Електрохромні пристрої як елемент «розумних» вікон можуть піддаватися впливу екстремальних температур через їх розташування і призначення. Висока температура може призводити до зміни експлуатаційних властивостей електрохромних пристроїв і виходу їх з ладу. Дане дослідження покликане заповнити прогалини, пов'язані з стабільністю електрохімічних і електрохромних параметрів одного з відомих матеріалів – гідроксиду нікелю (II).

Представлене дослідження висвітлює зміни деяких фізико-хімічних характеристик, що відбувається при тривалому впливі високої температури в різних середовищах. В експериментах композитну плівку $Ni(OH)_2$-полівініловий спирт, отриманий катодним темплатним методом, витримували при температурі 80 °С в повітряній атмосфері і в розчині робочого електроліту – 0,1 М КОН по 8 годин. Температура була обрана виходячи з можливих максимальних значень реєстрованих на землі, можливого нагрівання плівки та швидкої можливої деградації електрохромних покриттів.

В результаті було показано, що деградація відбувається в розчині лугу, в той же час на повітрі відбувалося деяке поліпшення характеристик. Запропоновано механізм, який пояснює результати досліджень і полягає в процесі «старіння» активної речовини $Ni(OH)_2$. Останній має місце в активних масах лужних акумуляторів. У статті також запропоновано можливі способи подолання деградації, які можуть бути реалізовані через використання загущених електролітів або спеціальних плівок, що наносяться зверху на електрохромне покриття

Ключові слова: електрохромний пристрій, електрохімічне осадження, гідроксид нікелю, темплат, полівініловий спирт, деградація, рекристалізація, лужний розчин, затемнення, освітлення

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A STUDY OF THE INCREASED TEMPERATURE INFLUENCE ON THE ELECTROCHROMIC AND ELECTROCHEMICAL CHARACTERISTICS OF $Ni(OH)_2$-PVA COMPOSITE FILMS

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1. Introduction

Climate change across the world affects lifestyle and economy [1, 2]. In these circumstances, leading research clusters are in search of approaches and solutions, evaluate the effect of climate and make estimations [3, 4]. One of the solutions that can be effectively used is the widespread usage of so-called "smart devices" that would also be the basis for creating "smart cities". Usage of "smart" devices allows for saving power and resources. One of the important functional tasks can be power saving for air conditioning, which can reach 50 % [5].

"Smart" windows are a subcategory of smart devices. Their key component are electrochromic elements [6, 7]. These elements can be based on different systems, such as: electrochromic systems [8, 9], liquid crystals [10, 11], suspended particles [12, 13]. Electrochemical electrochromes have become the most prevalent. Their key advantages – wide spectrum of available color, absence of constant power consumption (the power is consumed during color change). In turn, inorganic electrochromes do not undergo destruction under UV light, thus maintaining their optical properties. On the other hand, parameters of elec-

trochemical electrochromic materials are unstable and degrade over time.

$Ni(OH)_2$ is one of the well-known electrochemical electrochromic materials [14, 15]. It is an anodic electrochrome, as it undergoes a color change from transparent to dark-brown when polarized anodically. The color change reaction can be expressed by equation (1) and (2):

$$Ni(OH)_2 \leftrightarrow NiOOH + H^+ + e^-, \quad (1)$$

$$\underset{\text{transparent}}{Ni(OH)_2} + OH^- \leftrightarrow \underset{\text{dark-brown}}{NiOOH} + H_2O + e^-. \quad (2)$$

Reaction (1) is a solid-state reaction and occurs within the solid particle. Reaction (2) is a total reaction that includes alkaline media required for the material to operate.

There are many ways to deposit nickel hydroxide onto conductive transparent surfaces of substrates. Electrochemical deposition of nickel (II) hydroxide is cheaper than the traditional vacuum sputtering method. Such method is also easy to automate. Parameters of the resulting film can be easily adjusted by adjusting deposition current density, time, introduction of additives. Thus, research in the field of electrochemical deposition methods and characterization of resulting films are important in terms of applied science and production of metal oxide films.

