



Effect of Indium Doping on the Structural and Surface Properties of (Nio) Nickel Oxide thin Films synthesized by Spray pyrolysis Method

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ABSTRACT

In this study, thin films of pure and doped (Nio) nickel oxide doped with indium (3% and 5%) were deposited on glass substrates at a temperature of about 400 °C by spray pyrolysis method.. X-ray diffraction (XRD) and energy-dispersive X-ray spectroscopy (EDX) were used to study the structural properties of the samples. The results indicate that the growth of the films occurs in the preferred orientation of the (111) plane, and no peak suggesting the presence of indium was observed in the samples. Morphological analysis of the samples was performed using atomic force microscopy (AFM), which showed that surface roughness increases with higher doping levels. Additionally, field emission scanning electron microscopy (FESEM) analysis was conducted to further elucidate the structural and crystalline properties .

1. Introduction

Thin films are layers whose thickness is between 50 and 5000 angstroms. In other words, thin films. They are atomically precisely designed layers of various materials, including metals, insulators, and semiconductors[1]. Thin films can be classified as nanostructured coatings. Also, the main application of these thin films is in modifying the surface properties of solids[2]. Nickel oxide is one of the most widely used derivatives of the metallic element nickel. Nickel oxide is a semiconductor material with a large direct energy gap of about 3.67 to 4 eV [3]. The appearance of this material is in the form of a green powder and is a non-toxic material. Nickel oxide can be used as an electrochromic layer in the manufacture of solar cells, gas turbines and electronic components[3,4]. Many studies

have been conducted on nickel oxide thin films and their results have been reported [5] investigated the electrochromic properties of porous nickel oxide thin films prepared by chemical bath deposition. [6] investigated the electrochromic properties of nickel oxide thin films prepared by immersion deposition. [7] studied the electrochromic properties of nickel oxide thin films prepared by chemical vapor deposition. [8] used nickel oxide as the main adsorbent layer in the cell structure used dye-sensitized solar cells [9] used nickel oxide to fabricate a light-emitting diode. Singh et al. used nickel oxide in the diode structure. [10] used nickel oxide and zinc oxide to fabricate a PN junction [11] investigated a thin layer of nickel oxide as a gas sensor. [12] used a thin layer of nickel oxide as a hydrogen gas sensor. In these studies, the sensing agent was the change in conductivity.

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2. Experimental Work

2.1 Substrate Cleaning

Glass substrates, cut to 2cm x 2cm, were used to deposit the desired sample. Before the deposition process began, the substrate was first washed with a detergent, then rinsed with deionized water. The substrate was then washed once with ethanol and again with acetone using an ultrasonic device, followed by rinsing with deionized water. Finally, the substrate was dried using nitrogen gas.

2.2 Preparation of thin films of pure nickel oxide.

The substrate temperature was set to 450 degrees Celsius. After reaching the desired temperature, 100 cm³ of the prepared solution was sprayed onto the glass substrates at a rate of 5 ml/min. After completing the coating process, the temperature was slowly reduced to room temperature, and then the samples were removed for further analysis.

2.3 Preparation of a solution for preparing indium-doped nickel oxide layers.

The indium-doped nickel oxide (NiO:In) precursor and indium chloride were used to prepare the required solution. To investigate the effect of the impurity concentration on the structural and morphological properties of the thin nickel oxide layers, two solutions with different impurity concentrations (3% and 5% w/w) were prepared. The concentration of the working layer solution, like a pure sample at 0.1 M, was soluble in distilled water.

2.4 Fabrication of Nickel Oxide doped with indium by chemical spray pyrolysis.

Similar to thin film of pure nickel oxide, the temperature of the substrate was chosen 450 degrees Celsius. After reaching the desired temperature, the volume of 100 cc from the solution layer of the paste was sprayed with 5 ml / min on to(2cm x 2 cm) glass substrates that had been thoroughly cleaned and dried the substrates. After finishing the deposition process, the substrate temperature was slowly decreased to the ambient temperature and then the samples were removed for further analysis.

3. Result & Discussion

3.1 X-ray diffraction(XRD) analysis:

X-ray diffraction (XRD) analysis was used to examine the crystal structure of the samples. The spectra obtained are in agreement with the standard JCPDs card number 9374-001 of nickel oxide material. As can be

seen in Figure (1), in all samples, the preferred crystal growth is in the direction of the (111) plane and no change is observed with doping, and only the intensity of this peak decreases.

