# Effect of Mn Doping on Physical Characterization of $ZrO_2$ Thin Films by Green spraying technique

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**Abstract**

**Nanocrystalline $ZrO_2$ and $ZrO_2$: Mn with 1% and 3% concentrations are deposited via green chemical spray pyrolysis CSP method. XRD styles assure that films were polycrystalline, mixture of monoclinic and tetragonal phases, and the preferred crystal plane orientation was (111) corresponding to $2\theta = 31.47^\circ$, the crystallite sizes were increased from 13.53 nm to 15.56 nm as in XRD, whilst dislocation density (δ) decreased from 54.62 to 41.19, whilst strain (ε) decreased from 25.61 to 22.28, AFM images confirm the appearance of nanostructure. Average Particle size and rms values of the deposited films were (66.19, 64.57 and 52.84) nm and 7.69, 6.34 and 2.37) nm for $ZrO_2$ , $ZrO_2$:1% Mn and $ZrO_2$:3% Mn respectively.Transmittance reaches 80% to 90% in Vis- NIR regions. The optical band gap was in the zone of 5.35-5.25 eV. Results illustrate Refractive Index and extinction coefficient show an increment via Mn content.**

**Keywords: $ZrO_2$, Mn doping, CSP technique, XRD, AFM.**

## Introduction

$ZrO_2$ is a charming material due to its potential applications in optical filters, high power lasers, insulators, storage capacitors [1-5]. It can be utilized as a replace $SiO_2$ gate dielectric in MOS devices [6,7]. Its own direct and indirect transition at 5.87eV and 5.22 eV, respectively [9]..Different methods are employed to deposit $ZrO_2$ like, ,PLD , RF sputtering deposition was employed, ECD, spray pyrolysis, CVD, laser ablation, thermal evaporation method, Plasma spraying, ECD, , hydrothermal processing, magnetron sputtering, [44-64]liquid phase deposition, dip coating and sol-gel method [8–20]. spray pyrolysis was stellar low-cost for depositing identical, and good quality [21-23]. In this study, the investigation of Mn content on physical properties of $ZrO_2$ films was studied.

## Experimental

$ZrO_2$ films were deposited employing chemical spray pyrolysis method. A solution 0.1M $ZrOCl_2.8H_2O$ and oxalic acid was resolved in 100 mL re-distilled water. 0.1 M of Mn trichloride Mn $Cl_3$ was used as a doped material, was added to matrix solution to gain doping ratio of 1% and 3%. The base temperature was 425°C. After experiments, the preparation conditions arrived at the followings: space between base and spout was 30 cm. spraying time 8s , spraying rate 5 mL/min and time period between two spray processess was 1.30 min. $N_2$ was employed as carrier gas. Film thickness was evaluated by weighing method and was 325 ± 20 nm. XRD is used to know film structure, AFM is utilized to study film surface.Transmittance is caculated via double beam spectrophotometer UV–Visible.

## Result and discussion

XRD styles are shown in Fig. 1. We can observe that XRD patterns of grown ZrO2 and ZrO2:Cu thin film is 24,49°, 31,47°, 40,82°, and 55,40° correspond to anatase (110),(111), (112) and (310) planes, respectively. High peak at (111) was seen that, fit with ICDD card no 1314-23-4.

Debye Scherer's formula eq. 1 was used to evaluate crystallite size (*D*) [24-26]:

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

Where λ is the x-ray wavelength used, k = 0.9 and θ is Bragg's angle, β is FWHM. The acquired data are given in Table 1. It shown that *D* were increased from 13.53 nm to 15.56 nm as in XRD. The dislocation density ($\delta$) in the films was determined by [27-29]:

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

Table 1. displys dislocation density ($\delta$ ) that was decreased from 54.62 to 41.19 and strain (ε) in the films was determined by [30-32]:

$$\varepsilon = \frac{\beta cos\theta}{4} \qquad (3)$$

Table 1. It is shown that strain (ε) decreased from 25.61 to 22.28. Structural parameters ($S_P$)were seen in Figure (2).

