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STRUCTURAL AND OPTICAL PROPERTIES OF (ZnO/NiO) THIN FILMS MIXTURE[†]

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In this study, we prepared mixtures of nickel oxide (NiO) and zinc (ZnO) in different proportions as thin films on high-purity glass substrates, using pyrolysis spray technique. Where samples of mixtures were precipitated from two solutions of nickel nitrate ($Ni(NO_3)_2 \cdot 6H_2O$) and zinc acetate ($C_4 H_6O_4 Zn \cdot 2H_2O$) mixed in different proportions. Then the optical and structural properties of the prepared samples were studied. The transmittance decreases with the increase in the percentage of nickel oxide, which means that increasing the zinc oxide improves the transmittance in all the studied spectral fields. Samples with higher zinc oxide (ZnO) have two energy gaps. Scanning electron microscopy (SEM) showed that the surface morphology of the films has a relatively homogeneous composition. Where it was observed that increasing the proportion of zinc oxide leads to the appearance of zinc oxide granules clearly.
Keywords: *Mixtures of NiO and ZnO; Thin films; Pyrolysis spray technology; Energy gap; Optical properties*
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1. INTRODUCTION

ZnO and NiO thin films are among the most important films that have been scientifically and industrially exploited for their important applications in wide fields. Zinc oxide (ZnO) is a transparent n-type semiconductor with a wurtzite crystal structure. It has good potential in developing optoelectronic device technologies, mainly due to its outstanding physical properties, such as its high transparency in the visible spectrum and near infrared region [3, 4]. It has a bandgap ranging from (3.2-3.3) eV [5] and has a high binding energy [6, 7]. The outstanding optical and electrical properties of zinc oxide films have led to their use in many applications such as: solar cells [8, 9], gas sensors [10], piezoelectric sensors [11, 12], flat screens [13], acoustic devices [14], and optical waveguides [15], and laser diodes [16]. ZnO thin films can be produced by several techniques such as: spray pyrolysis [17, 18] hydrosol gel, thermal evaporation [19], and laser ablation (PLD) [20]. Nickel oxide (NiO) is a transparent p-type semiconductor that has a crystal structure (CFC) similar to that of sodium chloride (NaCl) and has a band gap between 3 and 4 eV [21].

Nickel oxide (NiO) is an attractive material due to its excellent chemical stability, good dynamic range and hardness, as well as its optical, electrical and magnetic properties [22]. Nickel oxide has important and extensive applications such as optical switching glass, antimagnetic materials, photoelectric display devices, lithium batteries, and functional layer materials for chemical gas sensors [23]. In addition, nickel oxide films oriented in the (111) direction can be used as dielectric layers that are deposited on oxide films of other orientations, such as C-oriented perovskite-type ferromagnetic films and superconducting films [00]. NiO films can be deposited using many different chemical and physical methods such as electrochemical deposition [24], thermal evaporation [25], ion beam deposition [26], laser ablation [27], spray pyrolysis [28] and gel coating by centrifugation [29].

In this work, we will prepare samples for mixtures of the aforementioned oxides in order to study their structural and optical properties in order to obtain the optimal mixture that has better properties than both oxides separately.

2. EXPRIMENTAL WORK

In this paper, mixtures of nickel oxide (NiO) and zinc oxide (ZnO) were prepared in the form of thin films on ultra-pure glass substrates using pyrolysis spray technique. Where two solutions were prepared from nickel nitrate ($Ni (NO_3)_2 \cdot 6H_2O$) and zinc acetate ($C_4H_6O_4Zn \cdot 2H_2O$) with the same molar concentration C=0.2 mol/L, by dissolving each mass of nickel nitrate and Zinc acetate in a volume of 12 ml of distilled water and add drops of chloride acid in order to facilitate the mixing process. To ensure complete dissolution of both solutions, a magnetic stirrer is used to mix each solution for 15 minutes at a temperature of 50ºC ensuring no sediment. Then, we prepare samples from the mixture consisting of nickel nitrate solution and zinc acetate solution in different proportions as shown in the table1.After preparing the precipitation mixture, preparing the glass substrates and preparing the spraying system, we start the precipitation process with the chemical spraying technique, which passes through several steps:

1- We put the spray nose at the vertical distance d = 30 cm.
2- We set the air compressor to a pressure of 2 bar.

