Effect of yttrium(Y) on structural, morphological and transport properties of CdO thin films prepared by spray pyrolysis technique

S. Ahmed, M. S. I. Sarker, M. M. Rahman, M. Kamruzzaman, M. K. R. Khan

Abstract

Cadmium oxide (CdO) and yttrium (Y) doped CdO (Y: CdO) thin films have been prepared onto glass substrate at temperature 300 °C by spray pyrolysis technique. The effects of yttrium (Y) doping on the structural, morphological, optical and electrical properties were studied systematically. The X-ray diffraction (XRD) study confirms that CdO films are polycrystalline in nature with cubic structure having lattice parameter of 0.4658 nm. Surface topographic and nano-structural analysis indicates cluster grain size and porosity decreased substantially with increase of yttrium (Y) content in CdO films. The optical transmittance exhibits excellent optical transparency, with an average transmittance of >70% in the visible range for 2 to 4% yttrium (Y) doping. The optical band gap widens in Y: CdO film from 2.24 to 2.62 eV through Burstein- Moss shift. Hall measurement confirms that material is of n type with a minimum resistivity of $4.7 \times 10^{-4}$ Ω-cm with carrier concentration of $4.2 \times 10^{21}$ cm$^{-3}$ were achieved for 2% yttrium (Y) doping.

Keyword: Materials science
1. Introduction

Research on metal oxide thin films are growing day by day due to their wide variety of uses in electronic, plasmonic, optical, thermal, and solar energy devices. Special attention paid on transparent conducting oxides (TCOs) CdO, ZnO, CuAlO2 and SnO2 due to their high conductivity and high transparency in the visible region of electromagnetic radiation [1, 2, 3, 4]. Moreover, tremendous effort has given in recent past to CdO thin film due to its important role to both fundamental and breakthrough development of technologies in various areas such as solar cells [5], interlayers for diodes [6], gas sensor [7], phototransistor [8], thin film photovoltaic [9], information storage [10] and many other opto-electronic devices. This is due the fact of Cadmium oxide (CdO) is an n-type semiconductor having direct band gap of 2.20 eV [11] with a cubic crystal structure. This low optical band gap of CdO offers the possibility of tuning the transparency and electronic structure with doping elements such as Ga, In and Y through Burstein Moessner effect [12]. CdO films also exhibits high electrical conductivity due to the presence of shallow donors caused by intrinsic interstitial cadmium atoms and oxygen vacancies; the transmittance and direct band gap of CdO can be modified by doping with other element to develop the design of opto-electronic devices [13, 14]. Yttrium (Y) doped CdO thin films grown on sapphire exhibits similar characteristics to that of dysprosium (Dy) doping [15] maintaining mobility 400 cm² V⁻¹ s⁻¹ over the dopant range 1.8 × 10¹⁹ cm⁻³ to 3.3 × 10²⁰ cm⁻³. The electron mobility surpassing 500 cm² V⁻¹ s⁻¹ at carrier density higher than 5 × 10¹⁹ cm⁻³ in Dy-doped CdO satisfy the condition for mid-infrared plasmonics and reduce the optical losses as reported by Sachet et al. [16]. Kyle P. Kelley et al. [17] reported that the carrier density and mobility of yttrium doped cadmium oxide thin film increased by increasing Y-flux accompanying a broad plateau of mobility in between carrier density 5 × 10¹⁹ cm⁻³ and 5 × 10²⁰ cm⁻³.

Transparent conducting CdO films have been fabricated by numerous methods, such as sol-gel [18], metal organic chemical vapor deposition (MOCVD) [19], molecular beam epitaxy [20], sputtering [21], pulsed laser deposition [22], thermal decomposition technique [23], chemical bath deposition [24] and spray pyrolysis technique [25]. Moreover, Y: CdO thin films by MOCVD and sol-gel spin coating techniques [26, 27] have been reported. Among these methods, spray pyrolysis (SP) has some advantages when compared with other methods. It offers flexibility in process mechanism, large area and nano-structured film preparation, also low cost technique but effective. However, so far study on Y: CdO films prepared by SP technique yet to be reported. Therefore, using this versatile technique we have made an attempt to prepare and study the Y: CdO films. Moreover, dilute concentration has been selected because in spray method we need homogeneous solutions so that during spray,
chemical reaction takes place in proportion, results in homogeneous thin film in its whole volume.

