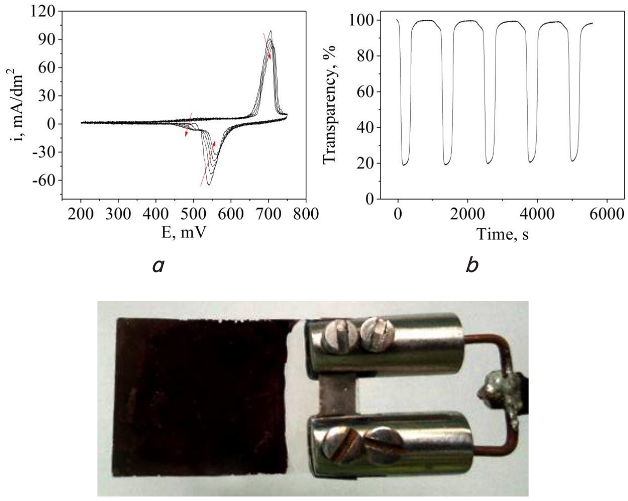
– number of cycles – five.

Additionally, after cyclic voltammetry, each film was colored potentiodynamically, starting at 201 mV to +751 mV at 1 mV/s.

5. Analysis and comparison of optical and electrochemical properties of $Ni(OH)_2$ films prepared under different conditions

As a result of conducted experiments, 2 series of films for electrolytes with a concentration of 0.01, 0.1 and 1 M were prepared. The difference between the series is the drying stage between synthesis and electrochemical tests.

Results for films prepared without intermediate drying stage are shown in Fig. 4–6. It can be immediately noted that the use of dilute solutions, 0.01 and 0.1 M $Ni(NO_3)_2$, resulted in high-quality uniform films. They were almost unnoticeable in a transparent state. The cyclic voltammetry curve for the film prepared from 0.01 $Ni(NO_3)_2$ showed well-defined peaks of oxidation and reduction processes, at about 700 mV and 550 mV respectively – Fig. 4, *a*. It should be noted that the shape of the cathodic peaks was constantly changing with each subsequent cycle. At the same time, coloration and bleaching processes were rather stable, Fig. 4, *b*. The coloration degree for this film was about 81 %. The electrode photograph with the colored film demonstrated good uniformity and intense coloration, Fig. 4, *c*.



a *b*

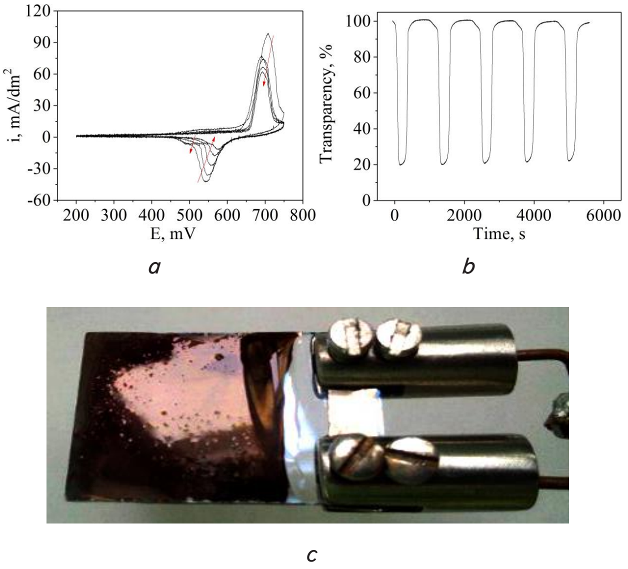
c

Fig. 4. Experimental results for the film deposited from 0.01 M $Ni(NO_3)_2$ without drying stage between deposition and cycling: *a* — cyclic voltamperometry curve (red arrows show changes during cycling); *b* — bleaching coloration curve; *c* — electrode in the colored state after cycling

Nickel hydroxide film deposited from a more concentrated solution; demonstrated somewhat worse results – Fig. 5. Peak current densities on cyclic voltamperometry curves were on average lower than those of the film deposited from 0.01 M $Ni(NO_3)_2$. The final peak current densities of oxidation and reduction peaks were 60 and 12 mA/dm^2, for the film deposited from 0.1 M $Ni(NO_3)_2$. At the same time, for the film deposited from 0.01 M $Ni(NO_3)_2$, the same values were about 80 and 32 mA/dm^2. The coloration degree of the film deposited from 0.1 M $Ni(NO_3)_2$ was also lower and was about 78 %. Other than this, electrochemical and optical characteristics are similar for both films: changes of the cathodic peak and shape of the coloration-bleaching curve.

