



Morphology and electrochromic property of chemical bath deposited NiO films at different NiSO₄ concentration

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Abstract

In this study, nickel oxide (NiO) films were prepared on indium tin oxide (ITO) glass by a chemical bath deposition (CBD) at different nickel sulfate (NiSO₄) concentrations. The NiO films were verified for their structural properties with field emission scanning electron microscopy (FE-SEM) and X-ray diffraction (XRD) while the optical properties were investigated using a spectrophotometer. Moreover, the NiO films were studied to assess their electrochemical properties by cyclic voltammetry in potassium hydroxide (KOH) electrolyte. The results showed that annealed NiO films exhibited the dominant crystal structure of the (111) plane. Meanwhile, the NiSO₄ concentration controlled morphological structure between dense and porous structures. The porous structure of NiO film was produced with the NiSO₄ concentration in the range of 0.2 M to 1.0 M and the most porous structure was NiSO₄ concentration at 0.8 M with a porosity of 64.56%. The optical contrast was calculated between bleached and colored states which were obtained at a maximum of 51.39% for NiO films at 0.8 NiSO₄ concentration. Morphological effects and electrochromic properties were highly consistent. Analysis of the cyclic voltammetry (CV) results revealed that the cyclic stability for the highly porous structure of NiO films was more deteriorating than the less porous films.

1. Introduction

The electrochromic glass has two states of color and bleach when the applied voltage. Important components of electrochromic glass are the transparent conducting electrode layer, an ion storage layer, electrolyte layer, and electrochromic layer. The electrochromic layer is an important film that undergoes redox reactions and causes the transition from transparent to color states [1]. The ability to change the optical properties depends on the type of metal oxide material used as the electrochromic layer. The popular materials include WO₃ [2-4], MoO₃ [5], and NiO [6-8]. Among these materials, NiO is an attractive material due to its low cost. It is easy to prepare and most importantly, NiO has good color performance and high stability. In addition, NiO has a high capacitance so it can be applied to other applications such as solar cells [9-10], displays [11], and super-capacitors [12-13].

The coloration efficiency and cycle stability of NiO films depend on the film thickness [14-15], temperature [16-17], concentration [5,18-20], preparation techniques [21], etc. Among these, the preparation with different conditions of the precursor significantly affects the morphology, thickness, optical, and electrochromic properties. Bazhan *et al* [18] prepared nickel ferrite (NiFe₂O₄) films by sol-gel method with different Ni/Fe M ratios. The result is that the increasing of Ni/Fe M ratios

enhanced electrochromic properties. S.A. Mahmoud *et al* [19] deposited NiO films by spray pyrolysis method with different precursor solutions of nickel chloride. This result demonstrated that as nickel chloride concentration increased from 0.05 M to 0.10 M, the optical contrast value increased, and then the optical contrast value decreased when nickel chloride increased. Rafia B. *et al* [20] studied the synthesized nickel oxide film by spray pyrolysis with varied solutions of nickel nitrate hexahydrate [Ni(NO₃)₂·6H₂O]. NiO films showed the transmittance value had decreased when solutions of nickel nitrate hexahydrate were increased.

These parameters also affect the morphology, thickness, optical and electrochromic properties of NiO film. NiO films can be prepared easily and in a variety of methods, such as sol-gel [18,21], electrodeposition [7,14], electron beam [8], sputtering [23-24], and chemical bath deposition (CBD) [21-22]. Of all the methods mentioned, the preparation with the CBD method has received a lot of attention because it allows preparation in a large area, at low temperatures, and at low cost. Previous research [21,22] has been prepared with the CBD method. Morphological effects on the electrochromic and electrochemical performances of NiO thin films were demonstrated. Xia *et al* [21,22] studied comparison between CBD and sol-gel, the highly porous NiO films prepared by CBD exhibited much better electrochromic compared to the smoothly compact NiO thin film

prepared by sol-gel. Moreover, annealing at different temperatures of 300°C to 400°C were studied, the NiO film annealed at 300°C exhibited a rather good memory effect and excellent electrochromic properties. This result does not yet show how the effect of the concentration of the preparations process affects them. In this research, NiO films were prepared by the CBD method with different concentrations of NiSO₄ solutions. We have demonstrated the influence of concentration on structural changes and electrochromic properties. Morphological results were used to determine the percentage of porosity that directly affected the electrochromic properties.

