

# Physical Characterization of Effect Ba Doping on Nanostructured NiO Thin Films by CSP Technique

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## Abstract.

Using a chemical spray pyrolysis (CSP) technique, thin films of undoped NiO and NiO:Ba were successfully grown. All the recorded films had a polycrystalline diffraction and that the predominant peak was (111) plane. the crystallite sizes were increased from 14.46 nm to 16.74 nm as in XRD, whilst dislocation density ( $\delta$ ) decreased from 4.78 to 3.56, whilst strain ( $\epsilon$ ) decreased from 2.38 to 2.07, AFM images assure the existance of nanostructure. Average diameter size, surface roughness and rms values of the deposited films were (79.36, 70.25 and 68.78) nm, (8.81, 7.07 and 4.23) and (5.85, 65.12 and 2.16) nm for NiO, NiO:2% Ba and NiO:4% Ba respectively. The transmittance of undoped NiO and Bs doped films reaches 70. 0% to 77.5% in Vis- NIR regions. The  $E_g^{op}$  decreased from 3.46 to 3.36 eV as the 4% Ba doping. Results illustrate both extinction coefficient and refractive index are increasing with increasing with Ba content in NiO thin films.

**Keywords:** NiO, Ba doping, chemical spray pyrolysis technique, XRD, morphology, optical properties.

## Introduction

Semi-transparent p-type conducting NiO films were recently enticed a lot of awareness due to their importance in a variety of scientific applications, such as (i) Materials for electrical display devices [1,2], (ii) layers to fabricate sensor [3], (iii) transparent films for electronic devices [4], and (iv) the magnetic property of nanoparticles [5–8]. A stoichiometric NiO film is a semiconductor material have a resistivity of  $\sim 10^{13} \Omega\text{-cm}$  [9]. NiO with an energy gap of 3.6 to 4.0 eV Recently . [10]. Metal oxide, in general, is very important because of wide range of band gap, conductive, transparent, and others [11-14].,To date, a variety of techniques for producing NiO thin films have been developed, such as dipping and electrochemical, sol–gel.[15], spin coating [16], chemical bath technique [17-19], PLD [20,51-71], ALD, sputtering [21-22], and CSP [23–32]. CSP is the most popular for wide area homogeneous NiO coatings because it eliminates the vacuum system and is simple to set up. The CSP technique is characterized by the uniform distributions and produces thin films with grain sizes that are controlled by a variety of parameters such as; the method or technique, doping material, and concentration. The effect of fabrication conditions such as the substrate temperature, has also been studied [33], and effect of thickness [34]. This study reflect the preparation of NiO thin film using CSP at 450 °C. XRD, AFM, and UV-visible Spectroscopy were employed to determine the physical characterization of the grown films.

## Experimental

NiO and NiO:Ba thin films were grown by CSP technique. 0.1 M of NiO Cl<sub>4</sub>. 5H<sub>2</sub>O was dissolved in deionized water. The doping agent was Ba provided by Sigma-Aldrich – German. drops of HCl were joined the mixture to get it clear. The Conditions for preparation as: The temperature of base was 450°C. The nozzle was 30 cm from the substrate. To prevent cooling, the spraying time was extended by 80 seconds, and the spray rate was increased to 5 mL/min. As a carrier gas, nitrogen gas was used. A weighing method is used to obtain film thickness, which was 330 nm. The AFM was done to know the surface of deposited films, while the XRD is utilized to know film structure. Optical transmittance was measured employing double beam Shimadzu UV-VIS spectrophotometer.

## Result and discussion

Fig. 1 shows XRD styles of Undoped NiO and doping Ba 2% and 4% thin films on glass substrate prepared via CSP technique. It can be observe that the films have peaks at 37.31°, 43.30° and 62.85° match anatase (111),(200) and (220) planes, respectively. High peak at (220) was seen that fit with ICDD card no 04-0835.