The film deposited from the most concentrated solution did not show electrochemical and optical activity – Fig. 6.

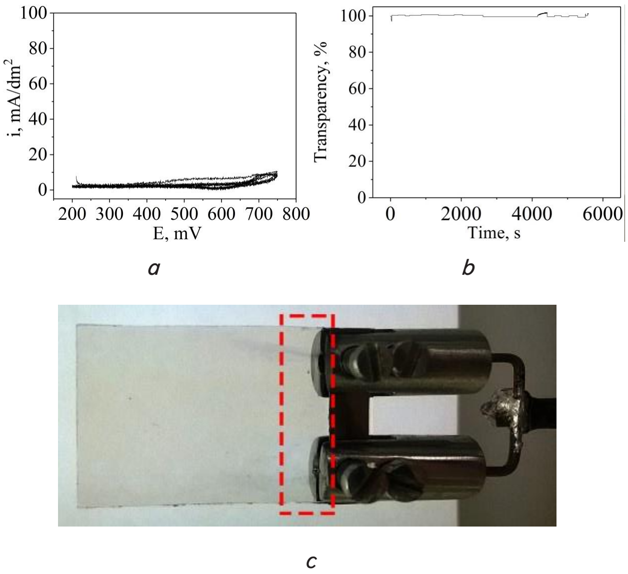
Upon visual inspection, there was an impression that the film was absent in the electrode. Nevertheless, some small currents were recorded on the CVA curve and the barely noticeable color change near the current lead revealed that some deposition did occur, Fig. 6, *a*, *c*.



a *b*

c

Fig. 5. Experimental results for the film deposited from 0.1 M Ni(NO$_3$)$_2$ without drying stage between deposition and cycling: *a* – cyclic voltamperometry curve (red arrows show changes during cycling); *b* – bleaching coloration curve; *c* – electrode in the colored state after cycling (pink spot – glossy reflection)



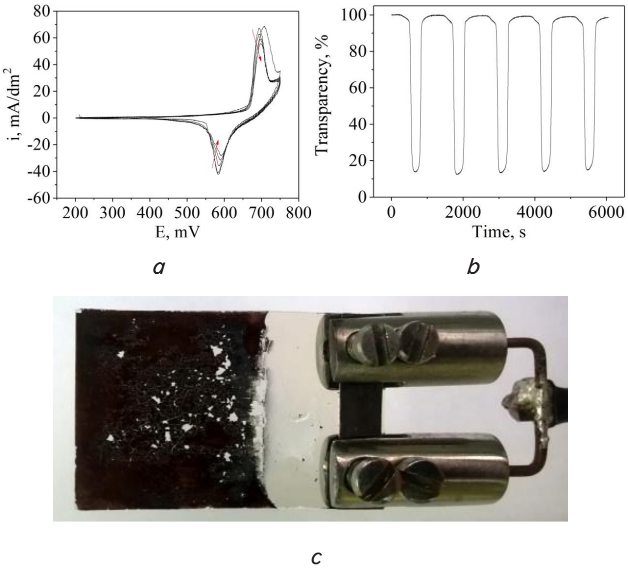
a *b*

c

Fig. 6. Experimental results for the film deposited from 1 M Ni(NO$_3$)$_2$ without drying stage between deposition and cycling: *a* – cyclic voltamperometry curve; *b* – bleaching coloration curve; *c* – electrode in the colored state after cycling (dashed lines show part of the electrode where film deposition occurred (dark lines))

In the second series of experiments, there was a drying stage between deposition and electrochemical tests. The results obtained for this series showed similar dependencies, however, there were some key differences.