In this research, we have prepared CdO and Y: CdO films and study the effects of yttrium (Y) on the structural, electrical and optical properties of n-type Y: CdO films. In order to use in the opto-electronic device applications, CdO films need higher optical transmittance in visible light region with suitable electrical conductivity. Therefore, the main objective of this work is to explore the applicability of CdO films in opto-electrical devices by yttrium (Y) doping.

2. Experimental

CdO and Y: CdO thin films were prepared from 0.1 M aqueous solution containing cadmium acetate dihydrate [Cd(CH\textsubscript{3}COO)\textsubscript{2} \cdot 2H\textsubscript{2}O] (0.1 M/L) dissolved with 50 ml of deionized water. Yttrium acetate tetrahydrate [Y(CH\textsubscript{3}COO)\textsubscript{3} \cdot 4H\textsubscript{2}O] was added to the solution for concentration of 0, 1, 2, 3, and 4 at % of yttrium (Y). Pure CdO and Y: CdO films were deposited onto glass substrate using the simple SP technique. The well cleaned glass substrates were placed on the heater and a distance of 21 cm is maintained between the tip of the nozzle and the surface of the glass substrate. The flow rate and gas pressure of the solution were kept constant at 5 ml/min and 10\textsuperscript{5} N/m\textsuperscript{2} respectively and the substrate temperature was fixed at 300 °C using a copper-constantan thermocouple. Thereafter, the compressed air along with the precursor solution was allowed to pass through the nozzle at constant pressure to reach the reactor zone where the film was deposited on the heated substrate. Since the growth parameters were kept constant for all the films, therefore, the thicknesses of all the films remain constant and the average thickness was 150 nm measured by Newton’s ring method. The possible chemical reactions that take place on the heated glass substrate to form pure CdO and Y: CdO films are as follows:

\[
\text{Cd(CH}_3\text{COO)}_2 \cdot 2\text{H}_2\text{O} + \text{H}_2\text{O} \xrightarrow{300°\text{C}} \text{CdO} + \text{CO}_2 \uparrow + \text{CH}_4 \uparrow + \text{steam} \uparrow
\]
\[
\text{Y(CH}_3\text{COO)}_3 \cdot 4\text{H}_2\text{O} + \text{Cd(CH}_3\text{COO)}_2 \cdot 2\text{H}_2\text{O} + \text{H}_2\text{O} \xrightarrow{500°\text{C}} \text{Y: CdO} + \text{CO}_2 \uparrow + \text{CH}_4 \uparrow + \text{steam} \uparrow
\]

To study the crystallographic structure of the pure CdO and Y: CdO films, a X-ray diffractometer D2 PHASER (BRUKER), with Cu\textsubscript{\text{Kα}} monochromatic radiation of wavelength \( \lambda = 0.154056 \text{ nm} \), tube current 10 mA and voltage 30 kV was used to record diffraction profile in between Bragg angle of (20—80)°. The film surface morphology of CdO and Y: CdO films were examined by scanning electron microscope (SEM) (Philips XL30 FEG). The optical transmittance \( T(\lambda) \) and reflectance \( R(\lambda) \) spectra of pure CdO and Y: CdO films were taken using a UV-visible spectrophotometer (UV-1601 PC SHIMADZU). To measure the electrical properties, samples were provided with four electrical contacts by silver paste. The carrier concentration (\( n \)) and mobility (\( \mu_H \)) of the films were estimated from Hall voltage...
measurement. The resistivity ($\rho$) of the films for different temperatures was determined by Van-der Pauw’s method of four probe arrangement.

