Structural and optical characterization of sprayed Mg and Ni co-doped CdS thin films for photovoltaic applications

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Abstract. In the present work, Mg and Ni co-doped CdS thin films are grown on glass substrates at a temperature of 400 °C through spray pyrolysis. The influence of Mg-doping on structural, morphological and optical properties of CdS:Ni thin films are examined. Mg level is changed from 0 % to 7 % for CdS: Ni samples just as Ni concentration is fixed 2 % for all CdS thin films. It is observed from the X-ray diffraction spectra that all the samples exhibit hexagonal structure of CdS thin films. The scanning electron microscopy (SEM) images showed a dense surface structure composed of crystallites whose average size increase with increasing the Mg doping. The optical transmission curves demonstrate that CdS:Ni thin films exhibit a best optical transparency in the visible range for 1 % Mg content compared to other specimens. The energy of the optical band gap tends to decrease from 2.46 to 2.40 eV with increasing Mg concentration in CdS: Ni.

Keywords: CdS, (Ni-Mg) co-doping, Structural properties, Optical properties, spray pyrolysis.

Abbreviations

\( \varepsilon_s \): structural strain.
\( \sigma_{st} \): compressive stress (GPa).
\( T_C \): texture coefficient of the hkl planes.
\( D_{hkl} \): Average crystallites size (nm).
\( \beta_{hkl} \): (FWHM) width half-height.
\( \theta_{hkl} \): diffraction angle
\( \alpha \): absorption coefficient
\( h \): Planck constant \( 6.62607004 \times 10^{-34} \) m\(^2\) kg / s
Eg: optical band gap (eV)
T: Optical transmission

1. Introduction

Cadmium sulphide CdS among the II-IV semiconductors with CdTe and CdSe, are materials of great interest in many practical applications, such as solar cells, optoelectronic devices, optical detectors, and so on. CdS characterized by good transmission, and also a direct gap with a forbidden bung width of the order of 2.42 eV, and n-type conductivity. The CdS uses, in the solar cells, as optical buffer layer for the heterojunctions, CdS / ZnS [1], CdTe / CdS [2], CdTe / CdS / ZnS [3], CdS / Cu (In, Ga) Se₂ [4].

The CdS has been developed by forming thin layers by several chemical methods such as chemical deposition bath [5], spray [6], dip coating [7], spin coating [8] CVD [9]. Some of these are pulsed laser deposition (PLD)[10],SILLAR [11] and metal organic chemical vapour deposition (MOCVD) [12]. The liquid phase spray technique is suitable for thin film deposition on large surfaces, it is also characterized by low cost and easy production process, and also provides high quality films and good adhesion to substrates.

The CdS doped by several elements to improve these physico-chemical properties, the literature shows several works on the doping of CdS by a single element, such as the elements of group III Al [13], In [14], Ga [15] and B [16] to improve the structural and electrical properties of CdS , Furthermore, by means of doping II-VI semiconductors with alkali metals like Na [17], K [18], and transition metals as Cu [19], Ni [20], Zn [21], Ag [22], Mn [23], Fe[24]. The co-doping of CdS with two elements of different natures such as a transition element Ni and an element Mg is not widely studied. Ni²⁺ and Mg²⁺ have standard ion rays of 0.069 nm and 0.072 nm, respectively, slightly lower than that of Cd²⁺ (0.097 nm). This information makes it possible to predicting that Mg and Ni ions can substitute for Cd²⁺ ions in the crystalline structure of CdS may improve the opto-electrical properties of CdS thin films.

The work of Mg and Ni co-doping in CdS has not been reported in the literature until recently. Hence, in this work, the investigation of the influence of Mg doping on structural, morphological and optical proprieties of 2 at% Ni doped CdS thin films prepared by spray pyrolysis has been carried out for first time. The technique of spray pyrolysis is used in this work successfully, this technique is quite simple and the required configuration is less time consuming and flexible for process modifications. In addition, using this technique, it is possible to produce large-area films without the need for a high vacuum and the films produced can be controlled step by step.

2. Experimental procedures

1.1 Preparation of thin films by spray pyrolysis

The nickel-magnesium co-doped CdS thin layers are prepared from a solution containing the basic precursors namely cadmium chloride hexahydrate (CdCl₂, 6H₂O), as a source of cadmium, and thiourea (CS (NH₂)₂), as a source of sulfur. To this initial solution we add a solution of nickel chloride hexahydrate (NiCl₂, 6H₂O) of a fixed concentration, such that the ratio [Ni] / [Cd] equal to 2%. Add a solution of magnesium sulfate MgSO₄ of different concentration by varying the ratio [Mg] / [Cd] between 0 and 7%. The solutions obtained are stirred vigorously with a magnetic stirrer for 20 minutes and then sprayed into fine droplets by means of a perfume atomiser on the preheated glass substrates at 400°C.