The crystallite size  $D$  in Table 1 estimated using Scherrer's equation [35-37]:

$$D = \frac{k \lambda}{\beta \cos \theta} \quad (1)$$

Where  $\lambda$  is the x-ray wavelength used,  $k = 0.9$ , and  $\theta$  is Bragg's angle, and  $B$  is FWHM

The acquired data are offered in Table 1. It shown that  $D$  were increased from 14.46 nm to 16.74 nm.

the dislocation density ( $\delta$ ) in the films is determined by [38-40]:

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

Table 1. It shown that dislocation density ( $\delta$ ) decreased from 4.78 to 4.15

the strain ( $\epsilon$ ) in the films was determined by [41-43]:

$$\epsilon = \frac{\beta \cos \theta}{4} \quad (3)$$

Table 1. It shown that strain ( $\epsilon$ ) decreased from 2.38 to 2.07.

The structural parameters  $S_p$  were shown in Figure (2). Table 1 displays FWHM of preferred reflections, which provide high precision values.

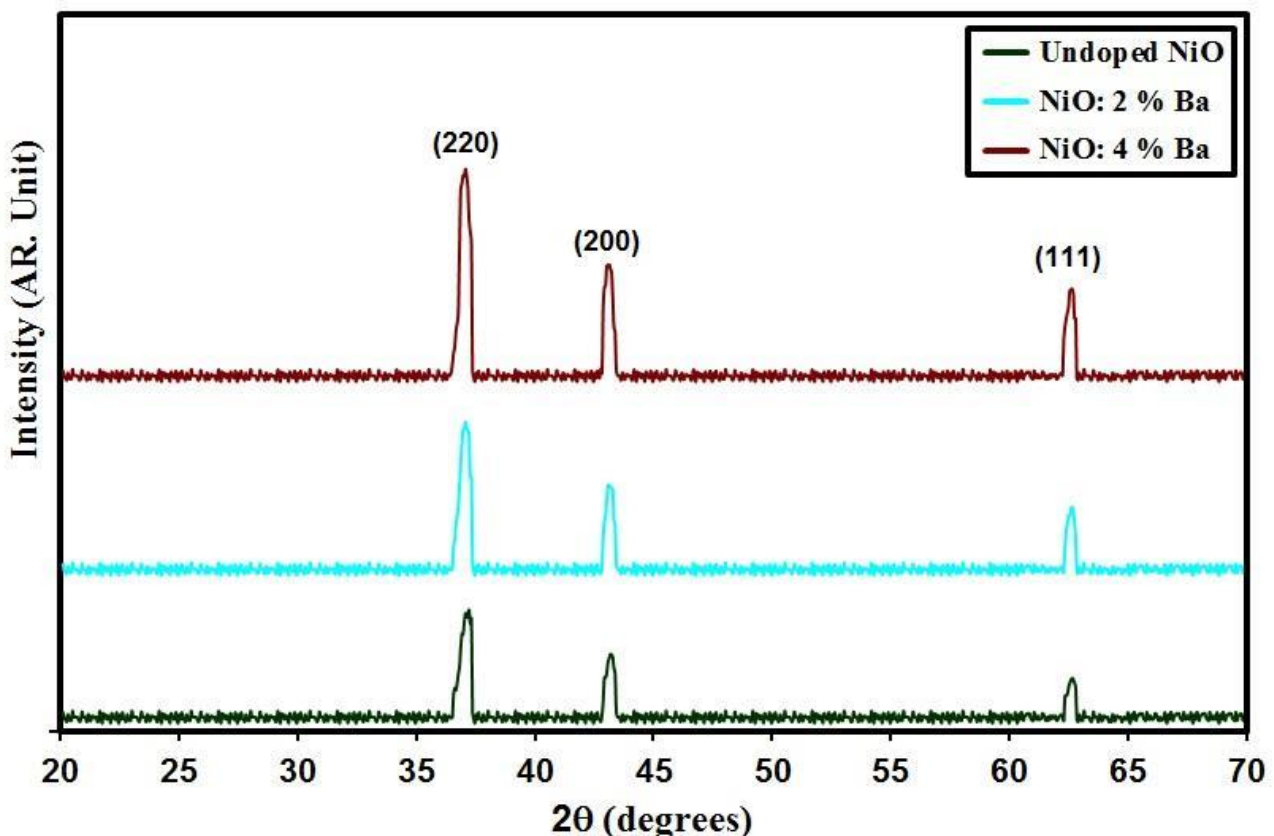
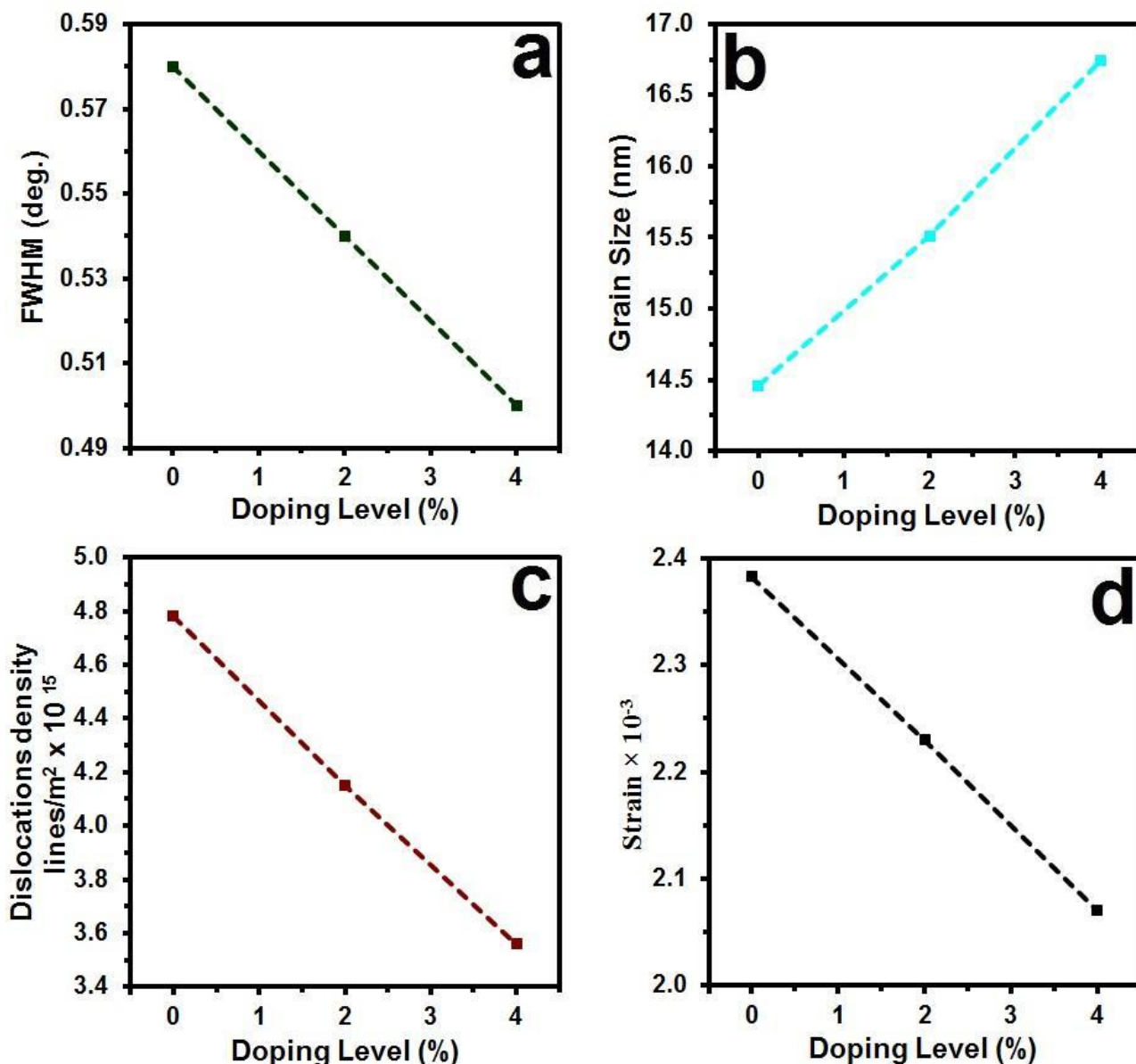


Fig.1. XRD styles of grown films.