3. Results and discussions

3.1. Structural properties

The XRD patterns of the deposited CdO and Y: CdO thin films are shown in Fig. 1. The characteristic peaks were found at $2\theta = 33.9997^\circ$, 39.4076$^\circ$, 56.3208$^\circ$, 66.8005$^\circ$ and 70.1883$^\circ$ corresponding to the plane (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) of standard values of cadmium oxide (JCPDS card no. 75-0592), indicating all samples are polycrystalline in nature with a face centered cubic structure. XRD patterns also confirm no impurity phases or structure destabilization. The higher intensities for (1 1 1) and (2 0 0) plane indicates the direction of crystal axis. The peak shift for 2% and 3% Y: CdO to the lower angle may be happened due to the replacement of Cd$^{2+}$ ions by Y$^{3+}$ ions in CdO lattice. This can be explained more explicitly from the lattice parameter 'a' calculation using the relation,

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

The average lattice parameter was determined using Nelson-Riley extrapolation method. The Nelson-Riley function can be represented as,

![Fig. 1. XRD patterns of CdO and Y: CdO films for 1 to 4% Y doping levels.](https://doi.org/10.1016/j.heliyon.2018.e00740)
where, $F(\theta)$ is the Nelson-Riley function and $\theta$ is the usual Bragg angle. The average crystal lattice parameter can be obtained using least square fit method from the plot of the lattice parameter ‘$a$’ versus $F(\theta)$ with respect to all planes of CdO films [28]. The average lattice constant of the un-doped CdO film was found as $a = 0.4658$ nm which is a bit smaller than the reported value $a = 0.4694$ nm [29], which is ascertained to the lattice contraction [30] or to the presence of oxygen vacancies [31]. The lattice constant ‘$a$’ remains nearly constant for 1% (0.4566 nm) and 4% (0.4564 nm) of yttrium (Y) doping in CdO crystal but for 2% (0.4578 nm) and 3% (0.4562 nm) yttrium (Y) doping it increases (as shown Table 1). This is due to the fact that covalent atomic radius of $Y^{3+}$ ion (0.180 nm) is greater than that of $Cd^{2+}$ (0.149 nm). The increase of $a$ for the doping of yttrium (Y) further suggest the replacement of $Cd^{2+}$ ion by the $Y^{3+}$ ions and it introduce stress in the system. The crystallite size, $D$ of the sample was determined using Scherrer’s formula,

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

(4)

The dislocation density, $\delta$ and strain, $\varepsilon$ were calculated using the following relations,

$$\delta = \frac{1}{\varepsilon^2}, \quad \varepsilon = \frac{\beta \cos \theta}{4}$$

(5)

where, $\lambda$ is the wavelength of X-ray radiation and $\beta$ is the full width at the half-maxima of the diffraction peaks. Calculated crystallite size shows unsystematic variation with respect to doping level of yttrium (Y) for (1 1 1) plane and are presented in Table 1. This type of variation may be due to the dependence of grain size mainly on substrate temperature, solution flow rate, vibration of the system, ion density of the solution, etc. Therefore, it is difficult to correlate the dependence of crystallite size with percentage of yttrium (Y) doping.

Table 1. Structural parameters of CdO and Y: CdO films for (1 1 1) plane.

| Doping (%) | Lattice constant (nm) | $D_{\text{scherrer}}$ (nm) | Dislocation density, $\delta \times 10^{15}$ lines m$^{-2}$ | Microstrain, $\varepsilon \times 10^{-4}$ lines$^{-2}$ m$^{-4}$ |
|------------|----------------------|-----------------------------|---------------------------------|-------------------|
| 0          | 0.4567               | 38                          | 2.27                            | 0.16              |
| 1          | 0.4566               | 21                          | 1.48                            | 0.13              |
| 2          | 0.4578               | 19                          | 1.89                            | 0.15              |
| 3          | 0.4652               | 42                          | 3.46                            | 0.19              |
| 4          | 0.4564               | 19                          | 2.77                            | 0.18              |
3.2. Surface morphology

Fig. 2 shows the SEM images of CdO and Y: CdO films. It is clearly seen from Fig. 2 that the nano structure are Cauliflower type up to 2% yttrium (Y) doping and the size of the flowers strongly dependent on yttrium (Y) content. With the increase of yttrium (Y) content, flower size being smaller and turns into spherical grain.

