Research Article

Quantum Effects of Indium/Ytterbium Doping on ZnO-Like Nano-Condensed Matter in terms of Urbach-Martienssen and Wemple-DiDomenico Single-Oscillator Models Parameters

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Conducting and transparent optical ZnO thin films were deposited on glass substrates by a simple mini spray technique. Alternatively, some of the obtained films were doped with indium and ytterbium at the molar rates of: 1, 2, and 3% (In) and 100, 200, and 300 ppm (Yb). In addition to the classical structural investigations including XRD, microhardness vickers (Hv), and optothermal techniques, thorough optical measurements have been carried out for comparison purposes. The refractive indices and the extinction coefficients of the differently doped layers have been deduced from their transmission-reflection spectra over an extended wavelength range. Analysis of the refractive index data through Wemple-DiDomenico single oscillator model yielded quantum characteristics along with the values of long-wavelength dielectric constant, average oscillator wavelength, average oscillator strength, average oscillator energy and dispersion energy. Real and imaginary parts of dielectric constant have also been used to calculate free carrier plasma resonance frequency, optical relaxation time, and free carriers concentration-to-effective mass ratio. Finally, analysis of Urbach-Martienssen model parameters allowed proposing nanoscale explanations to the divergence about doping-related evolution of Urbach tails, this intriguing item having been intensively discussed in the literature in the last decades.

1. Introduction

Transparent conducting oxides (TCOs) such as tin oxides and indium-doped oxide systems have been used in several optoelectronic devices such as gas sensors, panel displays [1, 2], and photovoltaic solar cells (PVCs). Among these oxides, zinc oxide has attracted considerable attention from those interested in the application to devices working in ultraviolet regions, with the interest specially lying in its wide bandgap, quantum confinement effects in accessible size ranges, and large exciton binding energy (≈ 60 meV) [1, 2].

It has been recorded that zinc oxide is a hexagonal wurtzite structured semiconductor with high piezoelectric and gas-detecting properties [1–6]. Its deposition on glass-like substrates has been widely experimented and applied [7–11].

On the other hand, many doping elements for ZnO have been tried [12–15]. In some studies, the merits of indium and aluminium as effective doping agents have been pointed out [16–19].

In this work, explanations to the paradoxical effects of indium and ytterbium doping on ZnO crystalline structure

are proposed. The elaborating techniques and doping protocols have been detailed along with common characterizing techniques. The last part of this paper is dedicated to the nanoscale analyses and discussions in reference to Urbach-Martienssen and Wemple-DiDomenico single-oscillator models.

2. Experimental Details

2.1. Undoped ZnO Films Preparation. ZnO thin films have been first prepared at a glass substrate temperature of 460°C, using propanol and zinc acetate $\text{Zn}(\text{CH}_3\text{CO}_2)_2 \cdot 10^{-2} \text{ M}$ according to the chemical protocol summarised elsewhere [20, 21].

The precursor mixture was acidified using acetic acid ($\text{pH} = 5$) and the carrier gas was nitrogen (pressure $\approx 0.35 \text{ bar}$) through a 0.5 mm diameter nozzle. The nozzle-to-substrate plane distance was fixed at the optimal value of 27 cm as demonstrated earlier, for the same disposal, by K. Boubaker et al. [22]. During the whole deposition process, precursor mixture flow rate was approximately 4 mL/min.

Thicknesses of the obtained layers have been deduced according to the method established by Belgacem and Bennaceur [23].

2.2. Indium and Ytterbium Doping. Under similar experimental conditions, indium-doped ZnO, In thin films solutions have been fabricated by adding hydrated indium chloride ($\text{InCl}_3 \cdot x\text{H}_2\text{O}$, 99.9% purity) to the precursor solution while maintaining acidity level. In the three elaborated samples, the indium-to-zinc molar ratios $[\text{In}^{3+}]/[\text{Zn}^{2+}]$ were 0.01, 0.02, and 0.03 for the samples designated as ($A_{\text{Ind},1}$), ($A_{\text{Ind},2}$), and ($A_{\text{Ind},3}$), respectively.

In ytterbium-doped ZnO, Yb thin films solutions were fabricated similarly by adding small amounts of ytterbium chloride (YbCl_3) to the precursor solution in which ytterbium-to-zinc molar ratios $[\text{Yb}^{3+}]/[\text{Zn}^{2+}]$ were 100, 200, and 300 ppm for the respective samples ($B_{\text{Yb},1}$), ($B_{\text{Yb},2}$), and ($B_{\text{Yb},3}$).

Undoped ZnO samples are designated by $A_{\text{ZnO},0}$.