2. Literature review and problem statement

In [16, 17], the authors propose designs of electrochromic devices that can significantly lower the cost of manufacturing and final product. To achieve cost reduction, the authors employed the following approaches – lower number of layers to be vacuum sputtered, changing the second electrode with the active material to a mesh, using aqueous electrolyte instead of propylene carbonate. The electrochromic electrode used in these designs was deposited electrochemically and is a $Ni(OH)_2$-polyvinyl alcohol composite [15, 18].

Analysis of works related to new electrochromic materials based on $Ni(OH)_2$ [19, 20] revealed that the majority of laboratory studies are limited to room temperature or close to it. The temperature can have a significant effect on the electrochemical process and can also affect the compound itself.

It's worth noting that temperature in the hottest places of the planet can reach 50 °C and above. For instance, the highest registered temperature of 56.7 °C was measured in the USA (Death Valley, California) [21]. And because electrochromic devices lower their transparency on coloration, part of incoming light is absorbed and transformed into heat. This can heat up the devices significantly, with the temperature increase of a few dozen degrees. Such operating conditions assume that in addition to the development of new electrochromic materials, significant attention should also be paid to the influence of temperature on their physico-chemical properties.

In modern scientific literature, not enough attention is paid to the effect of high and low temperatures, temperature spikes on the characteristics of electrochromic materials. Nevertheless, this information can be key in evaluating the possibility of realizing the technology. Literature search on this topic resulted only in a few sources related to the effect of temperature on the physico-chemical characteristics of electrochromic materials (devices).

The paper [22] describes the degradation of the NiO/WO_3 electrochromic device at temperatures above 60 °C. The device used a Li^+ conducting electrolyte – $LiClO_4$ in a mixture of polypropylene carbonate and polymethyl methacrylate. It is interesting that the degradation was also found at low temperatures in 0 to −40 °C range. It is noted that after degradation at low temperatures, the film characteristics were completely restored upon return to room temperature.

The authors of [23] describe that low-temperature synthesis of NiO films yields films with high specific characteristics, which is due to the resulting structure allowing for better migration of Li^+.

Other authors [24, 25] have subjected films of nanoparticles WO_x and nanowires $W_{18}O_{49}$ to low-temperature calcination (250–350 °C). The authors note that this operation improved the electrochromic characteristics and durability of films. It is noted that low-temperature calcination can significantly improve intercalation-deintercalation of Li^+ ions for both films and nanowires.

A series of papers [26, 27] and also the paper [28] describe the study on degradation of mirror electrochromic devices based on Mg_4Ni at 30–50 °C and relative humidity of 80 and 60 %. Based on a wide range of experimental methods, the authors conclude that the degradation of electrochromic films is related to oxidation of film elements, increasing surface roughness. The authors note that humidity plays a key role.

The same authors [29] also studied the effect of sub-zero temperatures on the same system. It is noted that at −5 °C, the change in characteristics is insignificant and degradation is almost absent. The papers [27, 30] proposed variants of a protective layer that would limit the degradation of the Mg_4Ni-based electrochromic device at elevated temperatures.

The analysis of the listed papers allows to conclude that there is little research regarding the effect of elevated temperatures on the characteristics of electrochromic devices. In addition, the existing studies do not consider degradation in aqueous electrolytes, including composite films.

3. The aim and objectives of the study

The aim of the study is to evaluate the effect of prolonged exposure to elevated temperatures on electrochemical and electrochromic properties of $Ni(OH)_2$-polyvinyl alcohol composite films. The found characteristics would be valuable information for understating the limit operating conditions of electrochemical devices built with this composite.

To achieve the aim, the following objectives were set:

– deposit $Ni(OH)_2$-polyvinyl alcohol composite films;

– conduct prolonged ageing of prepared $Ni(OH)_2$ films at elevated temperatures in the working electrolyte and on air;

– test treated films and conduct a comparative analysis of experimental data.

4. Materials and methods used to determine the effect of high temperature on the properties of $Ni(OH)_2$-PVA films

To achieve a more pronounced degradation of film characteristics, the ageing temperature was set to 80 °C. To compare the effect of the ageing medium on the electrochemical and electrochromic characteristics, the film was aged under air and in the working electrolyte, which was an aqueous

solution of 0.1 M KOH. Ageing time was set to 8 hours, which corresponds to working hours.