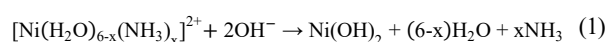
2. Experimental details

ITO glass substrate (2.5 cm² × 1 cm²) was cleaned with acetone, methanol, and deionized water for 15 min each, respectively. The surface of the ITO glass on the nonconductive sides and the conductive side on top for about 5 mm was masked with tape to prevent deposition on the NiO films. The solution for deposition on NiO films was prepared by mixing 40 mL of different NiSO₄ concentrations (0.1, 0.2, 0.4, 0.8, 1.0, 1.2, 1.4, and 1.6 M), 30 mL of 0.25 M potassium persulfate (K₂S₂O₈), and 20 mL of aqueous ammonia (NH₃, 25% to 28%). The ITO glass substrate is suspended in the middle of the solution and held at a temperature of 18°C to 20°C for 20 min. At the specified time, the ITO glass had the tape removed and was washed with deionized water and dried with hot air. After that, as-deposited films annealed in air at 330°C for 1.5 h.

NiO films were subjected to X-ray diffraction analysis to characterize the crystal structure of the nickel oxide films using CuKα radiation at λ of 1.54184 Å (Bruker, D2- PHASER). The morphology of the NiO films was studied by a Field emission scanning electron microscope (FE-SEM, TESCAN MIRA-3, Czech Republic). The transmittance at the bleached and colored states of the NiO film was examined with ultraviolet-visible spectroscopy (UV-Vis; G10S UV-Vis, Thermo Scientific) in the wavelengths between 200 nm and 1100 nm. The coloration efficiency of NiO films was analyzed by CV with Ag/AgCl as a reference electrode and Pt foil as a counter electrode in a 1.0 M KOH solution with a scan rate of 10 mV·s⁻¹ and applying voltages of -1.0 V to +1.0 V.

3. Results and discussion

Figure 1 shows the morphology from FE- SEM images of NiO films with the preparation of different NiSO₄ concentrations at the deposition time of 20 min. The porous structure of the NiO films increased as the NiSO₄ concentrations increased from 0.1 M to 0.8 M, and then the porous structure began to decrease as the concentration increased from 0.8 M to 1.6 M. Therefore, the NiSO₄ concentrations determined the porous structure of the NiO films. The NiOOH films formation is shown by reactions (1) and (2) [22]. The reaction products from equations (1) and (2) were Ni(OH)₂ and NiOOH, respectively. These results corresponded with previous work of Han *et al* [25]. The growth mechanism of NiOOH is initiated by the combination of properly combined Ni(OH)₂ and S₂O₈²⁻. However, increasing the concentration of NiSO₄ resulted in precipitation of a greater amount of Ni(OH)₂ nanoparticles. The amount of difference between Ni(OH)₂ and S₂O₈²⁻ causes limited NiOOH formation and residual Ni(OH)₂ content, resulting in different morphological features.



The NiO films were complete with annealing temperature at 330°C in air for 1.5 h. **Figure 2** shows the X-ray diffraction pattern of annealed NiO films prepared by the CBD with different NiSO₄ concentrations. The XRD result at all conditions showed a small peak at 2θ of 37.26° relative to the (111) plane [20]. The NiSO₄ concentration of 0.8 M and 1.0 M for annealing NiO films exhibited a XRD peak at 2θ of 43.28° with corresponding to (200) plane. The (200) plane is increased and disappears due to different NiSO₄ concentrations, causing strain within the film structure which corresponded to previous work [20,34]. These peaks corresponded to the NiO phase of a face-centered cubic (FCC) [26] with PDF 00-001-1239. Although the X-ray diffraction results exhibited similar characteristics and confirmed the crystal structure of NiO films, these results were unable to account for the different morphological formations based on the FE-SEM photographs and the electrochromic effects that would next explain.

