Synthesis and characterization of NiO for applications in photoelectronics devices

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ABSTRACT

In this work, nickel oxide was synthesized by the Sol-Gel process and deposited on glass substrates using spin coating technique. Three different thicknesses of NiO thin films were obtained: 50 nm, 60 nm and 70 nm, which were subsequently exposed to temperatures of 400°C, 425°C and 450°C for 12 minutes. The characterization was performed by X-ray diffraction and UV-Vis spectroscopy. The X-ray analysis shows a small crystalline structure with high purity NiO. At increasing temperature, a decrease in absorbance can be observed due to structural changes. The synthesized NiO thin films presented a band gap energy of approximately 3.8 eV.

Keywords: Sol-Gel, NiO, Thin Film.
**INTRODUCTION**

Nickel oxide (NiO) is one of the p-type semiconductor materials with optical band gap ranging from 3.6 to 4.0 eV [1]. It has cubic structure with lattice parameter \((a = 0.4816 \text{ nm})\) [2]. Its advantages include high durability and excellent chemical stability in any solvent, low toxicity in water, large optical density range, low cost and good thermal stability [3]. Due to the simplicity in its synthesis, it has diverse applications such as in solar cells [4], chemical sensors [5], photodetectors [6], organic light emitting diodes [7], UV detectors [8], among others.

Nickel oxide thin films have been prepared by various techniques, such as molecular beam epitaxy technique (MBE)[9], electrochemical deposition [10], chemical vapor deposition [11], sol-gel process [12], thermal evaporation [13], pulsed laser deposition techniques (PLD) [14], magnetron sputtering technique [15], spray pyrolysis technique [16] and sputtering [17]. In the present work, NiO was synthesized by the sol-gel process. Thin films of the synthesized material were deposited on glass substrates using the spin coating deposition method and submitted to thermal treatment before optical and electrical characterization. In addition, the influence of the film thickness on the structural and optical characterizations was investigated.

**OBJECTIVE**

The objective of this work is to synthesize Nickel Oxide via sol-gel process, deposit thin films of this material and characterize them using X-ray diffraction and UV-Vis spectroscopy after submitting the samples to a thermal treatment. It is expected that such investigation provides supporting information for future applications of NiO thin films on electronic devices, such as solar cells, sensors, photodetectors, among others.

**EXPERIMENTAL METHODS**

**Synthesis of NiO**

The reagents nickel acetate tetrahydrate, and diethanolamine from Sigma Aldrich were used as received. Nickel oxide was synthesized by sol-gel method using 1 g of 98% \(\text{Ni(OCOCH}_3)\text{\textsubscript{2}}\cdot4\text{H}_2\text{O}\) nickel acetate tetrahydrate as Ni source, adding 10 mL of methyl alcohol as solvent and stirring vigorously on a magnetic stirrer for 30 min without heating. Subsequently, 380 µL of diethanolamine \((\text{C}_4\text{H}_{11}\text{NO}_2)\) was added dropwise stirring for 30 min, observing that the mixture takes an intense green color. Finally, the solution is filtered with a 5 mL syringe,
adjusting in its tip a 0.20 \( \mu \text{L} \) filter, thus obtaining nickel hydroxide. Figure 1 schematizes the whole process of nickel hydroxide synthesis.

**Figure 1. Synthesis of nickel hydroxide.**

Deposition of NiO as Thin Films

The preparation of NiO thin films was performed on glass substrates, which require prior cleaning before use; the steps followed for such cleaning are as follows:

a) the glasses were placed on a support with channels, to avoid contact with the bottom of the beaker and prevent possible scratches on the surface of the glass.

b) The support with the glasses was placed in a 250 ml beaker, adding acetone until the glasses were well covered and initiating the cleaning process by shaking baths with ultrasound for 20 minutes; then, we repeated the same process with ultrapure deionized water (Mili-Q, resistivity 18.2 MΩ at 25°C) and isopropyl alcohol, respectively.

c) to finish, we dried the glasses with nitrogen. Once the above process was finished, the NiO was deposited on glasses using the sping coating technique.

Three different thicknesses were obtained by depositing 50 \( \mu \text{l} \) of nickel hydroxide solution on the substrate and spinning it for 30 s at three different rotation speeds: 2000 rpm, 2500 rpm and 3000 rpm. The thicknesses obtained were 70 nm, 60 nm and 50 nm, respectively. The thin films obtained were subsequently subjected to 3 levels of temperatures in a horizontal tube furnace, for each thickness, resulting in 9 samples of different conditions. Table 1 summarizes the values obtained.
Table 1. NiO films deposited by Sping Coating Technique.

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<tr>
<th>Deposition (µl)</th>
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Characterization

Wide-angle X-ray diffraction (XRD) patterns were recorded with a Shimadzu XRD-7000 diffractometer operating at 40 kV and 20 mA (Cu – Kα radiation λ = 1.5418Å) with monochromator (scan rate of 2°min⁻¹) in the 2θ angle range from 30° to 70°, from which it was possible to determine the crystalline phase composition. UV-Vis transmittance spectroscopy was made using a UV visible spectrophotometer Perkin Elmer Lambda 1050.

RESULTS AND DISCUSSION

Structural Analysis

Figure 2. shows the X-ray diffractograms of NiO thin films heated at 400°C, 425°C and 450°C. Well-defined peaks with orientations in the (222), (400) and (440) crystal planes are observed. The absence of impurity peaks suggests the high purity of the nickel oxide; furthermore, it can be seen from the figure that the peaks are relatively broadened, indicating that the deposited material has a very small crystallite size [18], which increases with increasing temperature.

Figure 2. X ray diffraction patterns of NiO film at different temperatures.
Optical study

From the absorption spectrum of the NiO thin films, the absorption coefficient $\alpha$ was calculated using equation (1):

$$\alpha = \frac{A}{0.4343 \cdot d} \hspace{1cm} (1)$$

$A$ is the absorbance and $d$ is the thickness of the film and presented on Figure 3(a). It was observed that with increasing thermal treatment temperature there is a decrease in the absorbance attributed to changes in the crystalline structure when exposed to different temperatures. In general, the absorbance of the films has low values in the visible and near infrared region, because of its semiconductor character. By this way, it is possible to estimate the optical band gap energy using Tauc’s method [19], as shown in Figure 3(b). The values obtained are 3.77 eV, 3.79 eV and 3.80 eV. These results are in agreement with theoretical calculations made for the direct band gap energy of NiO [20]. This variation in energy taken by the NiO thin films is due to the possible structural changes that the films undergo when exposed to higher temperatures.

![Figure 3. a) Absorption coefficient and b) estimated band gap energy of NiO.](image)

**CONCLUSIONS**

X-ray measurements showed that the crystalline structures of the NiO thin films are small and of high purity, which suggests that the method used for the synthesis is adequate for thin film formation. UV-Vis spectroscopy showed that the NiO thin films have good transparency in the visible region of the spectrum, a situation that favors their application in photoelectronic devices. The variation in the values obtained in the band gap energy is due to the possible structural changes that the films undergo when exposed to higher temperatures.
REFERENCES