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3- The glass substrates are placed over the electric heater and set at a temperature of 500°C.

4- After heating, we do intermittent spraying for 5 seconds to keep the temperature of the glass substrate within 500°C. Where we obtained thin films with acceptable homogeneity and high adhesion strength on the substrate and with close thickness.

3. RESULTS AND DISCUSSIONS

3.1. Structural Properties

Figure 1 shows the XRD patterns of films of a mixture of nickel oxide (NiO) and zinc oxide (ZnO) prepared with different proportions of aforementioned oxides. In case of pure nickel oxide curve S_1 it can be seen two main diffraction peaks positioned at 2θ ≈ 37.296° and 43.272° assigned to the (111) and (200) crystal planes respectively.

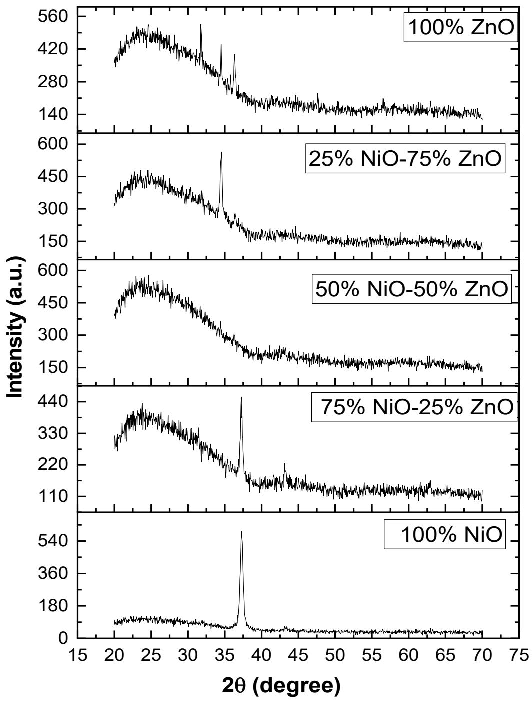

Figure 1. XRD patterns of the deposited ZnO/NiO thin films on glass substrate at different percentages

These peaks accurately correspond to the crystal structure of NiO (NaCl) with CFC crystal structure of which belongs to the space group Fm3m. The XRD patterns shows good agreement with JCPDS (47-1049) [1]. The presence of such peaks indicates that the films are polycrystalline in nature.

The same results can be deduced for sample S_2 where it is observed that three main peaks are identified in the angle range 2θ (37.247°, 43.197°, and 62.844°) corresponding to the following diffraction planes (111), (200) and (220), respectively. These results are consistent with the high percentage of nickel oxide present in this sample. In the case of equal proportions of oxides in the sample S_3, no diffraction peaks were observed, and the sample was amorphous. For sample S_4 two specific peaks appear at angles 2θ (34.515° and 36377°) corresponding to the diffraction levels (002) and (101) according to JCPDS (36-1451), which is consistent with a high percentage of ZnO in these sample. Sample S_5 showed three prominent main diffraction peaks (100), (002) and (101) corresponding to the angle (31.564°, 34.510° and 36.336°). These peaks correspond to the wurtzite hexagonal structure of pure ZnO. It is noted that there is a high compatibility with the international s showed three prominent main diffraction peaks (100), (002) and (101) corresponding to the angles 2θ (31.56°, 34.51° and 36.33°). These peaks correspond to the wurtzite hexagonal structure of pure ZnO. It is noted that there is a high compatibility with the international standard card JCPDS (36-1451).