Before the film deposition, the glass coated with SnO_2:F (FTO) (R<10 Ω/sq., Zhuhai Kaivo Optoelectronic Technology Co. Ltd., China) was degreased using aqueous Na_2CO_3 paste. The substrate was then washed with distilled water and ultrasonically treated in 96 % ethanol (10 min, 60 W, 41.5 kHz) and dried. Directly before use, the substrates were additionally wiped with a microfiber cloth soaked in ethanol. FTO substrates were 3×2 cm pieces with 2×2 cm working electrode area. $Ni(OH)_2$–polyvinyl alcohol composite films were deposited using the cathodic template method [31]. Deposition conditions: cathodic current density 0.1 mA/cm^2, deposition time 600 s, solution 0.01 M $Ni(NO_3)_2$ and 50 g/L PVA [32]. The resulting films were dried at room temperature for 1 h. One of the films was subjected to cyclic voltammetry studies and its optical behavior was recorded. The other two were aged in air and working electrolyte for 8 h respectively. After ageing, the films were subjected to cyclic voltammetry analysis as the first film. For simplicity, all prepared films are labeled as follows: no treatment – Fresh, treatment on air – Air, treatment in the working electrolyte 0.1 M KOH – KOH.

Electrochemical measurements.

Electrochemical characteristics were evaluated using cyclic voltammetry (CV) analysis. Measurements were conducted using a 3-electrode setup [33] at 1 mV/s with the potential window of [+201; +701 mV] vs NHE. Ag/AgCl (KCl sat.) was used as a reference electrode and nickel foil – as a counter-electrode. 0.1 M KOH solution was used as a working electrolyte. Cell material – transparent acrylic. During cycling, optical characteristics were recorded. Experimental measurements were conducted using the setup shown in Fig. 1.

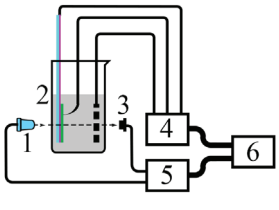


Fig. 1. Simplified schematic of the setup for characterizing electrochromic films: 1 – light source (5500 K); 2 – cell with free electrolyte and studied electrochromic electrode; 3 – photoresistor; 4 – digital potentiostat (Elins P-8); 5 – ADC (E-154) and power supply for the light source; 6 – computer; – glass; – conductive later; – $Ni(OH)_2$-PVA composite film

The setup includes analog-to-digital converter E-154 (Russia) and digital potentiostat Ellins P-8 (Russia) with software supplied by manufacturers.

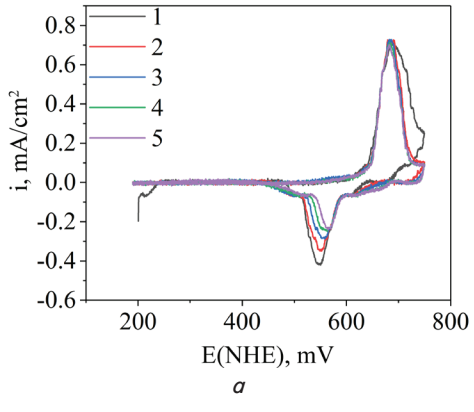
At the end of each potentiodynamic cycling, the films were photographed for visual evaluation of film quality.

5. Comparison of data obtained for film electrodes

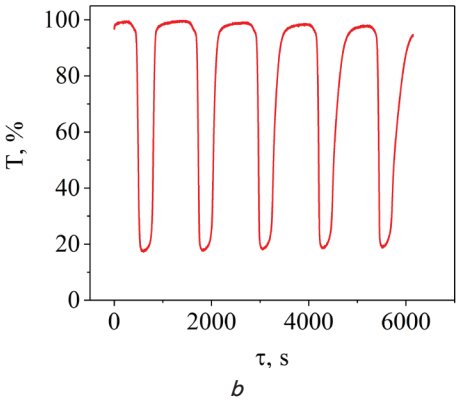
5. 1. Electrochemical and optical characteristics of the untreated electrode

To evaluate the effect of treatment regimes, one of the obtained films was not subjected to any kind of treatment. The freshly prepared film was subjected to cyclic voltammetry studies – Fig. 2, *a*, during which the optical behavior of the film also was recorded – Fig. 2, *b*. Transparency (T, %) is plotted against the duration of the experiment.