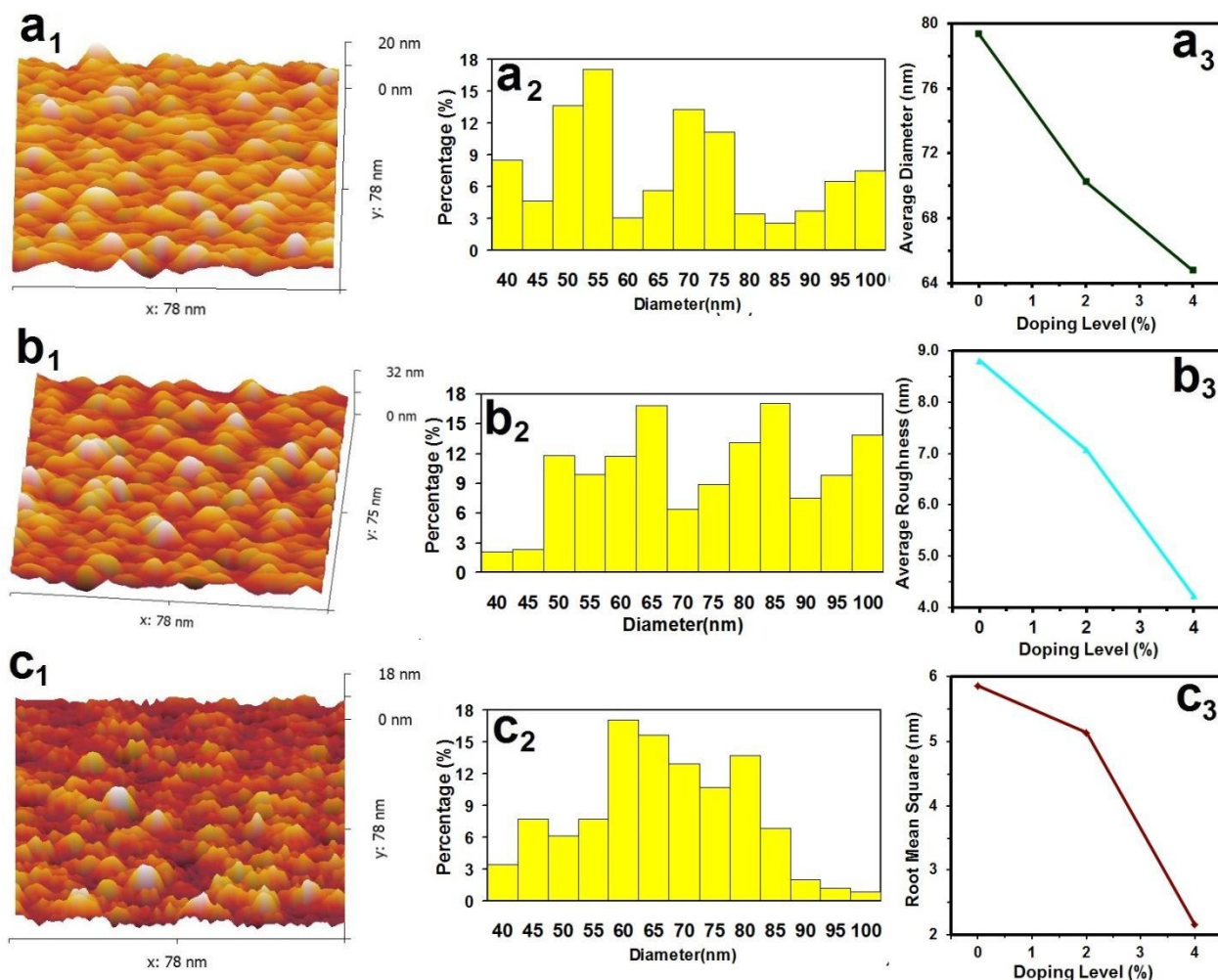
**Table 1.**  $S_p$  of deposited films.

Samples	$2\theta$ (°)	(hkl) Plane	FWHM (°)	$D$ (nm)	$E_g$ (eV)	$\delta$ ( $\times 10^{15}$ )(lines/m <sup>2</sup> )	$\epsilon \times 10^{-3}$
Undoped NiO	37.31	111	0.58	14.46	3.46	4.78	2.38
NiO: 2% Ba	37.00	111	0.54	15.15	3.40	4.15	2.23
NiO: 4% Ba	36.50	111	0.50	16.74	3.36	3.56	2.07



**Fig.2.** FWHM (a)  $D$  (b)  $\delta$  (c)  $\varepsilon$  (d) of intended films.

Fig. 3 offers the AFM micrograph for Undoped NiO and NiO: Ba thin films. Average Particle size  $P_{av}$  and rms values of the deposited films were (79.36, 70.25 and 68.78) nm and 5.85, 5.12 and 2.16) nm for Undoped NiO, NiO: 2% Ba and NiO: 4% Ba respectively. The surface roughness increases from 8.81 nm to 7.07 nm by increment of Ba content. The AFM parameters  $A_p$  were listed in Table (2).