Obtained spheroid grains may be implemented for solar cells application. It is evident from Fig. 2 that the surface roughness, pore volume and grain size decrease with the increase of Y content in CdO due to the intrinsic stress may be caused by the incorporation of foreign atoms.

3.3. Optical properties

3.3.1. Study of film transparency

Optical properties of a series of undoped CdO and Y: CdO films have been studied from the transmittance and reflectance spectra as shown in Fig. 3. It is observed from Fig. 3(a) that the transmittance of Y: CdO films were greater than that of the undoped CdO films. Since transmittance depends on material characteristics as well as film thickness, the increasing of roughness with the increase of Al doping in CdO film decreased the transmittance as reported by Helen et al. [32]. On the contrary, in our case roughness of the film is decreased with the increasing of the % of yttrium (Y) doping in CdO films, as illustrated from SEM image in Fig. 2. Increasing the % of yttrium (Y) doping may lead the formation of stoichiometric film with increasing transmittance.

Fig. 2. SEM images for (a) 0% Y and (b), (c), (d), (e) for 1%-4% Y doped CdO films.
Moreover, the transmittance of the Y: CdO films increased with the increase of yttrium (Y) concentration due the change in grain size as well as Y$^{3+}$ ions may replace the Cd$^{2+}$ ions or may take the position of oxygen vacancies in the films which also cause the slight change in free carrier density [33, 34]. From Fig. 3(b) we observed that the reflectance of the un-doped CdO and 1%, 2% and 3% Y doped CdO films remain nearly unchanged in visible range but decreased both in UV and near IR region. Moreover, the reflectance increased with the increase of yttrium (Y) doping levels. Therefore, it is clear that the optical properties strongly depend on yttrium (Y) doping concentrations in CdO.

### 3.3.2. Optical bandgap

The optical band gap of the CdO and Y: CdO films was estimated through the calculation of absorption coefficient, $\alpha$ from transmittance data using the relation,

$$\alpha = \frac{1}{t} \ln \left( \frac{1}{T} \right)$$

(6)

where, $t$ is the film thickness and $T$ is the transmittance. The optical direct band gap $E_g$ was obtained by using Tauc’s relation [35],

$$\alpha h\nu = A(h\nu - E_g)^n$$

(7)

where, $\alpha$ is the absorption coefficient, $h\nu$ is the photon energy, $A$ is a constant and $n$ is another constant whose value assumes 1/2 for allowed direct transition. $E_g$ is the optical band gap which can be determined from the extrapolation of the linear section of the plot of $(\alpha h\nu)^2$ versus photon energy $h\nu$. Fig. 4 shows the plot of $(\alpha h\nu)^2$ versus photon energy $h\nu$ of CdO thin film as a function of yttrium (Y) doping. The intercept of the energy axis provided the values of $E_g$ which increased from 2.24 eV (undoped) to 2.62 eV with the increase of yttrium (Y) doping level. Such increase
in the band gap can be interpreted due to the fact of Burstein-Moss band gap widening [12]. For degenerate semiconductor like CdO, the Fermi level lies within the conduction band where its position depends on the free electrons density. Determined optical band gaps are related to the excitation of the electrons from the top of the valence band to the bottom of the conduction band. In Y: CdO thin film, the probability of donor states may be increased and at the same time oxygen vacancies may be decreased in any form in the system. Therefore, lifting of the Fermi level into the conduction band of the degenerate semiconductor due to the increase in the carrier density provides the energy band broadening.

3.3.3. Optical constants

The refractive index, \( n' \) and extinction coefficient, \( k \) was obtained by computing the reflection and transmission data using following equations,

\[
n' = \left( \frac{1 + R}{1 - R} \right) + \sqrt{\left( \frac{1 + R}{1 - R} \right)^2 - 1 - k^2}
\]

\[
k = \frac{\lambda}{4\pi t} \ln \left[ \frac{1}{T} \right]
\]

The variation of refractive index, \( n' \) and extinction coefficient, \( k \) with photon energy \( (hv) \) is shown in Fig. 5(a) and (b), respectively.