The analysis of the obtained dependency in Fig. 2, *a* revealed that the film is electrochemically active. The curve shows peaks corresponding to oxidation and reduction of $Ni(OH)_2$. Anodic current density is about 0.71 mA/cm^2.



a



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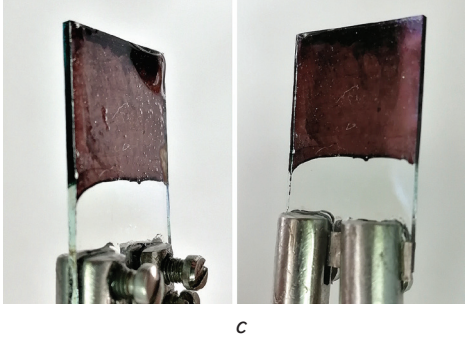

c

Fig. 2. Results for the film prepared without treatment (Fresh sample): *a* – CV; *b* – coloration-bleaching curve; *c* – photographs of the electrodes in the colored state after cycling

The height of the cathodic peak varies and on average is 0.27 mA/cm^2, while its width increases with each cycle. The potentials of the anodic and cathodic peaks are about 680 and 560 mV respectively. It is interesting that despite changes on the CV curves, the coloration-bleaching curve shows relative stability – coloration degree (difference of T, % between the colored and bleached states) is about 80–82 %, and the shape is almost constant. The change in the curve shape is only observed in parts at which the film transits into the bleached state – the transition becomes less sharp. The photograph of the film reveals a uniform and saturated color without significant irregularities.

5. 2. Electrochemical and optical characteristics of the electrode aged for 8 hours at 80 °C under air atmosphere

The film treated at an elevated temperature in the air atmosphere showed significantly different behavior. The cyclic voltammogram of this film differed – Fig. 3, *a*. The peaks on the CV curves are higher and sharper when compared to those of the reference Fresh sample. Both the height and position of both peaks have changed: anodic 710 mV and 0.66 mV/cm^2, cathodic 580 mV and 0.36 mA/cm^2. Additionally, the cathodic peaks were sharp without broadening at the base. The coloration-bleaching curve revealed stable optical characteristics of the sample – Fig. 3, *a*.

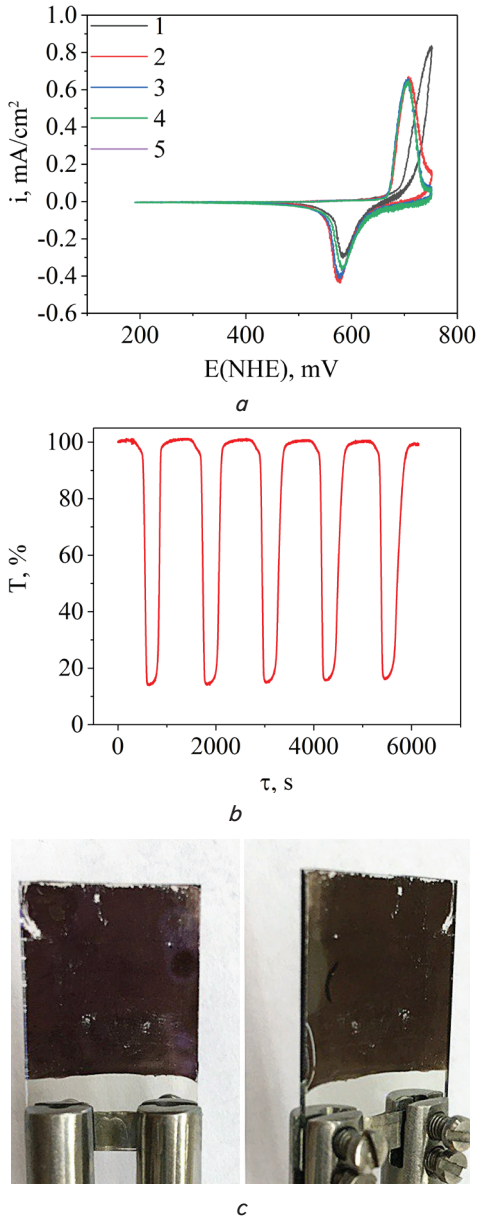

Fig. 3. Results for the film, aged for 8 hours at 80 °C under air atmosphere (Air sample): *a* – CV; *b* – coloration-bleaching curve; *c* – photographs of the electrode in the colored state after cycling

This is related to the fact that the shape and path of the curve were almost constant. The stability of optical characteristics was even higher than that of the Fresh sample. In addition, the coloration degree was also higher than that of the Fresh sample, reaching 86 %. The photograph of the film in the colored state shows the presence of a rather uniform film with almost uniform coloration.