1.2 Characterizations
The as deposited films show good adhesion to the substrate and exhibit a yellow color. The crystal structure and phase transition for undoped and Mg and Ni co-doped CdS thin films were determined with a diffractometer ‘XPERT-PRO’ model using CuKα radiation with 2θ ranging from 10° to 70°. The surface morphologies of the deposited films were observed using scanning electron microscopy (SEM). Optical measurements were carried out with a SHIMADZU 3101 PC UV-VIS-NIR spectrophotometer in the wavelength range of 300-2500 nm.

3. Results and Discussion

2.1 Structural properties

The results of the X-ray diffraction on the thin layers of cadmium sulphide give very clear peaks, Figure 1 shows the spectra of the X-ray diffraction of the thin layers of CdS co-doped with nickel and magnesium. These spectra show several diffraction lines indicating that the resulting CdS layers are polycrystalline. The lines of the CdS sample doped with 2% Ni are located at 2θ = 25.00°, 26.70°, 28.37°, 36.88°, 43.94°, 48.06°, and 52.06° (Figure 1. (a)) showing that CdS exhibits polycrystalline behavior with a hexagonal crystalline structure [25], due to the diffraction planes (100), (002), (101), (102), (110), (103), (112), with the most intense peak (002) which correspond to the preferred orientation [26]. After the doping of Cd$_{0.98}$Ni$_{0.02}$S with 1% Mg, the peak (002) remains more intense, which implies that the preferential orientation is retained, with a remarkable increase in the peak (101). But, when Cd$_{0.98}$Ni$_{0.02}$S doped with 3% Mg the peak (101) becomes more intense, which shows that the preferential orientation is changed towards the plane (101) with the decrease of the peaks (102) and (103), and the birth of a new peak located at 2θ = 53.11°. After the doping of the CdS at 5% and 7% there is a decrease in the peak (101) which can be attributed to the low degree of crystallinity. It should be noted that no peak related to Ni and Mg metal elements and their compounds including NiS, MgS, MgO, NiO, etc. can be observed.

The values of the inter-reticular distances d$_{hkl}$ corresponding to the (100), (002), (101), (102), (110) and (103) planes are compared with those reported in reference [25]. A mapping of the calculated d$_{hkl}$ values to the standard values confirms the hexagonal crystal structure (wurtzite) of CdS for all deposited layers. It is well known that CdS has two crystal structures; hexagonal (wurtzite) and cubic (blende). The latter is considered the most stable phase between 50 and 900°C. In addition, the hexagonal structure of CdS thin films is preferable for the applications of this material in solar cells because of the parameters of the CdS and CuInSe$_2$ cells (absorber material in the solar cell) which are very close. The difference is 0.7% while that between cubic CdS and CuInSe$_2$ is 1.2% [27].
Figure 1. XRD data of a) Cd$_{0.98}$Ni$_{0.02}$S, b) Cd$_{0.97}$Ni$_{0.02}$Mg$_{0.01}$S, c) Cd$_{0.95}$Ni$_{0.02}$Mg$_{0.03}$S, d) Cd$_{0.93}$Ni$_{0.02}$Mg$_{0.05}$S and e) Cd$_{0.91}$Ni$_{0.02}$Mg$_{0.07}$S thin films.

Figure 2 shows a displacement $\Delta$ ($2\theta_{101}$) of the position of the diffraction line (101) of CdS co-doped with Ni and Mg towards the large Bragg angles. It goes from 28.37° for a layer of Cd$_{0.98}$Ni$_{0.02}$S to 28.42° for Cd$_{0.95}$Ni$_{0.02}$Mg$_{0.03}$S. This peak displacement results from the structural stress associated to the co-doping with Ni and Mg, calculated by the relation [28].

$$\varepsilon_s = -\Delta(2\theta_{101})\cotg(\theta_{101}) \ (1)$$

This structural stress is caused by a compressive stress $\sigma_s$ which can be estimated by:

$$\sigma_s = (\varepsilon_s)B \ (2)$$

With B a “bulk modulus” constant of CdS estimated by the value of 69.44 GPa [28].
Figure 2. Variation of the (101) peak position of Cd$_{0.98}$Ni$_{0.02}$S, Cd$_{0.97}$Ni$_{0.02}$Mg$_{0.01}$S, Cd$_{0.95}$Ni$_{0.02}$Mg$_{0.03}$S, Cd$_{0.93}$Ni$_{0.02}$Mg$_{0.05}$S, and Cd$_{0.91}$Ni$_{0.02}$Mg$_{0.07}$S thin films.