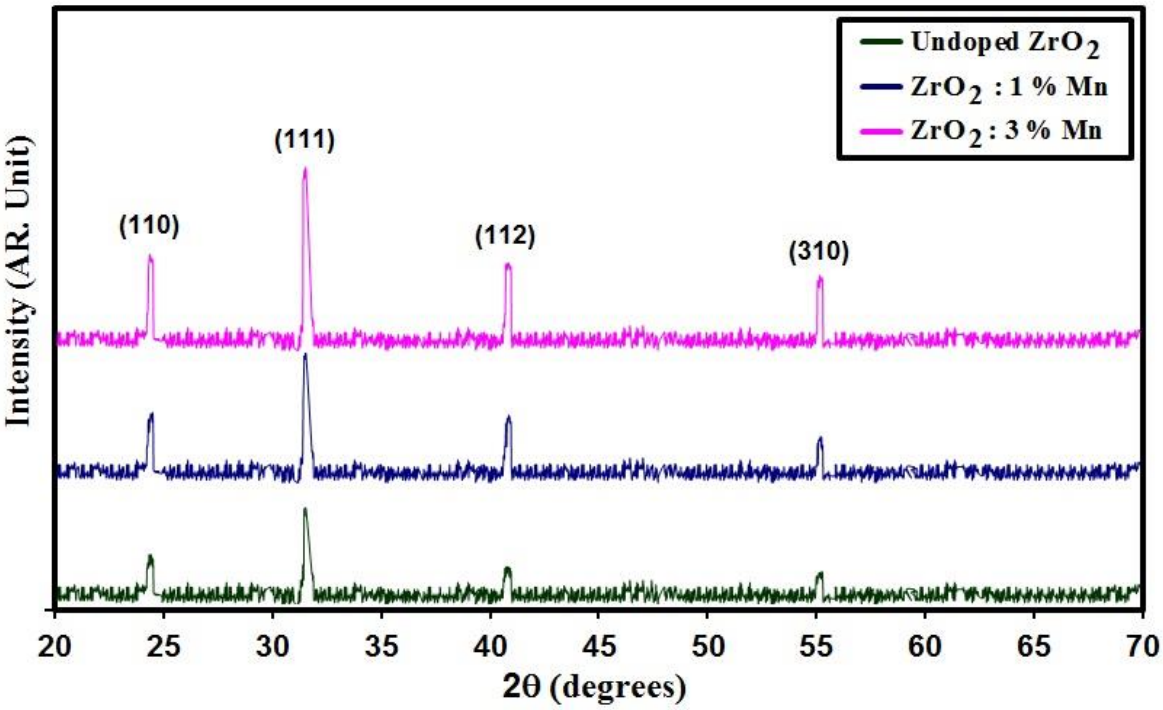


Fig.1. XRD styles of deposit films.

Table 1. *D*, $E_g$ and $S_P$ of deposited films.

| Samples | (hkl) Plane | 2 □ (°) | FWHM (°) | *D* (nm) | Eg (eV) | δ (× $10^{14}$)(lines/$m^2$) | ε × $10^{-4}$ |
|---|---|---|---|---|---|---|---|
| Undoped $ZrO_2$ | 111 | 31.47 | 0.61 | 13.53 | 5.35 | 54.62 | 25.61 |
| $ZrO_2$: 1% Mn | 111 | 31.42 | 0.57 | 14.48 | 5.30 | 47.69 | 23.93 |
| $ZrO_2$: 3% Mn | 111 | 31.00 | 0.53 | 15.56 | 5.25 | 41.19 | 22.28 |