**Fig.3.** AFM images (a<sub>1</sub>, b<sub>1</sub>, and c<sub>1</sub>), granularly distributed (a<sub>2</sub>, b<sub>2</sub>, and c<sub>2</sub>), and  $A_p$  variance through doping (a<sub>3</sub>, b<sub>3</sub> and c<sub>3</sub>).

**Table 2.**  $A_p$  of the intended films.

Samples	$P_{av}$ nm	Ra (nm)	rms (nm)
Undoped NiO	79.36	8.81	5.85
NiO: 2% Ba	70.25	7.07	5.12
NiO: 4% Ba	68.78	4.23	2.16

The transmittance (T) spectra are shown in Fig.4 T decreases from 77.5 % to 70% as Ba doping increases from 1% to 3 %.. The absorption coefficient ( $\alpha$ ) is obtained from [44-46]:

$$\alpha = (2.303 \times A)/t \quad (4)$$

Where (t) is film thickness.

$\alpha$  decreases with increasing Ba doping levels of 2% or 4%, as noticed in Fig.6.

The optical energy gap  $E_g$  was calculated from the Tauc relation [47,48]:

$$(\alpha h\nu) = A(h\nu - E_g)^{\frac{1}{2}} \quad (6)$$

Where A is the constant,  $(\alpha h\nu)^2$  against  $(h\nu)$ , plots are offer in Figure 4. As NiO is a direct transition semiconductor.  $E_g$  decreased from 3.46 to 3.36 eV as the 4% Ba doping.

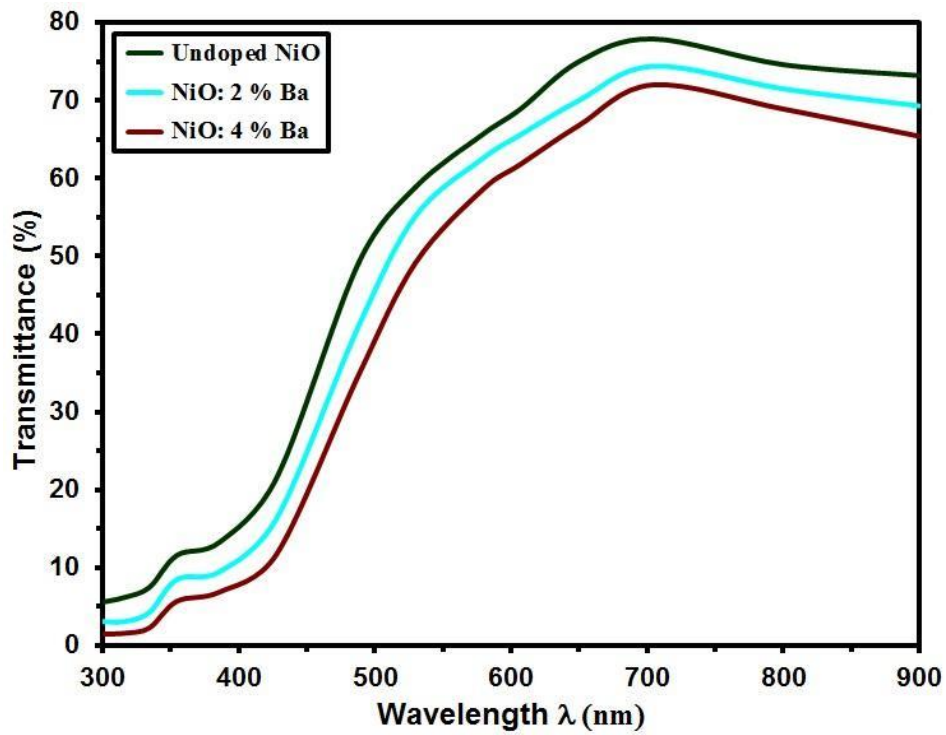


Fig. 4. T of grown films.