Figure 3 also shows that the compressive stress varies between 0.08 to 0.14 GPa for 1 % and 3 % Mg doped CdS:Ni. This small decrease in the value of the constraint is not able to create a change in the crystalline structure, it can only produce a slight decrease in the lattices parameters (Table 1). The lattice parameters, a and c, are calculated from the peaks position using the formula of hexagonal system (Eq. (3)).

\[
\frac{1}{d^2_{hkl}} = \frac{4(h^2 + k^2 + hk)}{3a^2} + \frac{l^2}{c^2} \quad (3)
\]

Table 1: Lattice parameters of Cd$_{0.98}$Ni$_{0.02}$S, Cd$_{0.97}$Ni$_{0.02}$Mg$_{0.01}$S, Cd$_{0.95}$Ni$_{0.02}$Mg$_{0.03}$S, Cd$_{0.93}$Ni$_{0.02}$Mg$_{0.05}$S, and Cd$_{0.91}$Ni$_{0.02}$Mg$_{0.07}$S thin films.

| Sample            | a=b (Ǻ) | a=b (Ǻ) | a=b (Ǻ) | a=b (Ǻ) |
|-------------------|---------|---------|---------|---------|
| Cd$_{0.98}$Ni$_{0.02}$S | 4.112   | 4.120   | 6.678   | 6.682   |
| Cd$_{0.97}$Ni$_{0.02}$Mg$_{0.01}$S | 4.111   | 4.120   | 6.674   | 6.682   |
| Cd$_{0.95}$Ni$_{0.02}$Mg$_{0.03}$S | 4.109   | 4.120   | 6.674   | 6.682   |
| Cd$_{0.93}$Ni$_{0.02}$Mg$_{0.05}$S | 4.115   | 4.120   | 6.684   | 6.682   |
| Cd$_{0.91}$Ni$_{0.02}$Mg$_{0.07}$S | 4.114   | 4.120   | 6.680   | 6.682   |

However, with increasing doping up to 5 % and 7 % with Mg, the 'a' and 'c' 'values increase, which can be explained by the fact that Mg$^{2+}$ ions take up the interstitial sites in the CdS structure. The interstitial site...
occupancy by \( \text{Mg}^{2+} \) ions causes an enlargement in the volume of the crystal lattice of CdS, causing a decrease in the quality of the crystal.

![Graph showing structural strain and stress of Mg and Ni co-doped CdS.](image)

**Figure 3.** Structural strain and stress of Mg and Ni co-doped CdS.

The preferred orientations of Mg and Ni co-doped CdS layers, giving an indication of the degree of crystallization of the lattice planes (hkl) were evaluated by the coefficient of texture \( (T_C) \) of the two main planes (002) and (101), this coefficient is calculated from the data of the X-ray diffraction spectra, using the formula [29].

\[
T_C(hkl) = \frac{2\pi I_0 I}{N} 
\]

(4)

Where \( T_C(hkl) \) is the texture coefficient of the planes (hkl), \( I \) is the measured intensity, \( I_0 \) is the standard intensity of CdS, and \( N \) is the number of reflection peaks observed on the spectrum.

Figure 4 represents the evolution of the crystallization coefficients of the two main planes (002) and (101) as a function of the percentage of doping of Ni and Mg in the starting solution. It can be observed that the coefficient of texture is maximum for both planes (002) and (101) with a doping level equal to 3% of Mg (2% Ni is fixed). This reflects the high crystallization degree of these two crystallographic planes.

Figure 5 shows the variation of the half-height width (FWHM) as a function of the Mg doping concentration of the diffraction line (101). This figure shows that the width at half height has a minimum value for the two doping rate values at Mg 0% and 3%. The fineness of the X-ray diffraction line is the consequence of an improvement of crystallinity. This will result in the formation of crystallites with a large size in the layers \( \text{Cd}_{0.98}\text{Ni}_{0.02}\text{S} \) and \( \text{Cd}_{0.98}\text{Ni}_{0.02}\text{Mg}_{0.01}\text{S} \).
Figure 4. $T_C(101)$ and $T_C(002)$ plotted against the $[\text{Mg}] / [\text{Cd}]$ ratio (Ni fixed at 2 at%).

Figure 5. Variation of the width at half height according to the rate of doping.