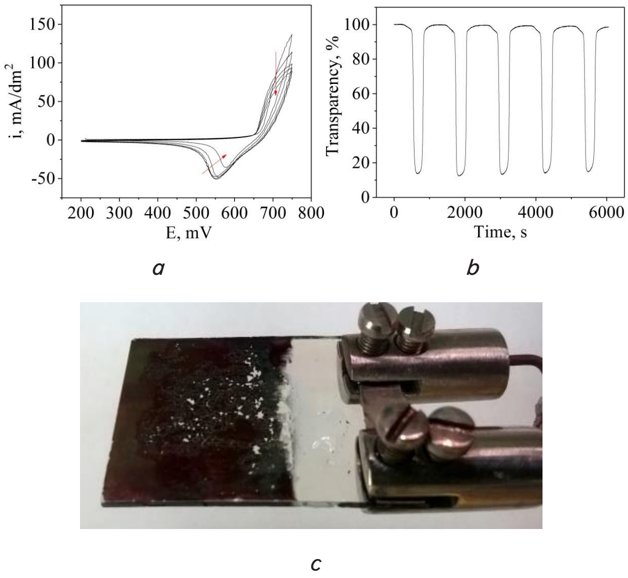
Cyclic voltamperometry curves for the films deposited from 0.01 M Ni(NO$_3$)$_2$ and 0.1 M Ni(NO$_3$)$_2$ without drying were significantly different from the films prepared with drying, Fig. 7–9, *a*. Peak currents for these films were about 60 and 30, 80 and 40 mA/cm^2. The coloration degree was about 84 % for both films. Films prepared with intermediate drying showed significant cracking in colored state Fig. 7, *c* and Fig. 8, *c*.



a *b*

c

Fig. 7. Results for the film deposited from 0.01 M Ni(NO$_3$)$_2$ with drying between deposition and testing: *a* – cyclic voltamperometry curve (red arrows show changes during cycling); *b* – coloration-bleaching curve; *c* – electrode after cycling in the colored state



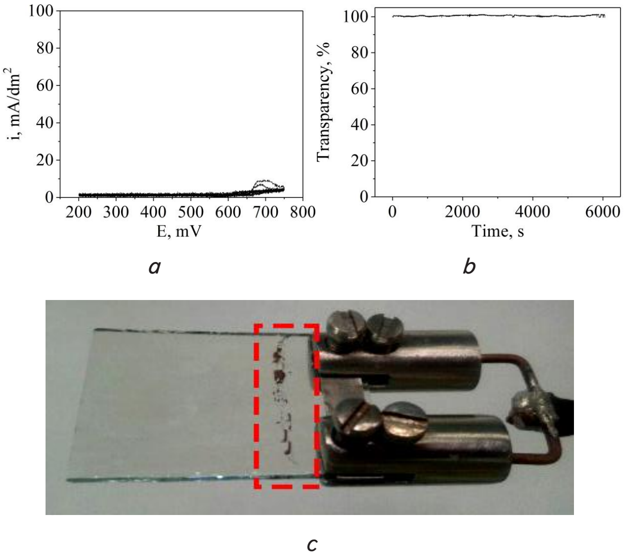
a *b*

c

Fig. 8. Results for the film deposited from 0.1 M Ni(NO$_3$)$_2$ with drying between deposition and testing: *a* – cyclic voltamperometry curve (red arrows show changes during cycling); *b* – coloration-bleaching curve; *c* – electrode after cycling in the colored state

In case of the film deposited from the most concentrated solution with intermediate drying stage (Fig. 9), electrochemical and optical activities were almost non-existent.