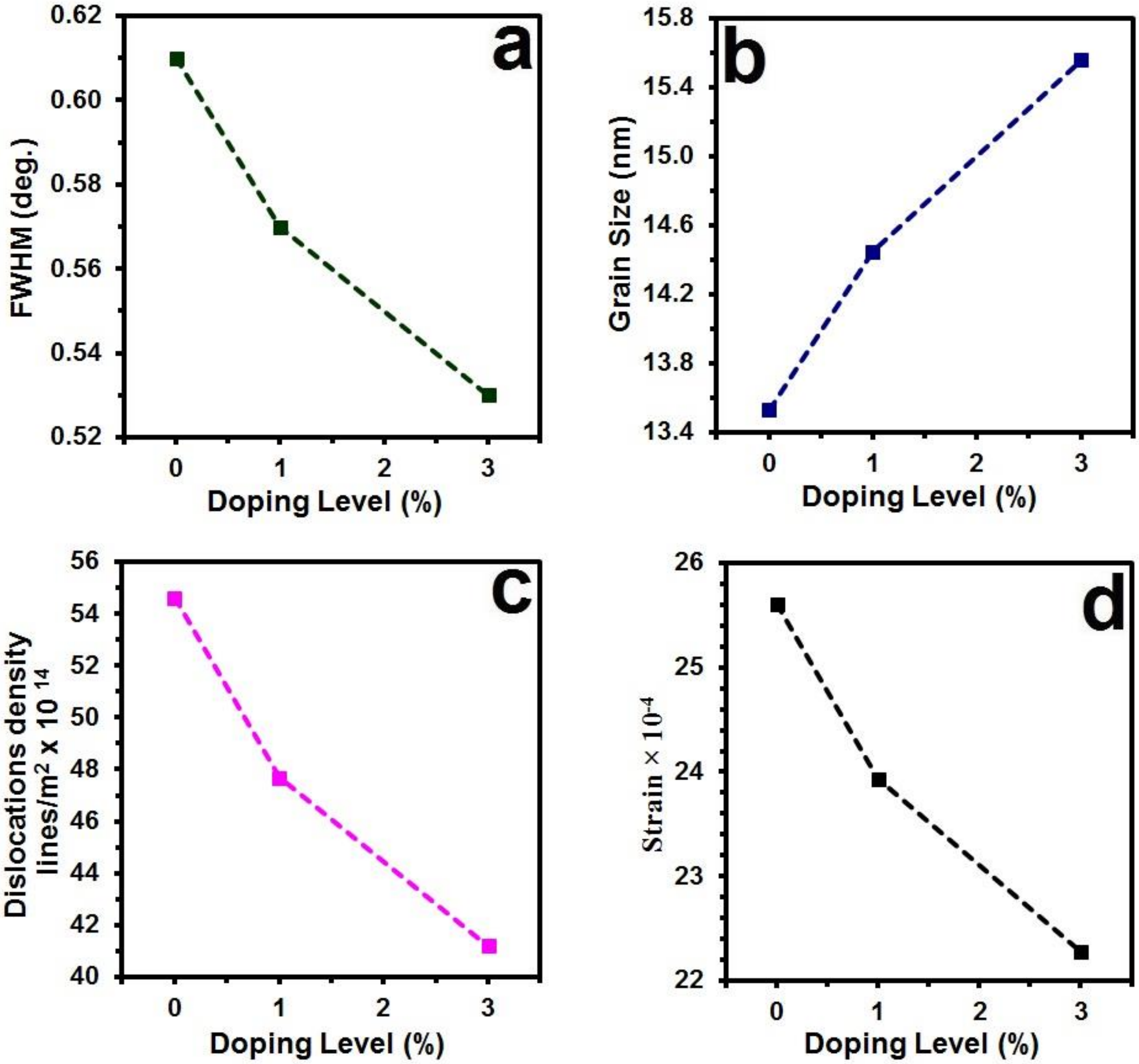


Fig.2. FWHM (a) *D* (b) δ (c) ε (d) of intended films.

Fig. 3 offers AFM micrograph for Undoped $ZrO_2$ and $ZrO_2$:Mn thin films. sharp peaks appeared in the domain and display densely packed columnar crystalline. The surface roughness $R_a$ increases from 6.78 nm to 3.26 nm by increasing $ZrO_2$:3% Mn. From Figure 3 ($a_3$, $b_3$ and $c_3$). Average Particle size $P_{av}$ and rms values of the deposited films were (66.19, 64.57 and 52.84) nm and 7.69, 6.34 and 2.37) nm for $ZrO_2$ , $ZrO_2$:1% Mn and $ZrO_2$:3% Mn respectively.

Table (2) displays AFM parameters $P_{FM}$.