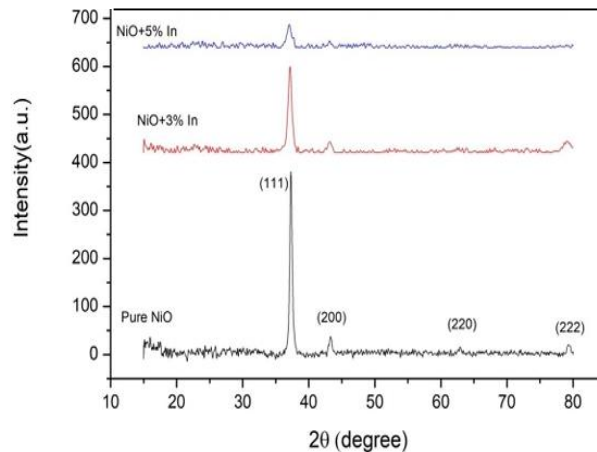


Figure 1. XRD patterns of pure nickel oxide films and doped with indium (3% , 5%)

Using the results of this analysis and the following relations [13], the average crystal size and strain in the samples were calculated and presented in the table.(1) is given.

$$D = \frac{K\lambda}{\beta \cos(\theta)} \dots\dots\dots(1)$$

$$\varepsilon = \frac{\beta}{4 \tan(\theta)} \dots\dots\dots(2)$$

In these relationships, D is the crystal size, a K constant value equal to 0.9, the λ wavelength of the X-ray, β is the width at half the peak height in the diffraction pattern, θ is the angle between the direction of the incident X-ray and the surface of the sample, ε and the strain present in the sample

Table 1 Crystal size and strain of samples

Crystal size (nm)	Dimensionless strain	sample
21.3	5.08×10^{-3}	Pure sample
15.2	7.14×10^{-3}	sample with contamination 3%
15.2	7.17×10^{-3}	sample with pollution 5%

Strain indicates the difference in the unit cell of the sample and the standard sample. With increasing impurity and the presence of indium ions in the crystal

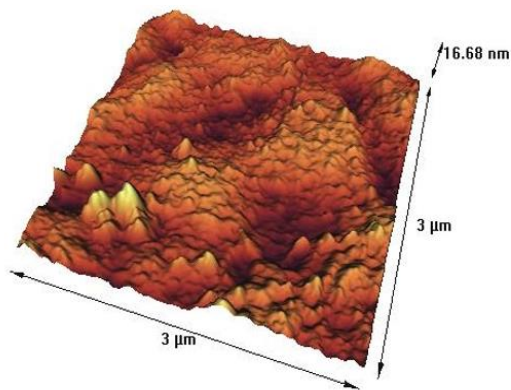
structure of nickel oxide, this difference increases and, as can be seen in the table, the strain value increases.

3.2 Atomic force microscopy (AFM) analysis:

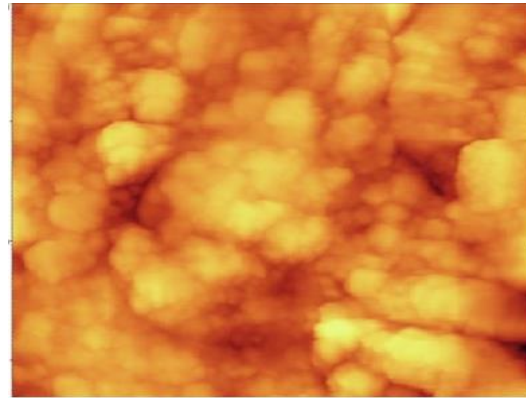
All images were prepared in non-contact mode. Using this analysis, two-dimensional and three-dimensional images of the sample surface were prepared for morphological studies, and the surface roughness of the samples was examined. It was found that there is a change in the surface morphology due to the addition of doping. The surface roughness values for the undoped sample and the samples doped with 3% and 5% respectively were obtained as follows: 0.364, 0.398, and 0.908. This indicates that pure nickel oxide has a smooth surface, whereas with 3% In, enters the NiO crystal lattice, which increases the number of nuclei, leading to a slight increase in grain size, making the surface rougher. At 5% In, the increased indium causes distortion of the crystal lattice and grain agglomeration, resulting in increased surface roughness, which is consistent with the morphological and structural analyses using FESEM and XRD.