It is seen from Fig. 5(a) that the refractive index, \( n' \) of the Y: CdO films was greater than that of undoped CdO films and which varied with doping level of Y. Whereas
the extinction coefficient, \(k\) of Y: CdO films in Fig. 5(b) was found less than that of the undoped CdO and which decreased with decreasing doping level. The maximum refractive indices both for undoped CdO and Y: CdO films were found almost at photon energy 1.50 eV after which \(n'\) decreases slowly. On the other hand, the extinction coefficients, \(k\) of the undoped CdO and Y: CdO films remain almost constant up to band gap energy after which \(k\) increased with the increase of photon energy. The increase of \(n'\) with increasing the doping level of yttrium (Y) seems to happen due to the normal dispersion occurred before the absorption edge followed by an anomalous dispersion.

3.3.4. PL analysis

Photoluminescence (PL) is exploited to study the optoelectronic properties of semiconducting CdO and Y: CdO thin films, which depends on grain size, morphology and interaction between carriers. Fig. 6 shows the photoluminescence spectra of the Y: CdO thin films at room temperature with an excitation wavelength of 350 nm.
and 450 nm. In Fig. 6(a) PL spectrum show emission peak for Y: CdO films in the visible range centered at \( \lambda = 472 \) nm. This blue peak at \( \lambda = 472 \) nm may be attributed due to the excitonic transitions which is size-dependent and excitation wavelength-independent in certain wavelength range. Zhao et al. [19] observed a similar peak at 493 nm (2.48 eV), for CdO thin films which they treated as donor related luminescence peak. They attributed this green peak to electron transition from the bottom of the conduction band to the O\(_{\text{Zn}}\) level. In our study, we found the highest emission intensity for 2% Y doped CdO films. This may be happened due to the less yttrium (Y) content in CdO and which may be responsible to initiate excitonic transitions. Again, for higher yttrium (Y) concentration in CdO, the emission peak intensity decreases linearly but it does not show any detectable shift. Observed low intensity peaks could not be treated to any emission process but may be regarded as noise or surface defects in the films. For higher excitation energy, 2.75 eV (\( \lambda = 450 \) nm) deep level photo emissions were observed as shown in Fig. 6(b). These peaks are quite sharp than near band edge peaks which may be treated as red emission peak corresponds to low energy.

4. Electrical properties

Temperature dependent resistivity (\( \rho \)) of CdO and Y: CdO films have been studied by Van-dar-Pauw method of four probe arrangement. Fig. 7(a) shows the electrical resistivity (\( \rho \)) as a function of temperature of the CdO and Y: CdO films. The resistivity of the undoped CdO films decreased lightly with the increase of temperature which confirmed semiconducting behavior of CdO. It is found that the resistivity of the film was highest when 1% Y doped in CdO. In this case, Y\(^{3+}\) ions may not form any substitutional solid solution rather it may take the position in grain boundary of CdO lattice. Therefore, in this stage yttrium (Y) dopant sites may not contribute mobile charge carriers. Poor crystal orientation may also be responsible for this higher resistivity [36]. For 2% and 3% Y doping CdO films showed lower resistivity than undoped CdO film.

Fig. 7. Variation of (a) resistivity with temperature and dependence of (b) resistivity, carrier concentration, and mobility for un-doped CdO and Y: CdO films at room temperature.
It was also observed that resistivity of 2% and 3% Y doping in CdO films increased slightly with increasing temperature which seems to show poor metallic behavior. In this case, $Y^{3+}$ ions may act as effective dopant to replace $Cd^{2+}$ ions sites in the lattice and made substitutional solid solution donating electrons to increase the charge carriers. Further increasing doping level i.e. for 4% Y in CdO, the resistivity of the films again increased but lower than 1% Y doping. This may be happened due to scattering and may also be due to the highest thermodynamic affinity for oxygen of yttrium (Y) [37]. Therefore, doping level of 2–3% Y is suitable to obtain less resistive films of CdO.