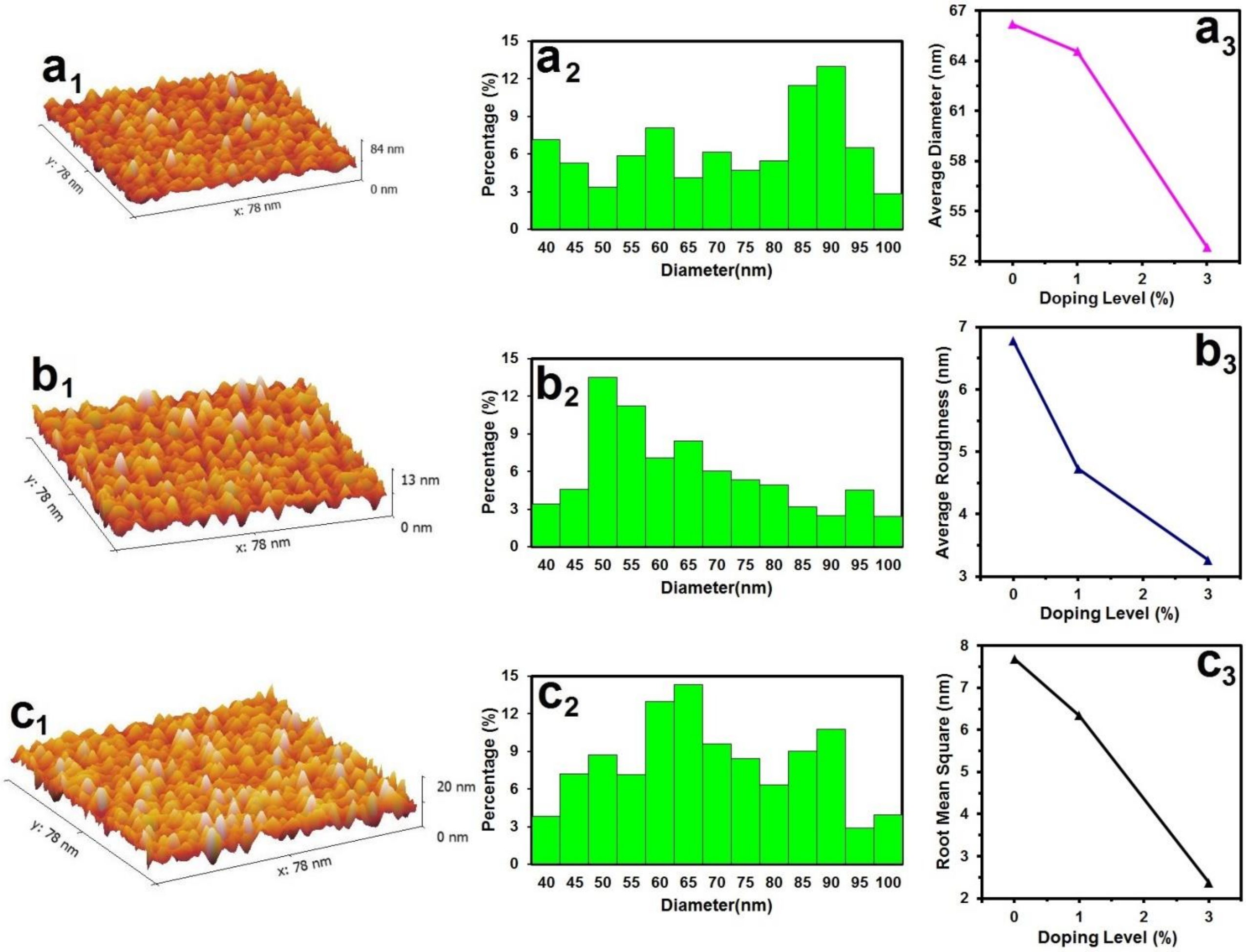


Fig.3. AFM information of the deposit films

Table 2. $P_{FM}$ of the intended films.

| Samples | $P_{av}$ nm | Ra (nm) | rms (nm) |
|---|---|---|---|
| $ZrO_2$ | 66.19 | 6.78 | 7.69 |
| $ZrO_2$: 1% Mn | 64.57 | 4.73 | 6.34 |
| $ZrO_2$: 3% Mn | 52.84 | 3.26 | 2.37 |

The transmittance T spectra of Undoped $ZrO_2$ and $ZrO_2$:Mn films are seen in Fig.4 The deposited films with an average T value of 80%, but the transmittance decreases when the Mn % doping increases.

