Structural, Morphological and Opto-Electrical Properties of CdMgs Ternary Thin Films for Photovoltaic Applications

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Abstract

Nanostructured ternary cdmgs thin film with different Mg concentrations (0, 1, 2, 3, 4 wt.%) were prepared by spray pyrolysis technique using a perfume atomizer and their application potential for photovoltaic solar cells have been investigated. XRD studies revealed that the films exhibit wurtzite (hexagonal) structure with a preferential orientation along the (0 0 2) direction. The Mg^{2+} doping markedly influenced the evolution of cds nanostructures, resulting in the formation of nano-sized needles due to the substitution of Cd^{2+} ions by smaller Mg^{2+} ions. An improved optical transmittance property was also achieved by Mg-doping with a blue shift in the optical band gap which is attributed to quantum confinement effect, Moreover, an improvement in electrical conductivity is also observed in the Mg-doped CdS thin films. The improved optical transparency, widened band gap and increased electrical conductivity observed in the Mg-doped CdS thin films suggests that Mg-doping is highly effective for applications as window/buffer layers in future solar cell applications.

Keywords: X-ray diffraction; thin films; crystal structure; optical band gap; electrical properties

Introduction

Cadmium sulfide (CdS) a II-IV metal chalcogenide semiconductor finds application in solar cells [1], optical detectors [2] and opto-electronic devices [3]. The medium band gap (2.42 - 2.62 ev), high absorption coefficient (10^4 - 10^5 cm^{-1}) and high mobility (440 cm^2 $V^{-1}s^{-1}$) make CdS a potential material in building nano-scale electronic and optoelectronic devices [4]. Thin film of n-type CdS are widely used as a window layer in hetero-junction realization of p-type CdTe and $CuInSe_2$ solar cells and it is widely accepted that the quality of the CdS window enhances the efficiency of the CdS/CdTe hetero-structures [5]. However owing to its sulphur deficiency and conductivity value, CdS yields poor photovoltaic performance. It is known that the conduction of pure CdS is entirely dependent on the native defects of sulphur vacancies and cadmium interstitials. Therefore, by controlling those defects it is possible to control the conductivity of pure CdS. Dzhafarov et al. [6] reported that diffusion of indium into the CdS lattice improves its electrical conductivity without changing its structural and optical properties. Improved optical and electrical properties of CdS thin films through zinc doping were reported by Anbarasi et al. [7]. Modification of the structural, optical and electrical characteristics of Ag-doped CdS films through ion exchange process was reported by Gonzalez et al. [8]. Enhancement in the electrical conductivity of CdS thin films through copper doping has been reported by Shah et al. [9]. These results infer that doping is found to be an important parameter to improve the optical and electrical properties of CdS thin films. Mg^{2+} is a transition metal ion which has a standard ionic radius of 0.72 Å, slightly less than that of Cd^{2+} (0.97 Å) and an electro negativity of 1.31 Pauling lower than that of Cd^{2+} (1.61 Pauling). So it is supposed that Mg^{2+} ions can substitute Cd^{2+} ions in the crystalline structure of CdS and may improve its physical properties. Several chemical and physical methods have been used to elaborate thin films of undoped and doped CdS, such as spray pyrolysis [10], chemical bath deposition [11], RF sputtering [12] and electron

beam evaporation [13]. Among the chemical methods, spray pyrolysis is very attractive due to its feasibility to produce large area thin films at low cost. In this work, Mg doped CdS thin films were fabricated with low concentration of Mg (0, 1, 2, 3 and 4 wt.%) by spray pyrolysis technique using perfume atomizer and the effect of Mg concentration on the structural, morphological, optical and electrical properties of the CdS thin films was investigated and the results are reported. The use of perfume atomizer has the following advantages over the conventional spray method such as: no need for carrier gas, fine atomization and enhanced wettability between sprayed microparticles and the previously deposited layers.

Experimental details

CdMgS thin films were prepared by spray pyrolysis technique using perfume atomizer. Aqueous solution (50 ml in volume) containing 0.1 M of cadmium chloride ($CdCl_2.H_2O$) and 0.1 M of thiourea ($CS(NH_2)_2$) were used as the precursor for cds thin films. Magnesium chloride ($MgCl_2.H_2O$) with different concentrations (0, 1, 2, 3 and 4 wt.%) was added to the spraying solution for Mg doping. Micro glass slides with dimensions 76 mm x 25 mm x 1.4 mm were used as substrates. The substrate temperature was maintained at 400°C. The samples were characterized using a XPERT proanalytical X-ray diffractometer with cukα radiation (λ = 1.5406 Å). Thickness of the samples was measured by gravimetric method and the thickness of the films coated with 0, 1, 2, 3 and 4 wt.% Mg concentrations were found to be equal to 418, 431, 426, 414 and 422 nm respectively. Surface morphological studies were done by HITACHI S-3000H scanning electron microscope. Electrical studies were done by using two-probe method and the optical absorption studies by SHIMADZU UV-1700 double beam spectrophotometer in the wavelength range 300 - 1100 nm. PL studies were made using Varian Cary Eclipse Fluorescence spectrophotometer.