The variation of room temperature resistivity with doping concentrations is shown in Fig. 7(b). Room temperature resistivity increases from $6.2 \times 10^{-4}$ to $13.8 \times 10^{-4}$ ($\Omega$-cm) when the percentage of yttrium (Y) increases from 0 to 1%, but further increase of 2–3% Y, decreases the resistivity to $4.7 \times 10^{-4}$ ($\Omega$-cm) and again increase the resistivity for 4% Y doping. The decrease in electrical resistivity with the yttrium (Y) doping can be explained as follows: the concentration of free charge carriers in CdO increases with the yttrium (Y) doping because it has one valance electron more than cadmium [38]. We may consider that yttrium (Y) substitutes the cadmium atom, or it occupies the interstitial sites and in both cases yttrium (Y) acts as the donor. But for 1% and 4% Y doping may destroy the crystalline structure, as the dopant hold the interstitial or grain boundary position for 1% Y doping or for excess strain introduced for over doped 4% Y and also due to the increase absorption of the free carriers, the resistivity of Y: CdO thin films increased.

Hall measurement was done at room temperature for undoped and Y: CdO films at constant magnetic field of 0.9815 T. From the measured value of Hall constant ($R_H$), we calculated the carrier concentration ($n$) for different percentage of Y: CdO using the relation $n = 1/eR_H$, where $e$ is the electronic charge. The mobility ($\mu_H$) of the carriers was calculated from the relation, $\mu_H = \sigma R_H$. Hall measurements reveal that CdO and Y: CdO films are n-type. Fig. 7 shows the variation of carrier concentration ($n$), mobility ($\mu_H$) and resistivity ($\rho$) of CdO films with respect to Y doping. The maximum carrier concentrations, ($n$) was found for 2% Y: CdO films as $4.2 \times 10^{21}$ cm$^{-3}$. This occurs because yttrium (Y) atoms can replace cadmium (Cd) atoms and donate extra electrons to the charge carriers. On the contrary, for 1%, 3% and 4% Y doping, carrier concentration is decreased. These phenomena suggest the yttrium (Y) dopants readily ionized for 2% doping and for other percentages, doping acts as neutral impurities. At the same time the Hall mobility, ($\mu_H$) for Y: CdO films was first decreases, reaching minimum up to $3.90$ cm$^2$/V-s at 2% Y doping, and then increases. In this case, Y: CdO films may provide less mobile charge carriers or there may increase carrier scattering.

The decrease of carrier mobility below 2% Y may be explained from the point of view of ionized impurity scattering and grain boundary scattering. It is reported that increase
of doping can lower the potential barrier height of grain boundaries, which makes it easier for charge carriers to move across grain boundaries [1]. This will increase the carrier mobility. Second, additional doping can cause the formation of more large-angle grain boundaries. The increase of large angle grain boundaries and the increase of both ionized and neutral impurity centers will increase charge carrier scattering which results in the decrease of carrier mobility. However, in this study our result suggests opposite behavior. SEM image of Fig. 2(b), (c) suggests that up to 2% Y doping the grain size is increased, results in the decrease of carrier mobility. The increase of mobility after 2% Y doping is due to the lower potential barrier height which make the charge carriers to move easily across the grain boundaries.

5. Conclusion

Highly conductive and transparent Y: CdO thin films have been obtained through SP technique. Doping yttrium (Y) on CdO has a great effect on structural, surface morphology, optical and electrical properties of the films. Optical transmittance of Y: CdO films increases promisingly relative to the as-deposited CdO films in visible light region. Optical band gap increased from 2.24 eV to 2.68 eV and refractive index also increased for Y: CdO films due to Burstein-Moss effect. The 2% Y doped CdO film exhibits lowest resistivity of 4.7 × 10⁻⁴ Ω-cm with maximum carrier concentration of 4.2 × 10²¹ cm⁻³. Lower mobility of 2% Y: CdO attributed to the large angle grain boundary scattering. In short, yttrium (Y) is an effective dopant element for CdO to introduce advance opto-electronic devices.

Declarations

Author contribution statement

Sumon Ahmed, M. Kamruzzaman: Performed the experiments; Analyzed and interpreted the data.

Md. Samiul Islam Sarker: Contributed reagents, materials, analysis tools or data; Wrote the paper.

M. M. Rahman: Conceived and designed the experiments; Analyzed and interpreted the data.

Md. Khan: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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