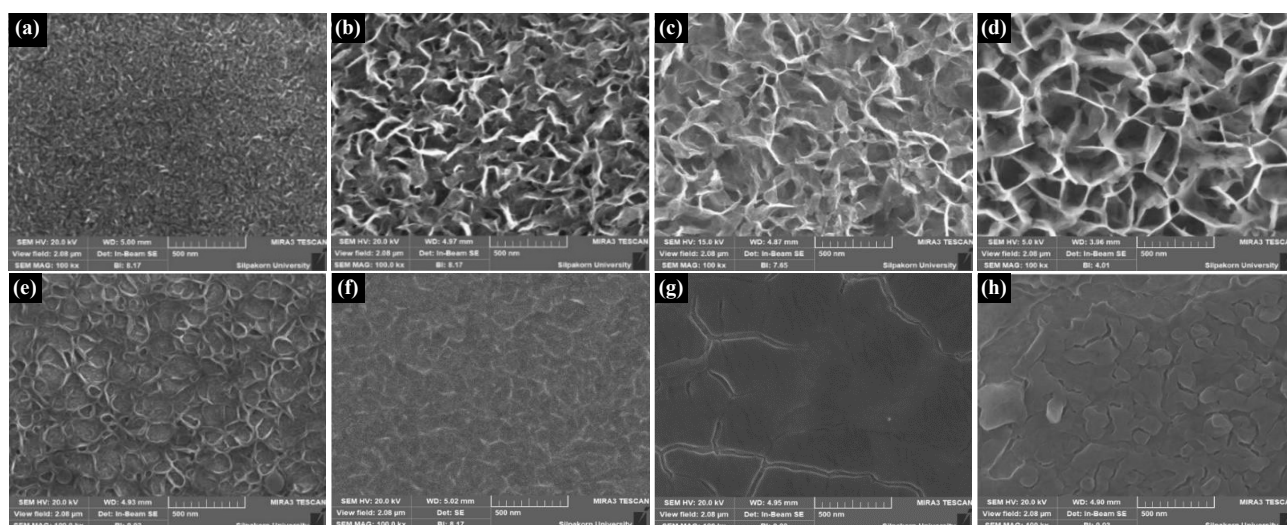


Figure 1. FE-SEM images of NiO films with different concentrations of NiSO₄(a) 0.1, (b) 0.2, (c) 0.4, (d) 0.8, (e) 1.0, (f) 1.2, (g) 1.4 and (h) 1.6 M.

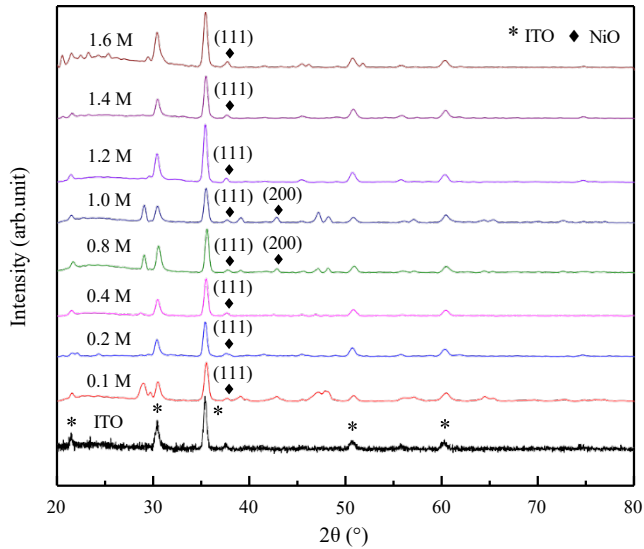


Figure 2. X-ray diffraction of NiO films prepared at various concentrations of NiSO₄ 0.1 M to 1.6 M.