The average size of Mg and Ni co-doped CdS crystallites ($D_{hkl}$) is determined from the X-ray diffraction patterns using Scherrer’s formula [30].

$$D_{hkl} = \frac{\lambda}{\beta_{hkl} \cos \theta_{hkl}}$$  \hspace{1cm} (5)

With $\lambda$ the wavelength of the X-ray ($\lambda = 1.5406$ Å), $\beta_{hkl}$ the width at half-height and $\theta_{hkl}$ the diffraction angle of Bragg.

Figure 6 shows the variation of the crystallite size as a function of the $[\text{Mg}] / [\text{Cd}]$ ratio in the solution, with the $[\text{Ni}] / [\text{Cd}]$ ratio set at 2%. We notice that its average value takes a maximum value for both the samples $\text{Cd}_{0.98}\text{Ni}_{0.02}\text{S}$ and $\text{Cd}_{0.995}\text{Ni}_{0.02}\text{Mg}_{0.005}\text{S}$.

Figure 5. Average crystallites size of Mg and Ni co-doped CdS.
2.2 Surface morphology

Plan view SEM images of all the samples are presented Figure 7(a)-(c) (with magnification of x40000). Figure 7(a) display the morphology of Cd$_{0.98}$Ni$_{0.02}$S thin films the size of the grains is not homogenously distributed over the surface, Figure 7(b) show the presence of homogeneously distributed grains having a relatively smooth surface, indicating that the films are homogeneous and show good substrate adhesion. As the Mg doping take the value 3 % in the spray solution, the average grains size increases which is in agreement with X-ray diffraction (XRD) results.

![Figure 3. SEM plan views of (a) Cd$_{0.96}$Ni$_{0.04}$S, (b) Cd$_{0.95}$Ni$_{0.02}$Mg$_{0.03}$S, (c) Cd$_{0.91}$Ni$_{0.02}$Mg$_{0.07}$S](image)

2.3 Composition analysis

The composition analysis of all films was made by energy dispersive X-ray spectroscopy. Figure 8 presents the EDAX spectra of Mg and Ni co-doped CdS films. EDAX spectra reveal that deposited CdS layers contain cadmium elements and sulfur, with low percentages of nickel and magnesium; the existence
of the chlorine (Cl) as a source of Cd (CdCl₂), and the carbon (C) is perhaps belongs to the source of sulfur CS (NH₂). When the Mg doping rate exceeds 7%, the oxygen appears with a small amount, which explains the formation of the CdO phase in the deposited films. Other elements such as the Si detected in our layers probably come from the glass substrate. The distribution of Cd, S, Ni and Mg elements on the surface is studied by EDAX mapping and the results are given in Figure 8 (a) and the individual elemental maps of Cd, S, Ni and Mg are also shown in Figure 8 (b, c, d and e) showing the presence and homogeneous distribution of the elements mentioned on the surface.

Figure 8. EDS spectrum of Cd0.95Ni0.02Mg0.03S thin films.
2.4 Optical properties

Optical properties such as transmission, absorption coefficient and band gap energy of Mg and Ni co-doped CdS thin films are determined from the variation in optical transmission as a function of the wavelength ($\lambda$) in the range of 300 to 1000 nm. The results show that CdS cadmium sulphide thin films have good transmittance in the visible region of solar spectrum, which allows them to be used as transparent windows or buffer layers in solar cells. Optical transmission curves of the thin films of $\text{Cd}_{0.98}\text{Ni}_{0.02}\text{S}$, $\text{Cd}_{0.97}\text{Ni}_{0.02}\text{Mg}_{0.01}\text{S}$, $\text{Cd}_{0.95}\text{Ni}_{0.02}\text{Mg}_{0.03}\text{S}$, $\text{Cd}_{0.93}\text{Ni}_{0.02}\text{Mg}_{0.05}\text{S}$ and $\text{Cd}_{0.91}\text{Ni}_{0.02}\text{Mg}_{0.07}\text{S}$ are shown in Figure 9.

The CdS sample doped with 2% Ni shows practically 65% of transparency in the visible region, after the doping of 1% with Mg, the transmittance of thin films $\text{Cd}_{0.98}\text{Ni}_{0.02}\text{S}$ decreases until at the wavelength of 700 nm and then increases to reach a transmission value of about 75%. When thin layers $\text{Cd}_{0.98}\text{Ni}_{0.02}\text{S}$ doped with 3, 5 and 7 at % of Mg, the transmission decreases remarkably up to a value of 50% for the ratio of concentration [Mg] / [Cd] equal to 7%. Films co-doped with nickel and magnesium show a slight decrease in transmission compared to undoped films in the visible and near infra-red regions. This can be explained by the increase in the optical absorption of the charge carriers, following an increase in their density by magnesium doping effect. The same behavior has been reported by T. Sivaraman et al. [31] for thin layers of CdS doped with Mg.