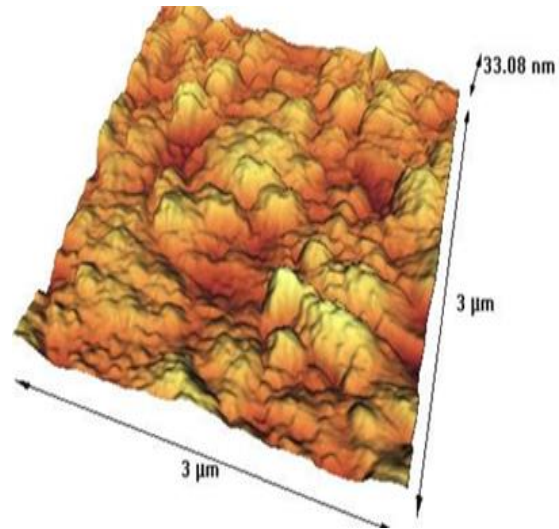
(A1)



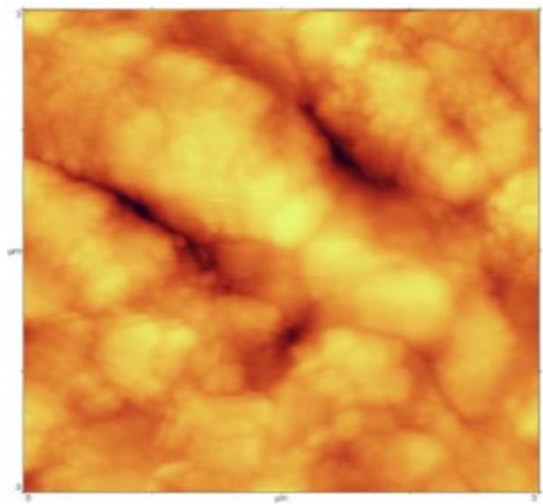
(A2)



(B1)



(B2)



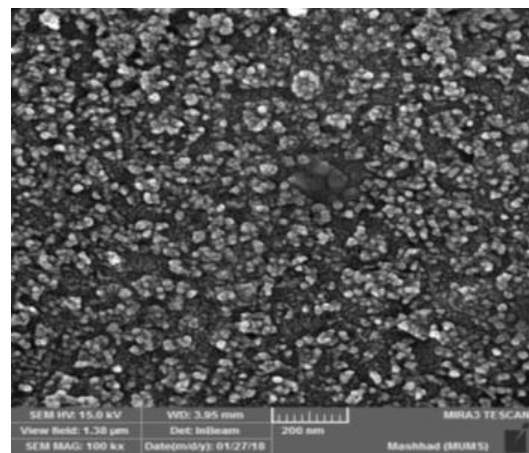
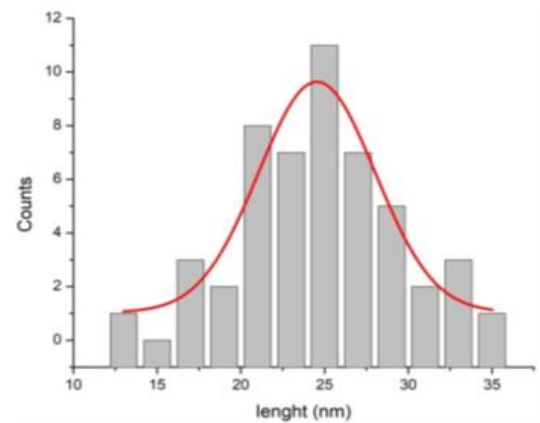
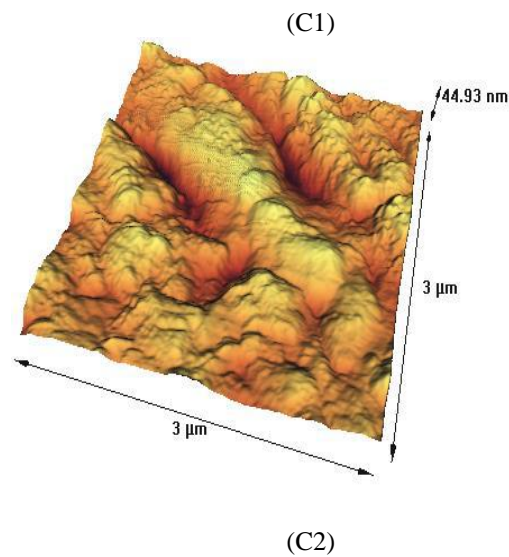


Figure 2. 2D and 3D- FAM images of a thin layer of (A1,A2) pure Nio (B1,B2)) Nio doped with 3% In,(C1,C2) Nio doped with 5% In.