The electrochromic properties of NiO films prepared with different concentrations were studied in KOH solution and the applied voltage ranged from - 1.0 V to + 1.0 V. Figure 3(a) shows the transmittance value at a wavelength of 550 nm for the as-deposited, colored state, and bleached state of NiO films. It was found that the transmittance value of the NiO film at the colored state decreases from a NiSO₄ concentration of 0.1 M to 0.8 M and increases from a NiSO₄ concentration of 1.0 M to 1.6 M. The results were used to calculate the optical contrast ($\Delta\%T$) value between the bleached (%T_b) and colored states (%T_c) with $\Delta\%T = \%T_b - \%T_c$, as shown in Figure 3(b). It was certain that the NiO film with NiSO₄ concentration of 0.8 M has the most $\Delta\%T$ which was 51.39%. This result was consistent with the morphological structure of the nanoscale highly porous film. This provides a greater surface area for ion exchange in electrochromic processes [6]. However, NiSO₄ concentrations at 0.2 M and 0.4 M had a more porous structure compared to 1.0 M and 1.2 M but found similar values of $\Delta\%T$ which decreased when the NiSO₄ concentrations moved away from 0.8 M. In addition, NiO films prepared at NiSO₄ concentrations of 0.1, 1.4, and 1.6 M showed the smallest values of $\Delta\%T$. These results clearly showed that morphology has a significant effect on electrochromic properties.

A common reason for good electrochromic properties is that the film has to be highly charged (electrons and ions) depending on the porosity of the film or its high reactivity contact surface area. In this work, the effect of porosity in NiO films from different preparation conditions was studied. Consider the refractive index (n) of NiO films can be calculated by the transmittance value as equations (3) and (4) [27].

$$n = \sqrt{N + \sqrt{N^2 - n_s^2}} \quad (3)$$

$$N = 2n_s \frac{T_{max} - T_{min}}{T_{max} + T_{min}} + \frac{n_s^2 + 1}{2} \quad (4)$$

where n_s is refractive index of substrate, T_{max} is the maximum of the transmittance curve, and T_{min} is the minimum of the transmittance curve. The refractive index of NiO films is calculated as porosity using the following equation (5) [28].

$$\text{porosity} = \left[1 - \frac{n^2 - 1}{n_d^2 - 1} \right] \times 100\% \quad (5)$$

where n is refractive index of porous film, n_d is refractive index of non-porous material ($n_d = 2.227$) [29]. Figure 4 shows the porosity of NiO films prepared with different NiSO₄ concentrations of 0.2, 0.4, 0.8, and 1.0 M. The results showed the highest porous structure with porosity of 64.56%, where the NiSO₄ of 0.8 M condition corresponds to the SEM image. The relationships of morphology and porosity enhanced the electrochromic properties with maximum optical contrast. Salawan *et al* [30] have clearly demonstrated that dense film compared to rod film structure by oblique angle deposition with sputtering method to increase the surface area of electrochromic property.