2.3. Common Characterization Disposals. The prepared films $A_{\text{ZnO},0}$, $A_{\text{Ind},i|_{i=1..3}}$, and $B_{\text{Yb},i|_{i=1..3}}$ were investigated using X-ray diffractometry by means of a Philips (PW1429) system. The optical transmittance $T(\lambda)$ and reflectance $R(\lambda)$ of the films were recorded using a Shimadzu UV 3100 double-beam spectrophotometer, within a (300–1800 nm) wavelength range. Finally, a mechanical characterization has been carried out using a diamond-pyramidal-indenter Vickers test disposal whose details are presented in precedent studies [20–23].

3. Already Achieved XRD, Mechanical, and Optothermal Characterization

XRD, mechanical, and optothermal characterization of the as-grown films has been already carried out and discussed in

FIGURE 1: (a) In-doped ZnO films $A_{\text{Ind},i|_{i=1..3}}$ XRD diagrams and (b) Yb-doped ZnO films $B_{\text{Yb},i|_{i=1..3}}$ XRD diagrams.

precedent studies [20, 21]. XRD patterns of the deposited In-doped ZnO films along with the Yb-doped ones are shown in Figures 1(a) and 1(b), respectively.

The mechanical microhardness of the ytterbium- and indium-doped ZnO layers has been evaluated in terms of Vickers hardness (H_v). The characterization test has been carried out using a common diamond-pyramidal-indenter Vickers test disposal whose main parameters are detailed in precedent studies [20, 21].

Optothermal investigation has been based on variation of the Amlouk-Boubaker Optothermal expansivity ψ_{AB} , which is a synthetic physical parameter defined and used in precedent studies [24–39]. This parameter has been defined by

$$\psi_{AB} = \frac{D}{\hat{\alpha}}, \quad (1)$$

where D is the thermal diffusivity and $\hat{\alpha}$ is the effective absorptivity, defined [30–33] as the mean normalized

FIGURE 2: Urbach tails scheme.

TABLE 1: Microhardness (Hv) and Amlouk-Boubaker Optothermal expansivity ψ_{AB} .

Material	Sample Name	Doping rate	Microhard. (Hv)	ψ_{AB} ($\times 10^{-12} \text{ m}^3 \text{ s}^{-1}$)
ZnO	$A_{\text{ZnO},0}$	—	341	18.2
In-doped ZnO	$A_{\text{Ind},1}$	1%	575	14.5
	$A_{\text{Ind},2}$	2%	649	12.9
	$A_{\text{Ind},3}$	3%	739	10.1
Yb-doped ZnO	$B_{\text{Yb},1}$	100 ppm	301	9.21
	$B_{\text{Yb},2}$	200 ppm	399	8.24
	$B_{\text{Yb},3}$	300 ppm	496	11.2

absorbance weighted by $I(\tilde{\lambda})_{\text{AM1.5}}$, the solar standard irradiance, with $\tilde{\lambda}$ being the normalised wavelength:

$$\hat{\alpha} = \frac{\int_0^1 I(\tilde{\lambda})_{\text{AM1.5}} \times \alpha(\tilde{\lambda}) d\tilde{\lambda}}{\int_0^1 I(\tilde{\lambda})_{\text{AM1.5}} d\tilde{\lambda}}, \quad (2)$$

where $I(\tilde{\lambda})_{\text{AM1.5}}$ is the reference solar spectral irradiance, fitted using the Boubaker polynomials expansion scheme (BPES).

The calculated values of the mechanical Vickers microhardness (Hv), along with Amlouk-Boubaker Optothermal expansivity ψ_{AB} , for the doped layers are gathered in Table 1.

4. Wemple-DiDomenico and Urbach-Martienssen Models Investigations

4.1. Theoretical Fundamentals. The Wemple-DiDomenico single-oscillator model is based on a particular dependence [40] of the dispersion of refractive index below the interband absorption edge:

$$n = \sqrt{1 + \frac{E_d E_0}{E_0^2 - (h\nu)^2}}, \quad (3)$$

where E_0 is the single-oscillator energy, ν is the frequency, h is Plank constant, and E_d is the dispersion energy, which is also a measure of the interband optical transition intensity.

In the Urbach-Martienssen model [40, 41], Urbach energy E_u represents the main parameter that determines the photon capture efficiency of a semiconductor layer. It is defined through the system:

$$\alpha(h\nu) = \alpha_0 e^{(h\nu/E_u)},$$

$$E_u = \alpha(h\nu) \left(\frac{d[\alpha(h\nu)]}{d[h\nu]} \right)^{-1} = h \left[\frac{d}{d\nu} (\ln \alpha(\nu)) \right]^{-1}. \quad (4)$$

Mott and Davis [42] noted that oppositely to crystalline structures, where the fundamental edge is mainly determined by conduction band minimum and valence band maximum levels, ion-doped binary semiconductor compounds present a particular optical absorption edge profile. In these materials, the absorption coefficient increases exponentially with the photon energy near the energy gap. This variation results in “blurring” of the valence-conduction bands and narrows slightly the band gap by appearance of the so-called Urbach tails (Figure 2).