The absorption coefficient α of Undoped $ZrO_2$ and $ZrO_2$:Mn was calculated employing the relation [33-35]:

$$\alpha = \frac{2.303A}{t} \quad (4)$$

Where t is the film thickness. Fig. (5) offers the dependence of α via wavelength. α increases with the increase of Mn. The position of the absorption edge slightly changes, this could be due to the presence of Mn atoms that limits the growth of $ZrO_2$ grains (decrease of grain size with metal concentration).

The optical energy gap $E_g$ is determined by Tauc [36-38] relation:

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

Where A is the constant,, (□h□)$^2$ versus incident photon energy (h□), plots were obtained. The graphs are represented in Figure 6, the estimated band gap energy values are found to be 5.35 eV for the $ZrO_2$ and 5.25 eV for $ZrO_2$:Mn thin films.

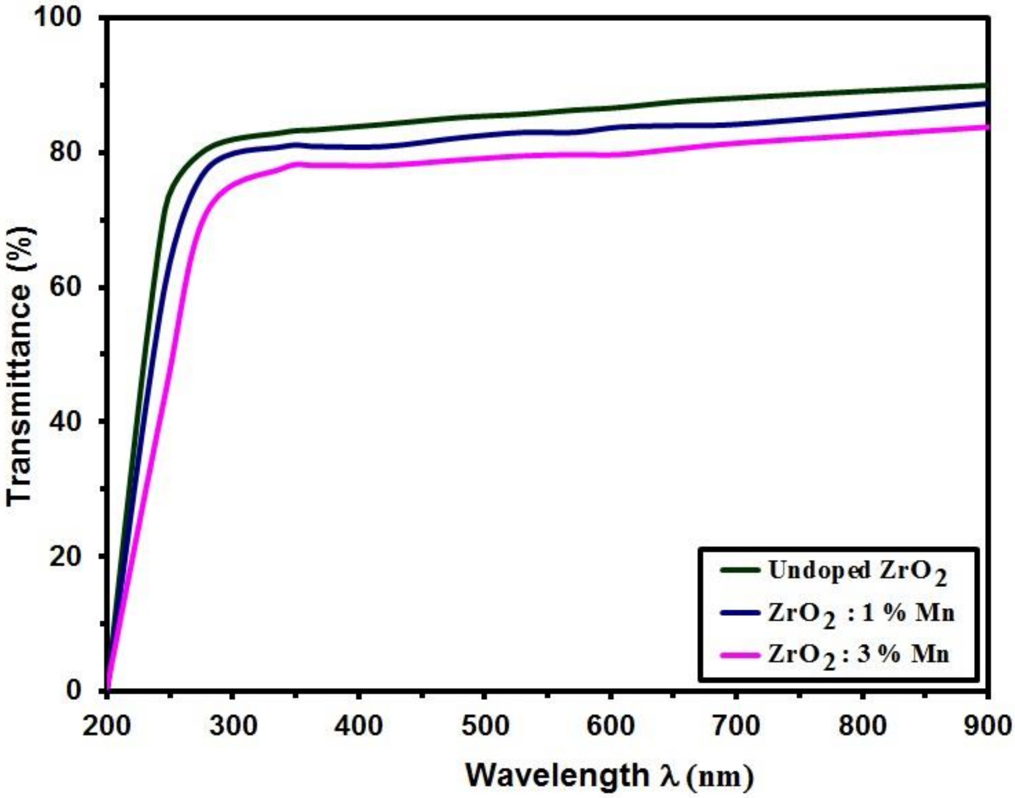


Fig. 4. T for grown films.