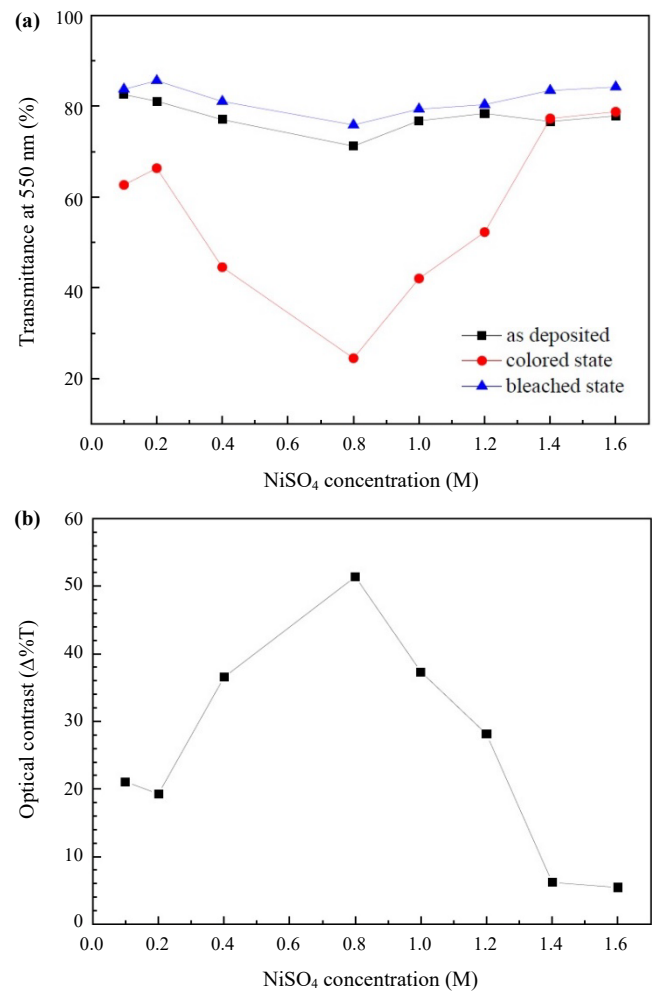


Figure 3. (a) transmittance at 550 nm of as-deposited, bleached and colored state and (b) optical contrast between bleached and colored state at 550 nm.

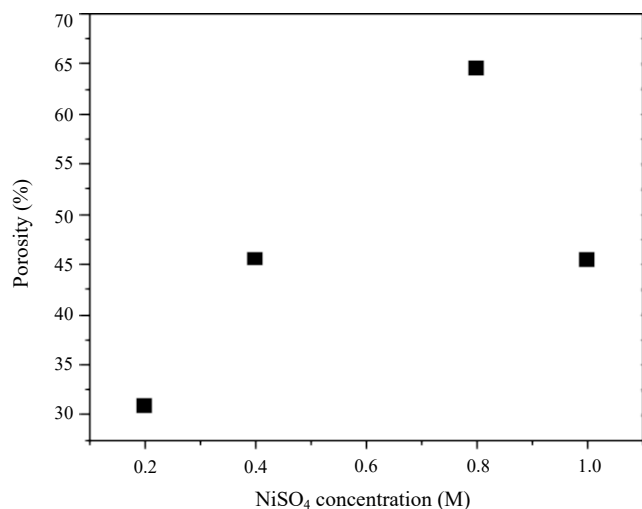
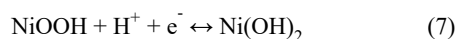
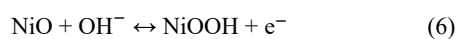


Figure 4. Porosity of NiO films at NiSO₄ concentrations of 0.2 M to 1.0 M.

Moreover, the nanoscale porous network structure was developed to optimize the electrochromic properties [31]. These results are well supported in the development of electrochemical properties.

Figure 5 presents the CV curves of the NiO films at NiSO₄ concentrations for different structures of 0.2 M (slightly porous structure), 0.8 M (highly porous structure), and 1.4 M (dense structure) with a scan rate of 10 mV·s⁻¹ and applying voltages of -1.0 V to +1.0 V. The CV curve can indicate the film's electrochromic performance, including the colorability and stability of the film to the electrolyte solution between switching. The result showed that NiO films exhibited an ascending stability cycle loop for 1 cycle to 10 cycles at conditions of 0.8, 1.4, and 0.2 M, respectively. The deterioration of the NiO film is a result of the electrochromic properties test. The redox reactions during the electrochromic test are as follows (6) and (7)



When there is a reversal between the colored state and the bleached state, NiO is gradually converted to Ni(OH)₂, which causes the films to have more hydration and less area for reaction. After that, the films are degraded [32]. Meanwhile, the area in the CV curve represents the electrochromic performance which was 0.8 M of NiSO₄ concentration and showed the largest area. It is known that the loop area of the CV curve means the charge capacity of the film [8]. These exhibited the most porous structure affinity, with a large area for ion exchange. It clearly showed that as the morphological structure tended from a porous to a dense structure, it showed increasing difficulty to insert the ions in the NiO film. Therefore, a large area in the loop means exchanging ions showed greater results for NiO films of 0.8 M than for 0.2 M and 1.4 M, but the highly porous films also degraded faster than dense films [21,31]. In order to develop a high precipitation structure to exhibit good color reproduction and to be durable, both morphology and crystalline formation are required. This was reported in WO₃ films [31]. In the case of NiO films [11,33] additional doping has been made and remains of interest to future developments.