In order to get more material structural information, we have studied different structural constants such as lattice constants a and c, grain size D, average stress ε and dislocation density δ for pure NiO and ZnO films as well as for the films of oxides mixtures. The lattice constant for the cubic structure phase of NiO films is calculated using the following equation [3]:

$$d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}} \quad (1)$$

Where is the distance between the crystal structure and (h,k,l) are the Miller's coefficients.

The lattice constants a and c for the hexagonal structure phase of ZnO films is calculated using the following equation [4]:

$$\frac{1}{d^2} = \frac{4}{3}\left(\frac{h^2+hk+k^2}{a^2}\right) + \frac{l^2}{c^2} \quad (2)$$

The grain size D of films is related to the physical and chemical properties of the material, where the Debye-Scherrer statement allowed estimating the size of the grains by X-ray diffraction, which is given by the following relationship [5]

$$D = \frac{0,9\lambda}{\beta \cos\theta} \quad (3)$$

Where λ is the wavelength of X-rays (λ=1,540593), β is the value of the middle of the highest peak width (the value (FWHM) computed with the radial angle) andθ is the Bragg's angle.

Dislocation density δ represents the linear defect inside the crystal, and is calculated from Williamson and Smallman's relationship [6]:

$$\delta = \frac{1}{D^2} \quad (4)$$

The average strain ε is the strain expresses the distortion in the network and is estimated by the following relationship [7]:

$$\varepsilon = \left(\frac{a - a_0}{a_0}\right) 100\% \qquad (5)$$

Where a_0 is the theoretical lattice constant and a is the experimental lattice constant.

Table 1. Structural parameters of (ZnO/NiO) thin films mixture

Samples	Coding	2θ(°)	d (Å)	(hkl)	a (Å)	c (Å)	D (Å)	μ_ε	δ (Å^{-2})
100% NiO	S_1	37,296	2,411	111	4,176	------	3,32	0,104	0,091
		43,272	2,091	200					
75% NiO 25% ZnO	S_2	37,247	2,414	111	4,181	------	4,44	0,078	0,051
		43,197	2,094	200					
		62,844	1,479	220					
25% NiO 75% ZnO	S_4	34,515	2,600	002	3,24	5,200	5,59	0,062	0,036
		36,377	2,470	101					
100% ZnO	S_5	31,564	2,834	100	3,272	5,198	4,82	0,102	0,043
		34,510	2,599	002					
		36,336	2,472	101					

3.2. Morphological Properties

The surface morphology of the thin film was studied by scanning electron microscopy (SEM). For films of pure nickel oxide NiO and zinc oxide ZnO, as well as samples consisting of a mixture of NiO and ZnO in different proportions which have been sputtered onto glass substrates, where it appears that the deposited films have a composition and a relative homogeneity despite the preparation of homogeneous solutions for the samples because they contain pure grains and prepared under the same conditions. Hence the shape of the granules which does not appear clearly with a vertical growth on the surface of the film for samples S_1 and S_2which contain a higher percentage of nickel oxide. It should be noted that the higher the percentage of ZnO and this for samples S_3, S_4 and S_5 the more clearly the zinc grains appear on the surfaces of the thin films and the development of the skeleton with the random growth of islands in the middle and on the edges of the substrate.