4.2. Experiment. Figures 3 and 4 present, respectively, the reflectance and transmission spectra of the In- and Yb-doped samples.

For the six elaborated samples $A_{\text{Ind},i}|_{i=1..3}$ and $B_{\text{Yb},i}|_{i=1..3}$, it can be noticed that the transmission coefficient increases with doping (from 40% to 85%) while the reflectance remains inside a narrow interval (5%–20%). These observations confirm the XRD-related consequences of doping on crystal c-axis alignment, which are favorable to light transmission.

Moreover, it is obvious that doping alters transmitting performance of the initial oxide ZnO. The synthetic absorption coefficient,

$$\alpha = \frac{1}{d\sqrt{2}} \sqrt{\left(\ln \left(\frac{1-R^2}{T} \right) \right) + \left(\ln \left(\frac{(1-R)^2}{T} \right) \right)^2} \quad (5)$$

FIGURE 3: Transmission spectra of the (a) In-doped and (b) Yb-doped ZnO films.

FIGURE 4: Reflectance spectra of the (a) In-doped and (b) Yb-doped ZnO films.

which is calculated according to [43–46], is involved [47] in the relation:

$$(\alpha h\nu) = A\sqrt{h\nu - E_g}, \quad (6)$$

where A is a constant and E_g is the bandgap energy.

The optical bandgap energy E_g is deduced from Figure 5, which presents plots of $\alpha h\nu$ versus $h\nu$, by extrapolating the straight line portion of each graph to the energy axis.

Similarly, Urbach energy E_u is deduced from local straight line portions slope in the plots of $\ln(\alpha)$ versus $h\nu$ (Figure 6).

On the other hand, the optical characteristics of dispersion $n(\lambda)$ and $k(\lambda)$ (refractive index and extinction coefficient, resp.), for values of the wavelength λ between 300 and 1800 nm, have been calculated using optical experimental measurements and the methods of Bathe and Patil [48] and Belgacem and Bennaceur [23]. The plots of $n(\lambda)$ and $k(\lambda)$ are presented in Figures 7 and 8.

The evolution of the index of refraction $n(\lambda)$ has been fitted to Cauchy [49] law:

$$n(\lambda) = A + \frac{B}{\lambda^2}. \quad (7)$$

The calculated values of A and B , along with E_g and Urbach parameters E_0 , E_d , and E_u , defined in Section 4.1, are gathered in Table 2.

Finally, interaction of the doped layers with electromagnetic radiation has been modeled through complex dielectric constant $\varepsilon(\lambda)$ [50, 51] defined by

$$\varepsilon(\lambda) = (n(\lambda) + ik(\lambda))^2 = \varepsilon_1(\lambda) + i\varepsilon_2(\lambda),$$

$$\varepsilon_1(\lambda) = n(\lambda)^2 - k(\lambda)^2,$$

$$\varepsilon_2(\lambda) = 2n(\lambda)k(\lambda). \quad (8)$$

The calculated values of $\varepsilon_1(\lambda)$ and $\varepsilon_2(\lambda)$ are presented in Figures 9 and 10.

FIGURE 5: Plots of $\alpha h\nu$ versus $h\nu$.

TABLE 2: Relevant Cauchy and Urbach parameters.

Sample	Cauchy constants		Bandgap E_g (eV)	Urbach parameters			
	A	B (nm ²)		E_0 (eV)	E_0/E_g	E_d (eV)	E_u (meV)
$A_{Ind,1}$	1.80	31232.59	3.25	6.10	1.87	13.91	119.68
$A_{Ind,2}$	1.82	28810.59	3.26	6.39	1.95	14.97	93.11
$A_{Ind,3}$	1.923	29376.15	3.35	6.77	2.02	18.44	88.49
$B_{Yb,1}$	1.62	35511.20	3.28	5.25	1.59	8.74	67.19
$B_{Yb,2}$	1.69	18848.93	3.28	6.68	2.03	12.59	51.14
$B_{Yb,3}$	1.73	27190.68	3.28	6.49	1.98	13.34	57.31

The obtained values were used [51] to evaluate ϵ_∞ , ω_p , and τ , high frequency dielectric constant, plasma frequency and relaxation time, respectively, through the relations

$$\begin{aligned} \epsilon_1 &= \epsilon_\infty - \frac{\epsilon_\infty \omega_p^2}{4\pi^2 c^2} \lambda^2, \\ \epsilon_2 &= 2nk \approx \frac{\epsilon_\infty \omega_p^2}{8\pi^3 c^3 \tau} \lambda^3, \\ \omega_p &= \sqrt{\frac{4\pi N e^2}{\epsilon_\infty m_e^*}}, \end{aligned} \quad (9)$$

where N/m_e^* represents free carriers concentration-to-effective mass ratio.