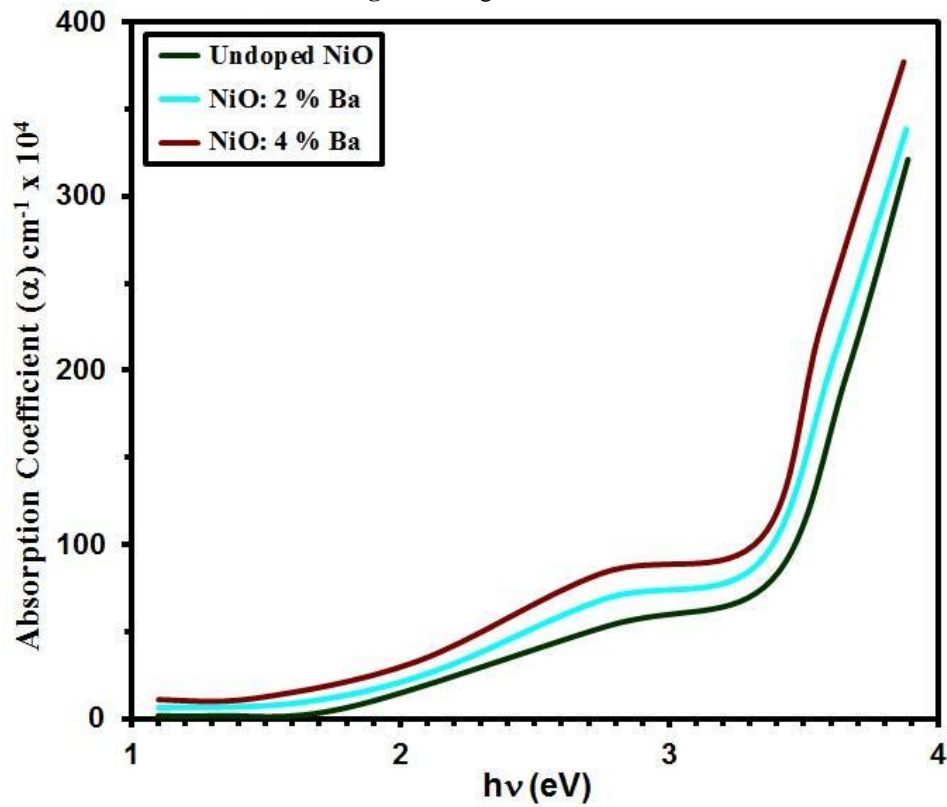


Fig. 5.  $\alpha$  Vs  $h\nu$  for intended films.