The optical band gap of (Ni, Mg) co-doped CdS has been evaluated using the Tauc’s relation [32]:

![Figure 9](image-url)
\[ (\alpha h \nu) = A(h \nu - E_g)^2 \] (6)

\( \alpha \) is calculated initially by the relation:

\[ \alpha = \frac{\ln (T^{-1})}{d} \] (7)

Where \( T \) is the optical transmittance, \( \alpha \) is the absorption coefficient, \( A \) is a constant and \( h \nu \) is the photon energy and \( d \) is the film thickness.

As for pure cadmium sulphide, the optical gap value of Mg and Ni co-doped CdS thin layers is estimated by extrapolation of the straight line of the curve that gives \((\alpha h \nu)^2\) as a function of photon energy \((h \nu)\), shown in the Figure 10. The optical gap values of the nickel and magnesium doped thin films found by extrapolation of the absorption coefficient curves as a function of energy are given in Table 3. We have found that the \( E_g \) values varied from 2.460 to 2.398 eV, we can explain it as follows: the nickel and magnesium atoms are donor atoms, so incorporation of Mg\(^{2+}\) ions create degenerate donor levels in the CdS conduction band, leading to the extension of the conduction band in the band gap region. This results in the reduction of the band gap of the co-doped layers. This is consistent with the results previously obtained by T. Sivaraman et al. [31] for the Mg doped CdS thin films, and C. Lokhande et al. [33] for Al doped CdS thin films.

**Table 2:** Variation in the value of the optical gap as a function of doping.

| Sample                  | \( E_g \) (eV) |
|-------------------------|----------------|
| \( Cd_{0.98}\text{Ni}_{0.02}\text{S} \) | 2.460          |
| \( Cd_{0.97}\text{Ni}_{0.02}\text{Mg}_{0.01}\text{S} \) | 2.455          |
| \( Cd_{0.95}\text{Ni}_{0.02}\text{Mg}_{0.03}\text{S} \) | 2.440          |
| \( Cd_{0.93}\text{Ni}_{0.02}\text{Mg}_{0.05}\text{S} \) | 2.440          |
| \( Cd_{0.91}\text{Ni}_{0.02}\text{Mg}_{0.07}\text{S} \) | 2.398          |

**Figure 4.** Transmission plots of \( Cd_{0.98}\text{Ni}_{0.02}\text{S} \), \( Cd_{0.97}\text{Ni}_{0.02}\text{Mg}_{0.01}\text{S} \), \( Cd_{0.95}\text{Ni}_{0.02}\text{Mg}_{0.03}\text{S} \), \( Cd_{0.93}\text{Ni}_{0.02}\text{Mg}_{0.05}\text{S} \) and \( Cd_{0.91}\text{Ni}_{0.02}\text{Mg}_{0.07}\text{S} \) thin films.

**Figure 11.** Tauc’s plot results of \( Cd_{0.98}\text{Ni}_{0.02}\text{S} \), \( Cd_{0.97}\text{Ni}_{0.02}\text{Mg}_{0.01}\text{S} \), \( Cd_{0.95}\text{Ni}_{0.02}\text{Mg}_{0.03}\text{S} \), \( Cd_{0.93}\text{Ni}_{0.02}\text{Mg}_{0.05}\text{S} \) and \( Cd_{0.91}\text{Ni}_{0.02}\text{Mg}_{0.07}\text{S} \) thin films.
3. Conclusions

Mg doped (Cd$_{0.98}$Ni$_{0.02}$S) films with different Mg concentrations (0, 1, 2, 4 and 7%) were synthesized by a simple Spray Pyrolysis technique. The effect of Mg doping on the structural and optical properties of Cd$_{0.98}$Ni$_{0.02}$S films were studied. The structural studies revealed that all the films exhibit hexagonal structure (wurtzite) with preferential orientation along the (101) from 3 % up to 7 % Mg-doping. The SEM analysis shows that the Cd$_{0.95}$Ni$_{0.02}$Mg$_{0.03}$S sample has a homogeneous morphology with a grain allocation. The optical study showed that all the films were highly transparent. The transmittance in the visible region varies between 50% and 75% depending on the Mg-doping. The gap energy decreases with the Mg doping increase. The good structural and optical properties of (Mg, Ni) co-doped CdS make it a potential candidate for window material in solar cell applications.

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