Visual inspection revealed that the film was only deposited at the boundary electrolyte-air. Such behavior is similar to the variant of the film deposited from the same solution but without drying. However, some dark spots on the electrode surface can be seen on the photograph of the electrode in the colored state – Fig. 9, *c*.



a *b*

c

Fig. 9. Results for the film deposited from 0.01 M $Ni(NO_3)_2$ with drying between deposition and testing: *a* — cyclic voltamperometry curve; *b* — coloration-bleaching curve; *c* — electrode after cycling in the colored state (dashed lines show part of the electrode where film deposition occurred (dark spots))

6. Discussion of the results obtained for the studied films

Analysis of the obtained dependencies allows making a few conclusions on the influence of $Ni(NO_3)_2$ and drying stage. It should be noted that electrolytes with nickel nitrate concentration within 0.01 to 0.1 M produce uniform films with good electrochromic properties. In turn, concentrations of $Ni(NO_3)_2$ 1 M and higher do not produce uniform films over the entire working area of the electrode. Another thing to note is that coloration degree of the films deposited from 0.01–0.1 M solutions is about the same. The latter leads to a conclusion that the thickness of the films deposited from these solutions is roughly the same, and in turn, the current yield doesn't change much within this range. However, the drying stage leads to film cracking, which most likely means that electrochemically deposited films contain a large amount of crystal water. This assumption is based on literature data [23], which described a significant amount of crystal water in $Ni(OH)_2$ precipitations prepared under similar conditions. Such cracking also shows that the $Ni(OH)_2$ film without any additives is inelastic and rigid. This means that the use of pure $Ni(OH)_2$ films in flexible or stretchable supercapacitors or electrochromic devices is not possible without special additives [24]. This also means poor adhesion to the substrate. It is obvious that drying also induces some structural changes. This can be seen from notable changes in the shape of cyclic voltamperometry curves. For instance, all dried samples do not show the region that is present in the samples that were not dried – Fig. 4, *a*, 5, *a* (highlighted with the arrow).

However, the most interesting behavior is shown by the film deposited from concentrated solutions. The disappearance of a uniform deposit is likely related to redistribution of current on the electrode surface due to the high conductivity of the electrolyte. This leads to a significantly higher current density near the electrolyte-air boundary and leads to two possible scenarios:

– shift of the reaction front away from the electrode, which leads to the formation of $Ni(OH)_2$ in the electrolyte volume instead of the electrode surface;

– formation of a very thick film near the electrolyte-air boundary, which peels off and falls to the bottom of the deposition cell.

In the end, both of these possible mechanisms would result in insufficient electrode coverage by the film.

As a result of conducted experiments , individual and combined influence of two parameters of astrochemical deposition of $Ni(OH)_2$ – electrolyte concentration and intermediate drying stage was found. Based on the obtained results, it can be recommended to avoid intermediate drying and conduct electrodeposition from the outmost diluted solution possible. The latter can reduce the cost of electrodeposition, as the main loss of valuable components in electroplating occurs during their removal with the plated piece. This removal occurs when the piece (which has the electrolyte film on it) is removed from and transferred to the washing stage. As such, the lower concentration of electrolytes in the bath the lower the removal (i.e. loss).

As such, further optimization of electrodeposition of electrochromic films can significantly reduce deposition cost. It is known that the cost of depositing 1 m^2 of the galvanic film is about 1–3 USD/m^2, while the cost of depositing 1 m^2 of the film using vacuum sputtering is at least 7.97 USD/m^2 [25]. The latter is commonly used for deposition of electrochromic and transparent conductive films.

The next problem to solve is which additives can be used to improve film adhesion to the substrate that can also be served over a wide range of temperatures. The latter is partially solved by the authors in [7, 12, 13, 16]. Wide temperature range is required because the absorbed light would be transformed into heat, which can result in the film temperature rising up to 80 °C and more. On the other hand, depending on the region, temperatures as low as –20 °C are possible. As such, the total temperature range approaches 100 °C, which is a real challenge for people developing new materials.

7. Conclusions

1. The concentration range in which deposition of uniform $Ni(OH)_2$ electrochromic films can be achieved is 0.01–0.1 M $Ni(NO_3)_2$.

2. It was found that drying process has a significant impact on the behavior of electrochromic films deposited from pure $Ni(NO_3)_2$ solutions, which is evidenced by changes in cyclic voltamperometry curves and visual changes in colored films, in comparison to the films that were prepared without drying. Drying also leads to film cracking which is unacceptable for electrochromic elements of smart windows.

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