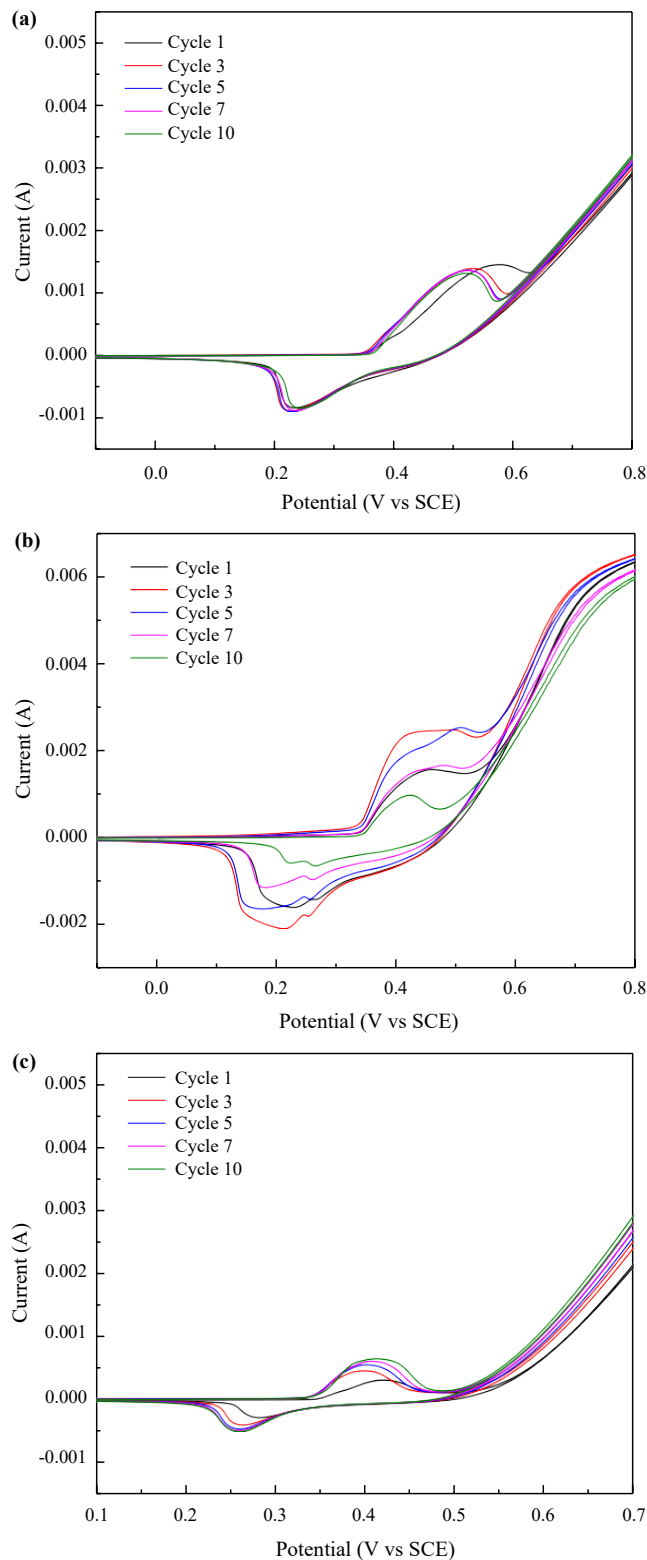


Figure 5. Cyclic voltammogram of NiO films with different NiSO₄ concentration (a) 0.2 M, (b) 0.8 M, and (c) 1.4 M.

4. Conclusions

The morphology of the NiO films was strongly controlled by the concentration of the NiSO₄ solution prepared by the CBD method. Increasing the NiSO₄ concentration from 0.1 M to 0.8 M had the

effect of improving the porous structure while increasing the NiSO₄ concentration from 0.8 M to 1.6 M changed the porous structure to the dense films structure. All NiO films showed crystal structures after annealed in air at temperature of 330°C. At 0.8 M of NiSO₄ concentration exhibited that the NiO films showed the highest porosity of 64.56% and indicated the maximum optical contrast of 51.39%. Moreover, the result of CV curve supported electrochromic property with the most area in CV loop for NiO films at 0.8 M of NiSO₄ concentration. While the stability of the NiO films at 0.8 M of NiSO₄ concentration still needs to be improved. This suggested that depends on the crystallinity and film density or doping of other substances for further restructuring.

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