Calculated values of high frequency dielectric constant, plasma pulsation, relaxation time, and free carriers concentration-to-effective mass ratio are gathered in Table 3.

5. Discussion and Analysis

Precedent analyses (Figures 1(a) and 1(b)) showed that the doped layers $A_{Ind,i}|_{i=1..3}$ and $B_{Yb,i}|_{i=1..3}$ developed an enhanced preferred orientation of the crystallites with respect

TABLE 3: Calculated parameters.

Sample	ϵ_∞	ω_p (10^{14} rad·S ⁻¹)	τ (10^{-15} s)	N/m^* (10^{16} g ⁻¹ cm ⁻³)
$A_{Ind,1}$	3.42	2.40	8.09	6.82
$A_{Ind,2}$	3.50	2.73	2.40	9.04
$A_{Ind,3}$	3.86	2.25	0.82	6.80
$B_{Yb,1}$	2.82	2.44	1.87	5.83
$B_{Yb,2}$	3.01	2.83	2.74	8.38
$B_{Yb,3}$	3.20	2.94	10.50	9.57

to the (002) reflection. Since ZnO layers are generally identified by XRD peaks, (101), (100), and (002) in hexagonal wurtzite system (JCPDS card file no. 361451 < $a = 3.24982$, $c = 5.20661$ Å>), the domination of the doped films c-axis-oriented (002) peaks perpendicularly to the glass substrate plane represents a drastic optical enhancement. This feature has been confirmed by mechanical and Optothermal measurements, which recorded an increase of the microhardness of the doped layers along with an obvious decrease of ψ_{AB} .

The actual analyses have been carried out on this basis: the ZnO wurtzite matrix is not meaningfully altered by In or Yb doping. Tiburcio-Silver et al. [52] excluded, in the same context, the existence of any indium or ytterbium chemically

FIGURE 6: Plots of $\text{Ln}(\alpha)$ versus $h\nu$. FIGURE 7: Plots of $n(\lambda)$. FIGURE 8: Plots of $k(\lambda)$.

FIGURE 9: Plots of $\epsilon_1(\lambda)$. FIGURE 10: Plots of $\epsilon_2(\lambda)$.

FIGURE 11: Doping ions intercalation inside ZnO wurtzite matrices.

established compound. In other words, In^{3+} and Yb^{3+} ions seem to be incorporated inside ZnO wurtzite unaltered matrices (Figure 11).

Since In^{3+} , Al^{3+} , and Yb^{3+} have the same valences and perform close oxygen-related electronegativity, the explanation of concentration-related stark differences of evolution of Urbach energy (Figure 12) and free carriers lifetime (Figure 13) should be investigated from the side of the doping element distinctive physical properties.

In fact, the regular decrease of Urbach energy and free carriers lifetime (Figures 12 and 13) for In-doped layers, as recorded also by Cody et al. [53] for incrementally aluminum-doped binary compounds, contrasts with the Yb-doped layers behavior. A phenomenon of saturation seems to occur for increasing doping amounts inside these latter layers (Figure 11).

In this context, nanoscale comparative estimation of atomic and ionic radii revealed an intriguing element: among the prospected entities, ytterbium represents the highest radius, which is moreover comparable to upper ZnO–O bond dimension inside ZnO wurtzite matrix (Figure 11).

As Dao [54], Wasim et al. [55], Narayanan et al. [56], Tauc et al. [57], and Grus and Sikorska [58] evoked a strong dependence of the Urbach energy E_u versus electron-phonon

FIGURE 12: Doping-related Urbach energy evolution.

FIGURE 13: Doping-related free carriers lifetime evolution.

and exciton-phonon interactions as well as crystalline structural static disorder, radiation inside the crystal and deviation from the ideal stoichiometry, it can consequently be suggested that indium incremental incorporation does not induce any meaningful additional static disorder, due to size adequacy and ion-matrix compatibility, while high-sized atoms (like ytterbium) cease to be incorporated at a given amount level and hence disturb the global layer crystalline order and widen Urbach tails.

6. Conclusion

Zinc oxide layers have been prepared and doped with indium and ytterbium using a low cost spray pyrolysis process. During elaboration and characterisation phases, it has been verified that in absence of any conventionally verified chemical reaction of In^{3+} and Yb^{3+} ions, the wurtzite structure of ZnO crystals was globally unaltered. Comparison between In^{3+} and Yb^{3+} doping in terms of Urbach energy and free carriers lifetime led to interesting observations. The

intriguing problem of the doping-related Urbach tails distortion has been discussed relatively to atomistic nanoscale considerations and some explanations have been proposed. Further studies on vanadium- (V-) and molybdenum- (Mo-) doped samples are actually in progress in order to confirm the suspected effects of ionic size on Urbach tails width and carriers lifetime.

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