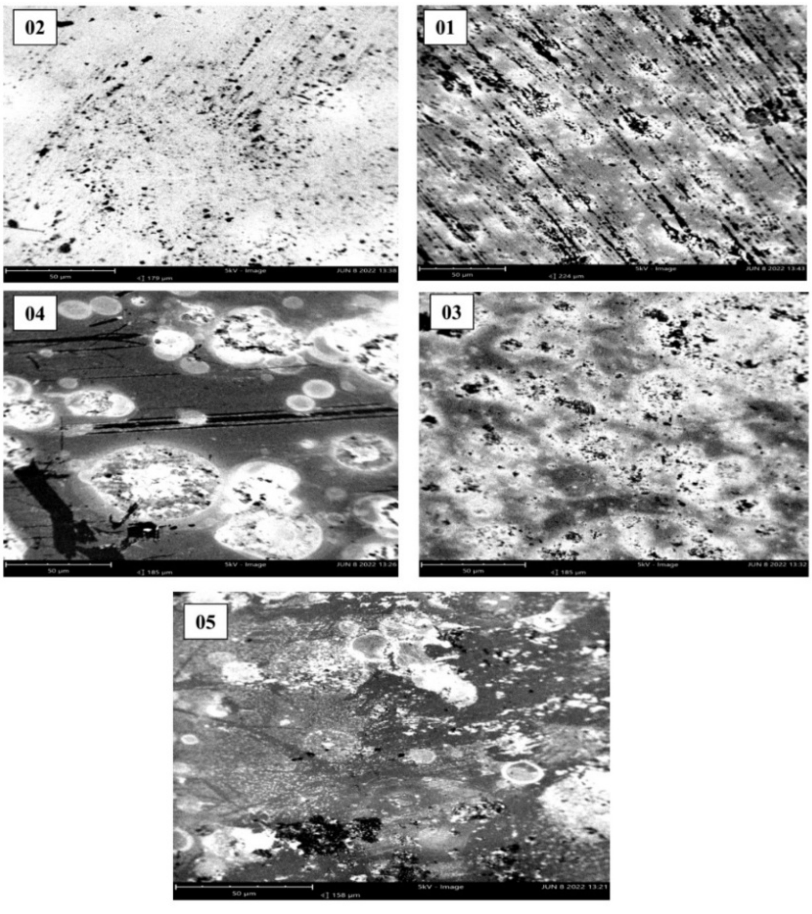

Figure 2. Images of samples S_1 – S_5 prepared by scanning electron microscopy (SEM)

3.3. Optical Properties

The absorbance and transmittance spectrum of all samples were studied in the ultraviolet, visible and near infrared rays. The values of the absorption coefficient, energy gap and Urbach energy are summarized in Table 2.

Figure3shows the transmittance changes with wavelength for all prepared samples. The results showed that the transmittance is as low in the ultraviolet region, i.e., in the short wavelength range of 300 to 380 nm. The low value of transmittance in this area is due to the fact that the absorption is high due to the proximity of the energy of the emitted photons to the energy of the basic absorption edge of these films [30]. The transmittance of the two samples S_1 and S_2 moves towards the blue spectrum, while the rest of the samples S_3, S_4 and S_5 moves towards the red spectrum.
The transmittance increases significantly for all samples in the visible range, especially for samples that contain a higher percentage of zinc oxide, as it is close to 98% at the end of the visible range for samples S_3, S_4 and$S_5$and 88% for the two samples S_1 and S_2[31].

Table 2. Structural parameters of (ZnO/NiO) thin films mixture

Samples	Optical gap energy E_g(eV)		Urbach energy **E_U (eV)**
S_1	3.55		0.466
S_2	3.53		0.571
S_3	2.35	3.13	0.333
S_4	3	4.1	0.133
S_5	3.23	3.17	0.076

The optical band gap energy is one of the fundamental properties of electro-optical materials. The measurement of the gap energy depends on the material and its properties. The optical energy bandgap is defined as the lowest energy required to move an electron from the top of the valence band to the bottom of the conduction band. By determining the values of the absorption coefficient, it is possible to calculate the optical band gap of the permissible direct electronic transitions using Tauc's equation [32]:

$$(\alpha h v)^2 = A\left(h v - E_g\right). \tag{6}$$

Where A is a constant, α is the absorption coefficient*,* hv is the photon energy and E_g is the band gap energy.

Table 2 shows a significant difference in the band gap energy values of the thin films of the mixture of nickel and zinc. This difference can be attributed to the phase presence for both oxides, and since there is a clear difference between the nickel and zinc elements in chemical and physical properties, the strong presence of one of them means that the other plays a role doping, and therefore we notice a clear difference in the values of the energy gap, especially in the case of samples S_3, S_4 and S_5, where there is the largest presence of zinc oxide, and perhaps the appearance of a double energy gap is a clear indication of this.