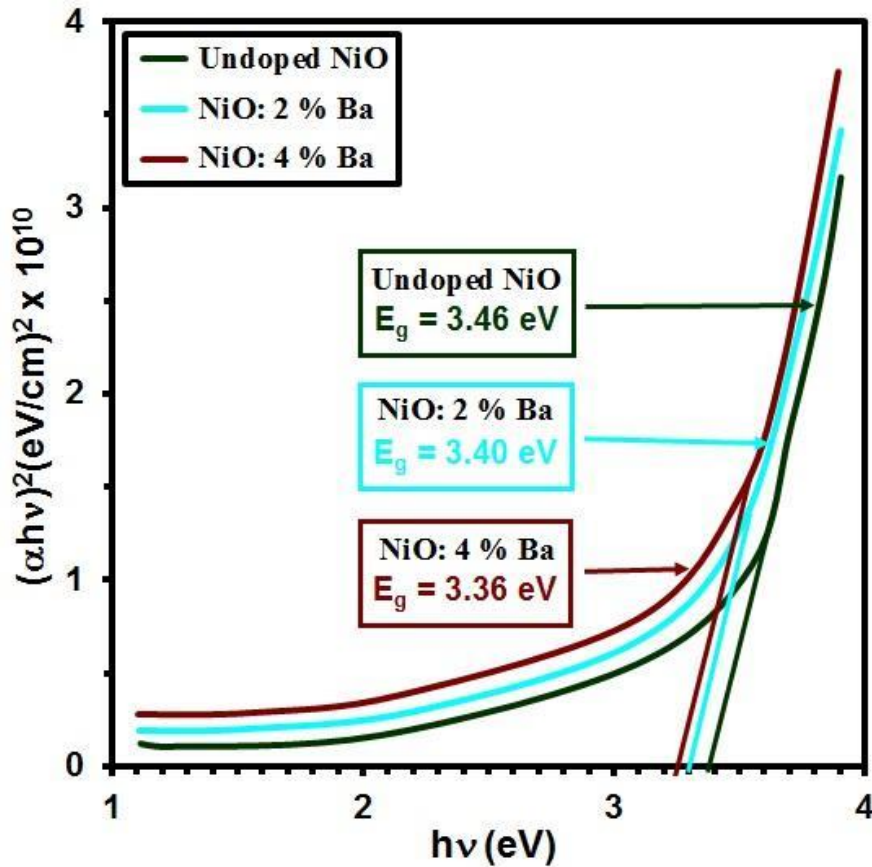


Fig. 6.  $(\alpha h\nu)^2$  Vs  $h\nu$  of deposit films.

To find the extinction coefficient ( $k$ ) following relation was employed [49]:

$$k = \frac{\alpha \lambda}{4\pi} \quad \text{----- (7)}$$

Where  $\lambda$  is the wavelength. Figure 7 offer the variance of  $k$  of NiO and Ba: NiO films versus the wavelength. Figure 7 offers that  $k$  increases with increasing the content of Ba. To calculate the refractive index ( $n$ ) of NiO and NiO: Ba thin films. We can use the following relationship [50]:

$$n = \left( \frac{1+R}{1-R} \right) + \sqrt{\frac{4R}{(1-R)^2} - k^2} \quad \text{----- (8)}$$

Where  $R$  is the reflectivity.

Figure (8) represents  $n$  plot vs. wavelength. Refractive index diagram, divided into two regions: abnormal dispersion and normal dispersion.  $n$  increases with increasing the Ba content, as shown in Fig (8).

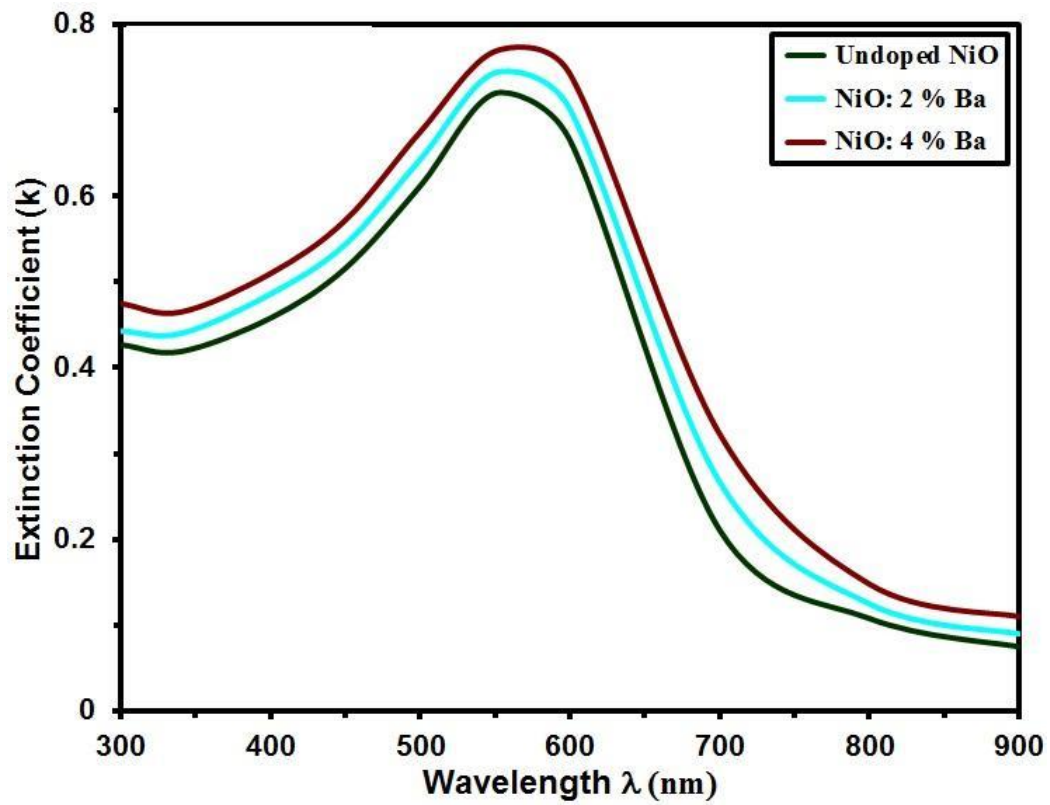


Fig. 7:  $k$  of intended films.