5. 3. Electrochemical and optical characteristics of the electrode aged for 8 hours at 80 °C in the working electrolyte

In the case of the film treated in hot alkali, the electrochromic and electrochemical properties have decreased significantly. There are no discernible peaks, with current densities being close to zero – Fig. 4, *a*. The coloration-bleaching curve also shows a significant decrease of electrochromic characteristics – Fig. 4, *b*. The curve shows that some coloration and bleaching take place, but they do not exceed 5 %. The film electrode gradually loses transparency and after 5 cycles transparency drops by about 15 %.

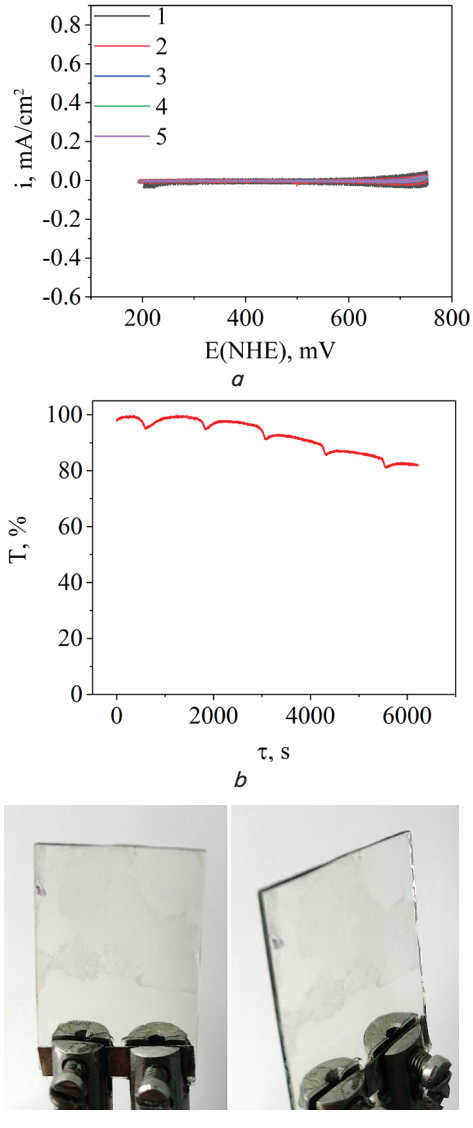

Fig. 4. Results for the film, aged for 8 hours at 80 °C in 0.1 M KOH (KOH sample): *a* – CV; *b* – coloration-bleaching curve; *c* – photographs of the electrode in the colored state after cycling

The photographs further testify to significant degradation of the electrode treated in hot alkali. The film is barely

visible and its coloration is poor. It is obvious that ageing in alkali at elevated temperatures causes severe degradation of the electrochromic film.

6. Discussion of the characteristics of film electrodes treated at elevated temperature and without treatment

To understand the quantitative effect of different treatment regimes at elevated temperatures, it was decided to calculate the capacity of the cathodic process for all three films. The specific capacities involved in the reduction of $NiOOH \rightarrow Ni(OH)_2$ are an integral value that can be compared with each other. The cathodic process was chosen specifically, because oxygen evolution can take place during the anodic process as a side reaction (3):

$$4OH^- \rightarrow H_2O + O_2 + 4e^-. \quad (3)$$

In order to exclude the capacity used in that reaction, only capacities for cathodic processes were considered. The specific capacities were calculated from the fifth CV cycle and are shown in Fig. 5.