It is noted that the energy gap of the thin layers of pure nickel oxide $S_1$and the mixture S_2 decreases to 2.35 eV, the decrease in the band gap energy is explained by the movement of the absorption front towards the lower energies, and this decrease can be explained by Roth effect because the impurities that make up the mixture have led to the formation of donor levels within the band gap near the conduction band and therefore to the absorption of low energy photons [35].

The Table 2 also shows for the samples S_3, S_4 and S_5 which contain a higher percentage of zinc oxide ZnO that they have two band gap energy and this is due to several reasons:

- This may be related to the formation of ZnO nanostructures on the bulk of the remaining ZnO thin film.
- This can also be explained by a defect in the composition and structure of our prepared sample [36].
- There are mixed phase or low crystallization [37].

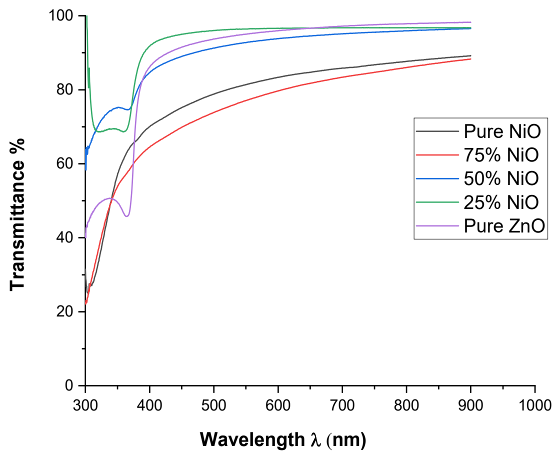

Figure 3. Transmittance of ZnO/NiO thin films

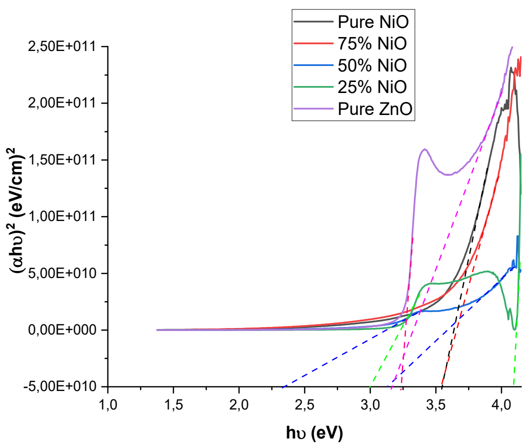

Figure 4. Band gap energy of ZnO/NiO thin films

The Urbach energy is a physical quantity that characterizes the disorder of matter, since it is considered one of the important constants that characterize the optical properties of thin layers [36], since it relates to the absorption coefficient according to relationship [37]:

$$\alpha = \alpha_0 . e^{\left(\frac{hv}{E_u}\right)}. \quad (7)$$

It can be written:

$$ln\alpha = ln\alpha_0 + \left(\frac{hv}{E_u}\right). \quad (8)$$

Where α_0 is the absorption coefficient for which the absorption value is minimum and E_u is the Urbach energy.

The value of Urbach's energy (E_u) can be determined from the drawing of the curve of the changes of the linear function (lnα) in terms of photon energy (hv) shown in Figure5, where the inverse of the slope represents the Urbach energy. There is an increase in the Urbach energy values for the samples of pure nickel oxide S_1 and S_2 which contain a greater percentage of nickel oxide. For the samples (S_3, S_4 and S_5), we find the opposite from what was obtained for the Urbach energy values for the first two samples. From the results of the Table2, we note that the optical behavior of the Urbach energy value changes inversely with the optical behavior of the energy interval.