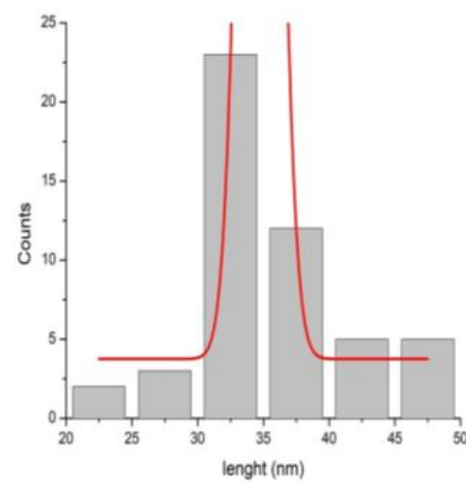
Finger 3 . FESEM image and the particle size diagram of pure distribution oxide prepared nickel thin film.

3.3 FE-SEM Surface morphology analysis:

According to the FESEM images taken from the surface of the samples, the nanostructure of the layers can be observed. The layers are made of nano-sized grains. According to the grain size graphs in terms of abundance, the average grain size was determined for each sample and reported in Table (2).

Table 2. seed size

Grain size (nm)	sample
24.5	Pure sample
34.7	sample with contamination 3%
14.36	sample with pollution 5%



To determine the thickness of the layer, a cross-sectional FESEM image was obtained from the samples. The thickness of all samples is about 150 nm and the cross-sectional image of one of the samples is shown in Figure (7).

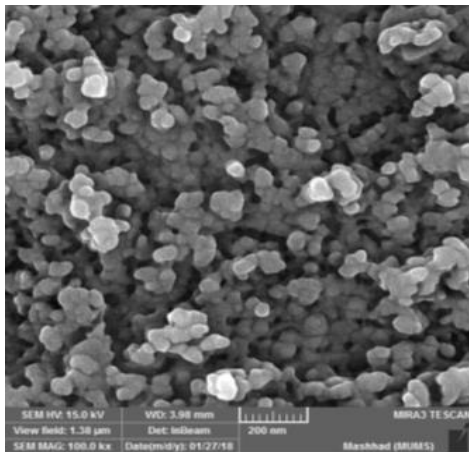
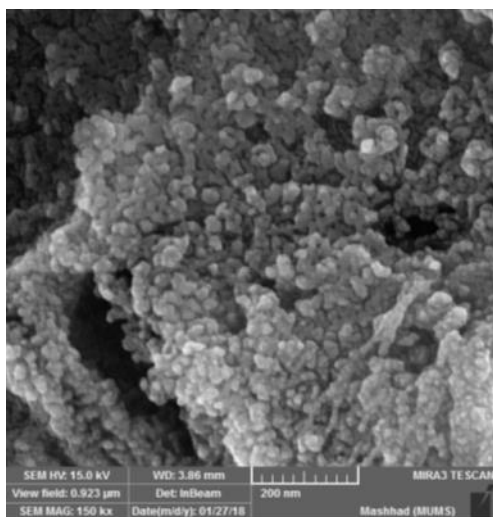
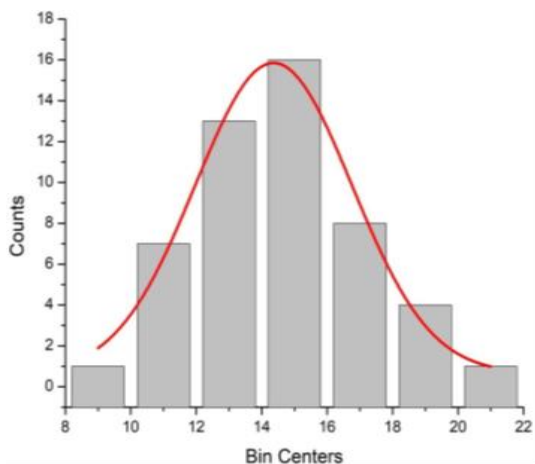


Figure 4 : FESEM image and particle size the distribution diagram layer of nickel oxide of thin with 3% doped In



Finger 5 . FESEM image and particle size the distribution diagram layer of nickel oxide of thin with 5% In doped

3.4 Energy dispersive X-ray spectroscopy (EDX) analysis:

Due to the lack of observation of a peak due to the presence of indium in the sample, EDX analysis was used to prove the presence of indium within the samples. This analysis was performed by the FESEM device. The results of this analysis indicate the presence of Indium is in the samples.