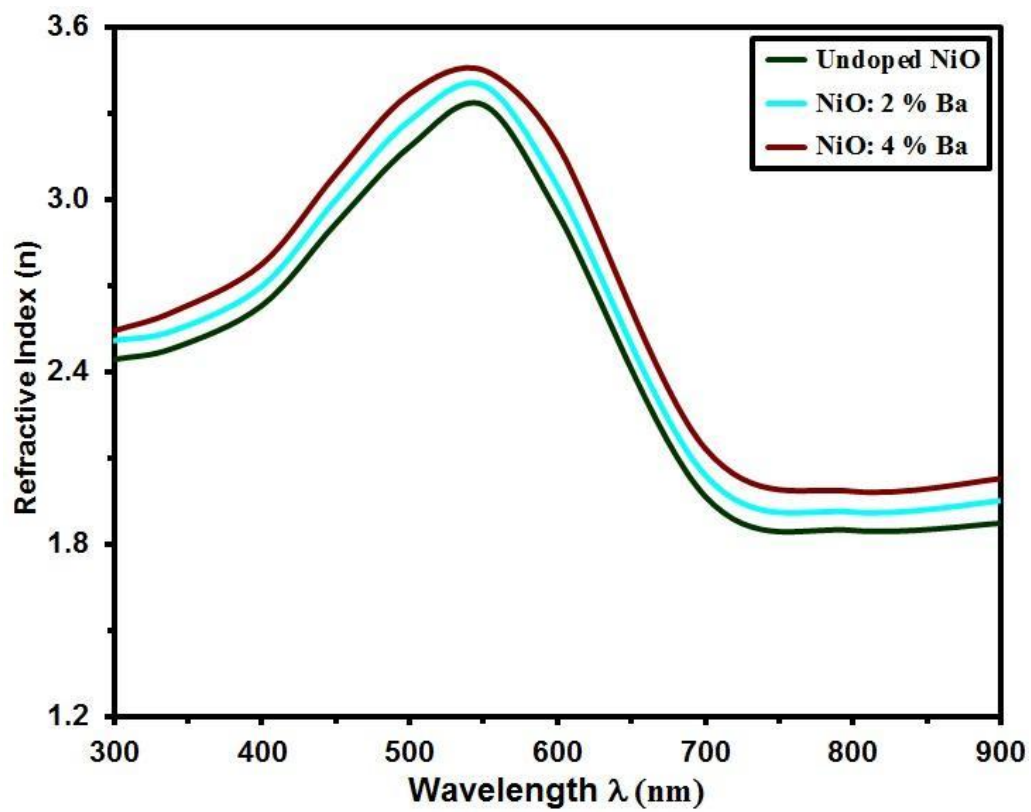


Fig. 8.  $n$  of deposit films

Conclusion

Chemical spray pyrolysis has been shown to be a useful technique for preparing high percentages of NiO and NiO: Ba deposition of 2% and 4%. The XRD shows a predominant peak (220) for NiO films, the crystallite sizes were increased from 14.46 nm to 16.74 nm, whilst dislocation density ( $\delta$ ) decreased from 4.78 to 3.56, whilst strain ( $\epsilon$ ) decreased from 2.38 to 2.07. AFM image showed that, Average Particle size increases from (79.36 to 68.78) nm, The surface roughness increases from 8.81 nm to 4.23 nm by increasing transmittance of Undoped NiO and NiO: Mn films decreases from 77.5 % to 70% as Ba content increases from 1% to 3 %.  $E_g$  decreased from 3.46 eV for pure to 3.36 eV for 4wt.% Ba doped NiO film.  $\alpha$ ,  $n$  and  $k$  are increasing with Ba content in NiO thin films.

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