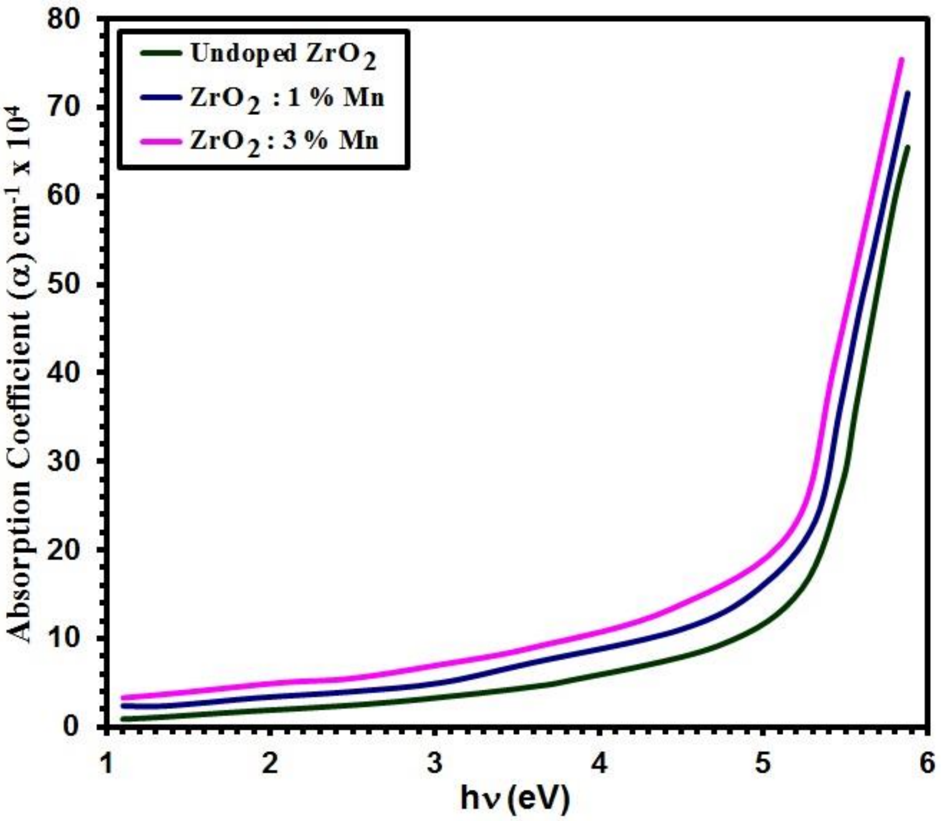


Fig. 5. α Vs hν for intended films.

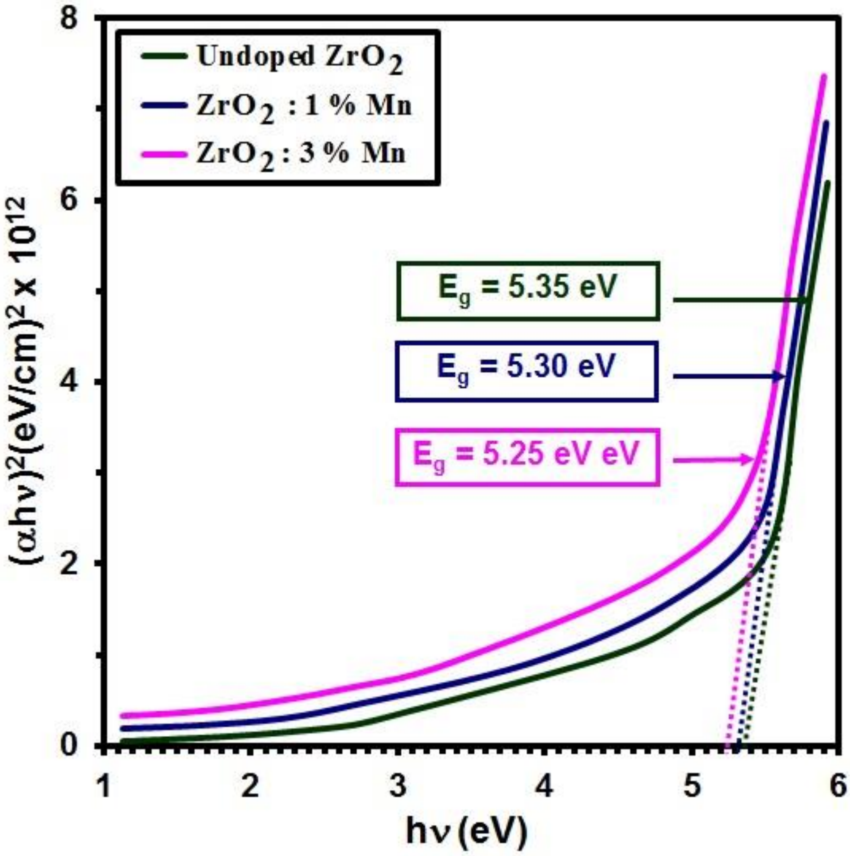


Fig. 6. $(\alpha h\nu)^2$ Vs hν of deposit films.