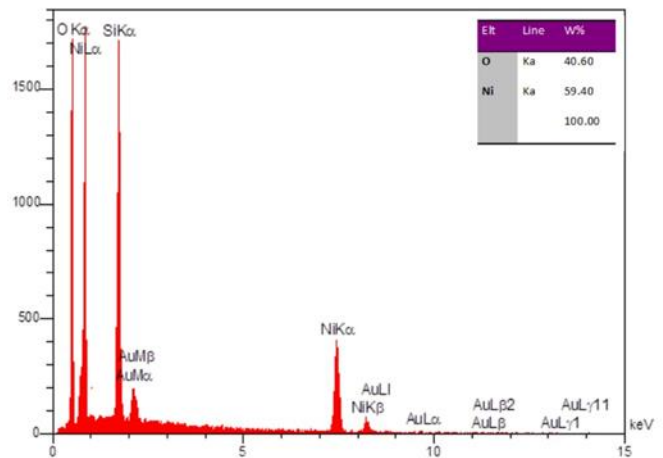


Figure 6 . Results of EDX analysis of the prepared of pure nickel oxide thin film

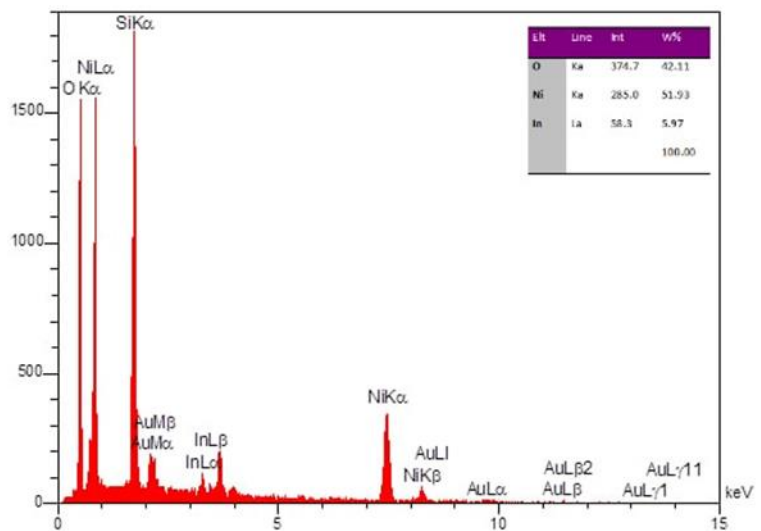


Figure 7. Results of EDX analysis of thin film of nickel oxide doped with 3% In

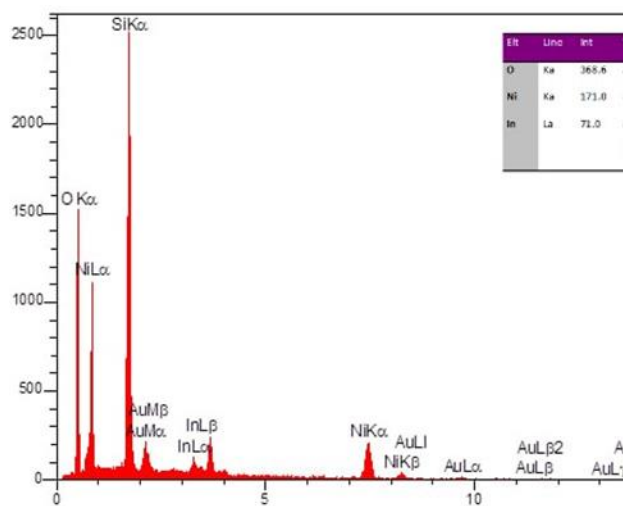


Figure 8 . Results of EDX analysis of thin film of nickel oxide doped with 5% In

4. conclusion

In this study, undoped and indium-doped nickel oxide thin films were successfully fabricated on glass substrates by spray pyrolysis. The structural properties of the samples were investigated by XRD analysis, which indicated that the layers grew along the preferred direction of the (111) plane. In the diffraction spectrum obtained from the doped samples, no peak refers to the presence of indium was observed, but EDX analysis showed the presence of indium. The surface roughness of the samples was determined by AFM analysis, and the morphology of the samples was examined using FESEM analysis. FESEM images reveals that the deposited films are smooth and uniformly covered the whole surface of the substrate.

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