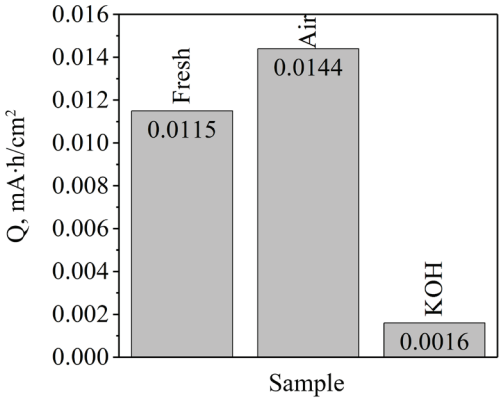

Fig. 5. Specific cathodic capacities of three samples: Fresh, Air, KOH

As shown in the previous paragraph, the worst performance was demonstrated by the sample aged at 80 °C in the KOH solution. Nevertheless, as can be seen from the comparison, Fresh and Air samples demonstrate electrochemical activity. It is also clear that the sample treated under the air atmosphere at 80 °C has a higher capacity than the freshly prepared sample. The shape and position of the peaks on the CV curves changed. These facts can be explained as follows. It is known that electrochemical deposition from the nitrate solution results in the formation of α and α-like forms of $Ni(OH)_2$ [34, 36]. These forms are unstable and contain structural water, which can be lost upon heating above 60 °C. Thus, such ageing is quite likely to cause the transformation from α into β- $Ni(OH)_2$. Even though the α-form is more active, and some decrease in characteristics had to occur, the opposite is observed – increase of peaks, capacity and coloration degree. This contradiction would require additional studies to solve, however, an assumption can be made. It is possible that upon treated under hot air, polyvinyl alcohol within the film [18] became softer and formed a stronger bond with the substrate. This assumption is based on the fact that the glass transition temperature of PVA is about 80 °C. A stronger bond of the film with the substrate leads to better characteristics and outweighs the impact from the transition from α to β- $Ni(OH)_2$.

In our opinion, the most interesting outcome is severe degradation upon ageing in alkali. This process would also occur in the assembled device. It is evident that the medium in which degradation occurs plays a key role, as even at other parameters being the same (time and temperature), ageing in the electrolyte solution causes the fastest degradation. Most likely, it is related to known "ageing" of active material, which lies in the re-crystallization of $Ni(OH)_2$ in aqueous and alkaline media. This process causes loss of activity and capacity of $Ni(OH)_2$ powders used in alkaline batteries and some types of asymmetric supercapacitors. The process is constituted by the loss of water, layer ordering, decrease of structural defect and partial loss of admixtures that could've been incorporated into the crystal structure during synthesis. This process takes place in basic solutions and is evidently accelerated by temperature [35].

It is obvious that this negative occurrence must be prevented. Because high temperature accelerates this process, the electrochromic films based on $Ni(OH)_2$ along with devices utilizing this material should not be subjected to high temperatures. Another possible approach is to employ thickened electrolytes and protective film layers with ionic conductivity. These two variants could contribute to a decrease in the rate of dissolution-precipitation reactions during recrystallization, since the aging process occurs by the liquid mechanism.

7. Conclusions

1. The characteristics of composite $Ni(OH)_2$-PVA films that have been subject to ageing at high temperature in different media: in air and in 0.1 M KOH electrolyte at 80 °C for 8 hours were studied. As a result, it was found that despite the same synthesis method, the resulting characteristics of the films after treatment differed significantly. High specific characteristics of the untreated film can also be noted: coloration degree of 82 % and capacity of the reduction process of NiOOH into $Ni(OH)_2$ 0.0115 mA·h/cm^2.

2. It was found that after treatment in air media, electrochemical and electrochromic characteristics of the film somewhat improved: coloration degree ≈86 %. The coloration degree of the untreated film ≈82 %. At the same time, the film treated in alkali solution has lost almost all of its electrochemical activity and electrochromic properties – coloration degree does not exceed 5 %.

3. The reasons for film behavior have been analyzed. It is assumed that high film degradation is related to the re-crystallization process, which occurs in active electrode materials of chemical power sources. Obviously, the re-crystallization of the $Ni(OH)_2$ film occurs according to the liquid-phase mechanism. Possible solutions were proposed for decreasing the degradation of the electrochromic film in the KOH solution. It is assumed that degradation can be retarded with the use of a thickened electrolyte and barrier films that would limit the rate of liquid re-crystallization processes.

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