The extinction coefficient (K) is evaluated by the formula[39-40]:

$$k = \frac{\alpha\lambda}{4\pi} \qquad \text{-----3}$$

Where λ is the wavelength. Figure (7) displays the varianence of K against wavelength. `The fall in K may be due to the absorption of light at the grain boundary [41]. K value show an increment with the increment of Mn at λ range of 200-600 nm. K of a material is related to its absorption characteristic.

The refractive index (n) was expressed as [42-43]:

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

Where R is the reflectance

Figure (8) shows n plot vs. wavelength of $ZrO_2$ and $ZrO_2$:Mn thin films. The *n* values of the Undoped $ZrO_2$ and $ZrO_2$:Mn thin films increase with increasing in abnormal dispersion region, while n values of $ZrO_2$ and $ZrO_2$:Mn thin films decrease with increasing wavelength in normal dispersion region. n values of $ZrO_2$ thin film change with doping with Mn. The refractive index (3.1) of the $ZrO_2$: 3% Mn thin film at λ = 390 nm is higher than that value (3.0) of the 1% Mn-doped $ZrO_2$ films at λ=390 nm a similar observed in the literature.

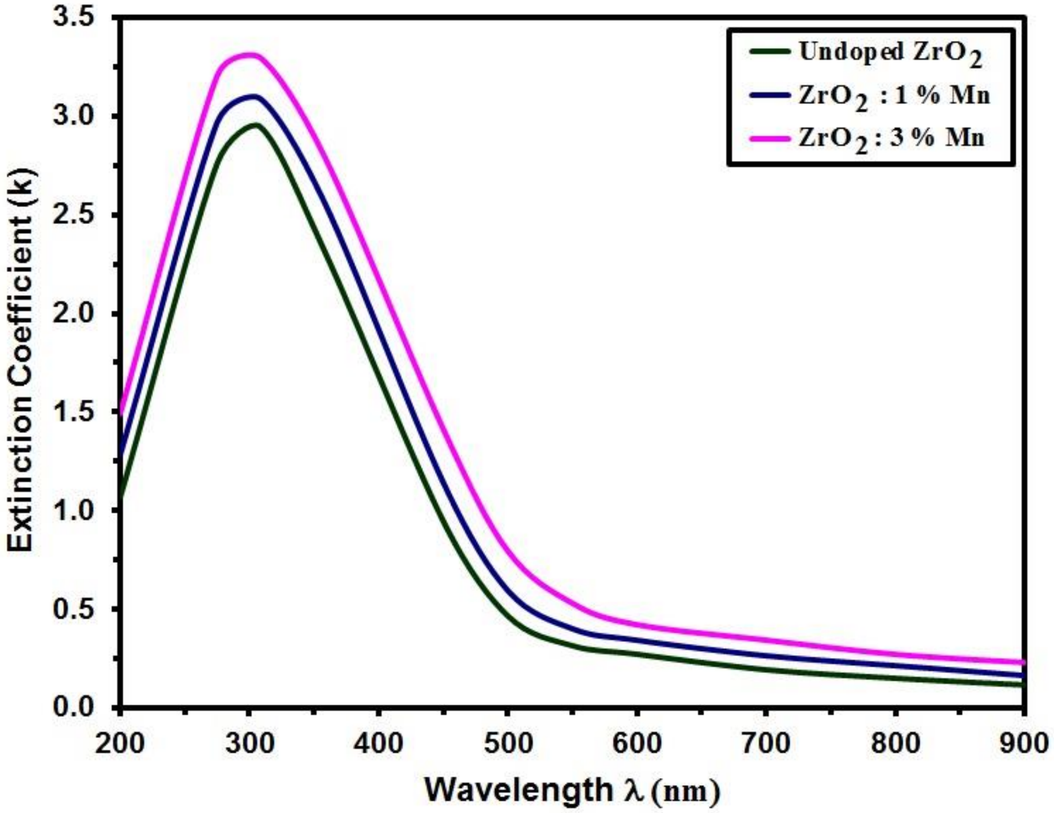


Fig. 7: k of the deposit films.