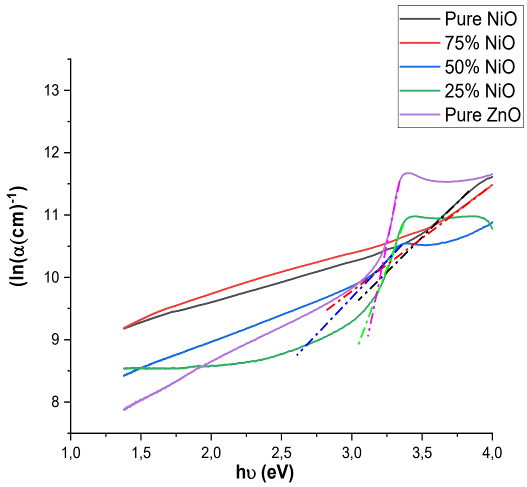

Figure 5. Urbach energy of ZnO/NiO thin films

4. CONCLUSIONS

In this study, we had the opportunity to prepare mixtures of different proportions of samples of nickel oxide (NiO) and zinc oxide (ZnO), which were deposited in the form of thin films on glass substrates, using pyrolysis spray technique at the same conditions.

The structure of the prepared mixtures, as well as the absorption and transmittance spectrum of all samples in the ultraviolet, visible, and near infrared, were studied. Where the values of the absorption coefficient, energy interval, and Urbach energy were summarized with the aim of deducing the relationship between these values and the prepared samples. Where the study showed:

- Samples that contain a higher percentage of nickel oxide (NiO) have the lowest possible transmittance in the ultraviolet region i.e., the short wavelength ranges from 300 to 380 nm while the absorption is high.
- The transmittance of samples with a higher percentage of NiO shifts towards the blue spectrum, while the transmittance of the remaining samples shifts towards the red spectrum.
- The transmittance increases dramatically for samples in the visible range, especially for samples that contain a greater percentage of zinc oxide, as it approaches 99% at the end of this range.
- Samples that contain a higher percentage of zinc oxide (ZnO) have two energy gaps.
- The optical behavior of the Urbach energy value changes inversely with the optical behavior of the energy gaps.
- The surface morphology of thin films has been studied by scanning electron microscopy (SEM), and it has been found to have a relatively homogeneous composition, where the shape of the granules does not appear clearly with a vertical growth on the surface of the film for the sample that contains a higher percentage of nickel oxide. It is noted that as the percentage of zinc oxide ZnO increases, the zinc granules appear on the surfaces of the thin films clearly, and the skeletal structure grows with the growth of random islands.

Through this general study it is clear that the determination of the optimal mixture depends on the application to which the mixture is directed. For example, in solar cell applications where we use transparent oxides as windows, the optimal mixture is one with greater transparency and an energy gap that fits the solar spectrum. In this case, sample S_4 is the best. Whereas in gas sensor applications where film morphology has a key role in optimal adsorption of gases with the film surface, in this case sample S_3 is ideal because the surface profile is more homogeneous and rougher.

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СТРУКТУРНІ ТА ОПТИЧНІ ВЛАСТИВОСТІ ТОНКІХ ПЛІВОК ІЗ СУМІШІ (ZnO/NiO)

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У цьому дослідженні були підготовлені суміші оксиду нікелю (NiO) і цинку (ZnO) у різних пропорціях у вигляді тонких плівок на скляних підкладках високої чистоти за допомогою техніки піролізу. При цьому зразки сумішей осаджували з двох розчинів нітрату нікелю ($Ni(NO_3)_2{\cdot}6H_2O$) і ацетату цинку $C_4H_6O_4Zn{\cdot}2H_2O$), змішаних у різних пропорціях. Потім були досліджені оптичні та структурні властивості підготовлених зразків. Виявлено, що коефіцієнт пропускання зменшується зі збільшенням відсотка оксиду нікелю, що означає, що збільшення оксиду цинку покращує пропускання в усіх досліджуваних спектральних полях. Зразки з вищим вмістом оксиду цинку (ZnO) мають дві енергетичні щілини. Методом скануючої електронна мікроскопія (SEM) показано, що морфологія поверхні плівок має відносно однорідний склад, де спостерігалося, що збільшення частки оксиду цинку призводить до появи гранул оксиду цинку.
Ключові слова: *суміші NiO та ZnO; тонкі плівки; технологія піролізу; енергетична заборонена зона; оптичні властивості*