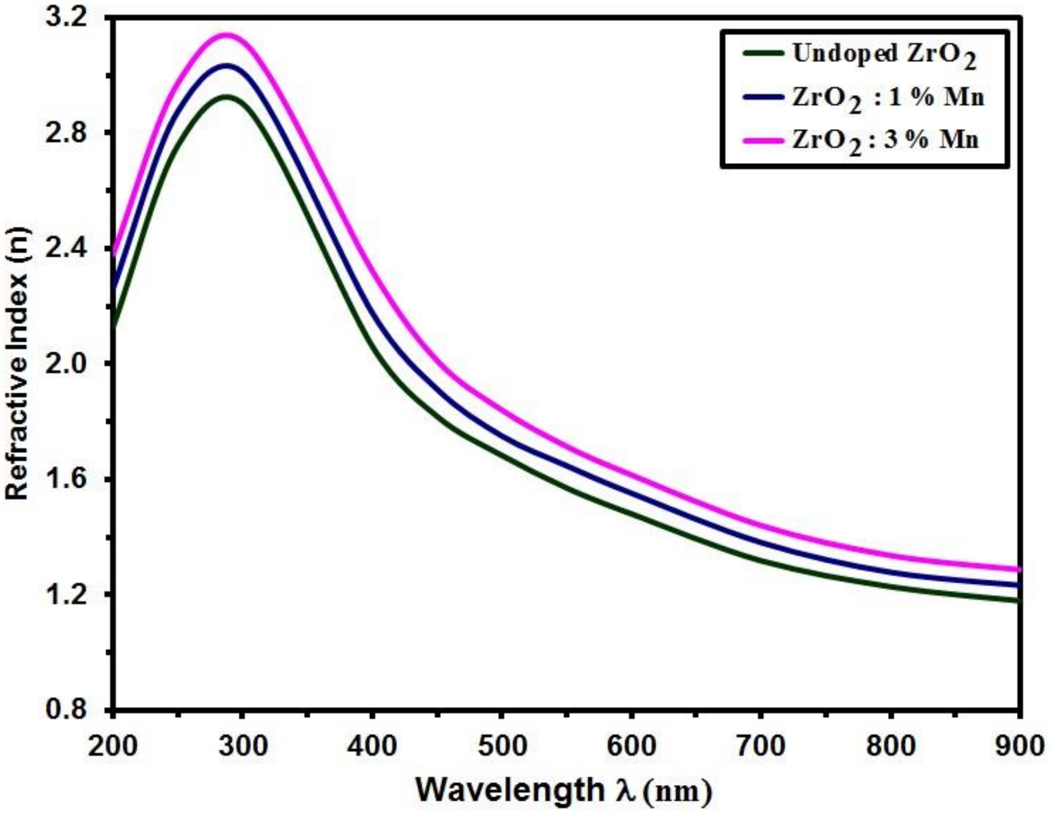


Fig. 8. n of the deposit films

**Conclusion**

Chemical spray pyrolysis was utilized to deposit Undoped $ZrO_2$ and ZrO2: Mn with 1% and 3% concentrations films. XRD has a dominant peak at (002) for $ZrO_2$ films at 3% Mn, the crystallite sizes were increased from 13.53 nm to 15.56 nm, whilst dislocation density (δ) decreased from 54.62 to 41.19, whilst strain (ε) decreased from 25.61 to 22.28. AFM results indicate the dependence of surface topography and roughness upon doping. AFM image showed that $R_a$ increases from 6.78 nm to 3.26 nm by increasing $ZrO_2$ to $ZrO_2$:3% Mn , transmittance of Undoped $ZrO_2$ and $ZrO_2$: Mn films decrease from 90% to 80% as Mn content increases from 1 to 3 at%. α increased with the increase of Mn content, Bandgap of Undoped $ZrO_2$ thin film is 5,35 eV, was decreased to 5,25 eV via Mn doping. the n and K are increasing with Mn content in $ZrO_2$.


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