Results and discussion
Crystallographic analysis

To obtain the information about various crystallographic aspects, XRD studies have been carried out on the CdMgS films. XRD traces of all the samples (Fig. 1) found to show almost similar trends. The XRD pattern is compared with the standard JCPDS data (Card No. 41–1049) for indexing the peaks. It can be observed that the films are polycrystalline in nature with hexagonal structure. The preferred orientation of both the undoped and doped cds films is mainly along the (0 0 2) direction. It is observed that the intensity of the (0 0 2) peak decreased with the increase in the Mg doping concentration in the films. No diffraction peaks for any Mg compounds (sulphides, oxides, etc.) Or other impurity phases were detected in the doped samples. These results suggest that Mg merely diffused into cds and did not form any chemical reaction with sulphur to form new compounds. Except for the film coated with 1 wt.%, the 2θ value of the (0 0 2) peak (Table 1) shifts towards higher angle side with increase in Mg incorporation which means a contraction in lattice volume, and this might be due to the replacement of Cd^{2+} ions (0.97 Å) with smaller Mg^{2+} ions (0.72 Å) in the lattice. The lattice parameters 'a' and 'c' were calculated from the position of the XRD (0 0 2) peak and the values are compiled in Table 1.

The small reduction of the lattice parameters of CdMgS films can be consequence of the incorporation of Mg into the CdS lattice. This supposition can happen if the magnesium ions enter substitutionally in Cd sites. This is in accordance with the results reported by Dzhafarov et al. [6] for indium doped CdS thin films. The c/a ratio of the CdMgS films remains almost constant inferring that Mg doping does not affect the fundamental CdS crystal structure.
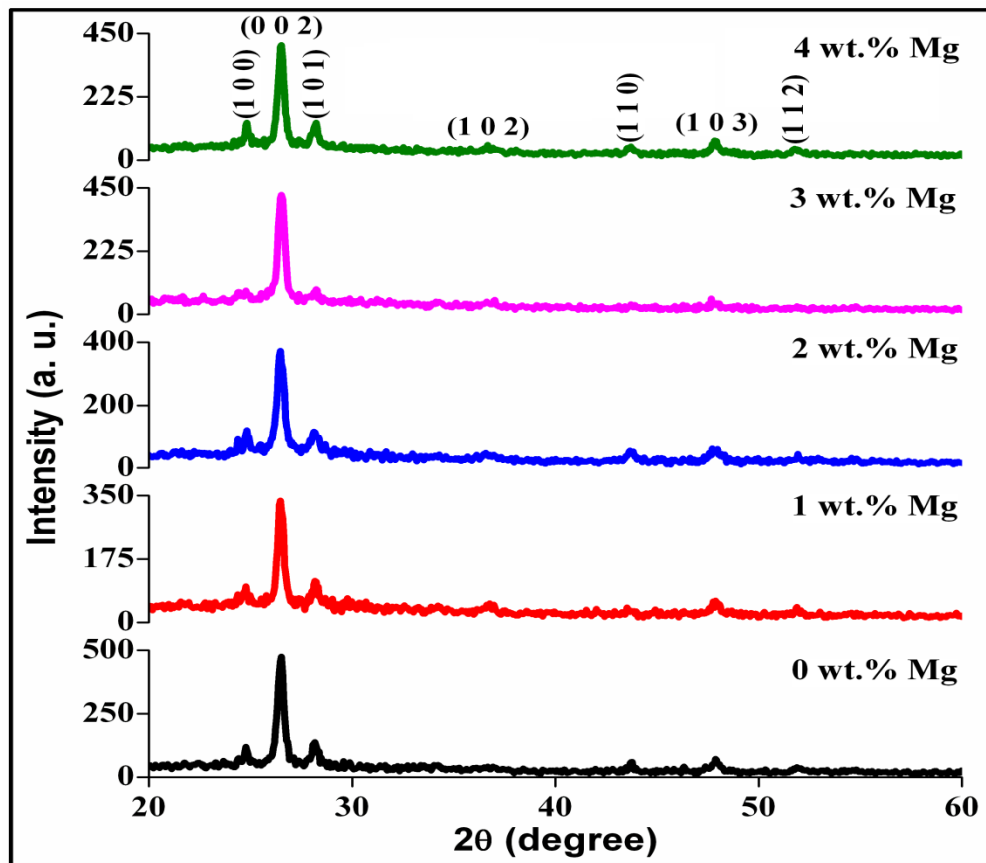
Fig. 1   XRD patterns of CdMgS thin films

The mean crystalline size (D) was calculated by Debye-Scherrer formula [14]:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \qquad (1)$$

where λ is the wave length of the X-ray used (1.5406 Å), β is the full width at half-maximum (FWHM) in radians and θ is the angle of diffraction. The calculated crystallite size values are given in Table 1. It is observed that except for the film coated with 1 wt.% Mg concentration, the crystallite size of all the other doped films show a decreasing trend. Microstrain is calculated by the formula $\varepsilon = \frac{\beta \cos\theta}{4}$ as reported by Balu et al. [15] and the calculated values are compiled in Table 1. It is observed that except for the film coated with 1 wt.% Mg concentration, the mean micro strain is enhanced with Mg concentration. Because of lattice contraction, strain is increased and it results in decrease in the crystalline size values of the doped films.

Table 1 Structural parameters of CdMgS thin films

Mg concentration (wt.%)	$2\theta_{(0\,0\,2)}$	$d_{(0\,0\,2)}$ (Å)	Crystallite size, D (nm)	Strain, $\varepsilon \times 10^{-3}$	Lattice parameters		
					a (Å)	c (Å)	c/a
0	26.462°	3.3655	24.35	1.486	4.1219	6.7311	1.6330
1	26.461°	3.3656	25.83	1.401	4.1220	6.7312	1.6330
2	26.464°	3.3654	23.68	1.528	4.1217	6.7307	1.6330
3	26.467°	3.3652	22.43	1.613	4.1215	6.7304	1.6330
4	26.469°	3.3649	23.04	1.571	4.1211	6.7298	1.6330

SEM and elemental analysis

The SEM micrographs of the undoped and doped CdS thin films are shown in Fig. 2 (a-e).
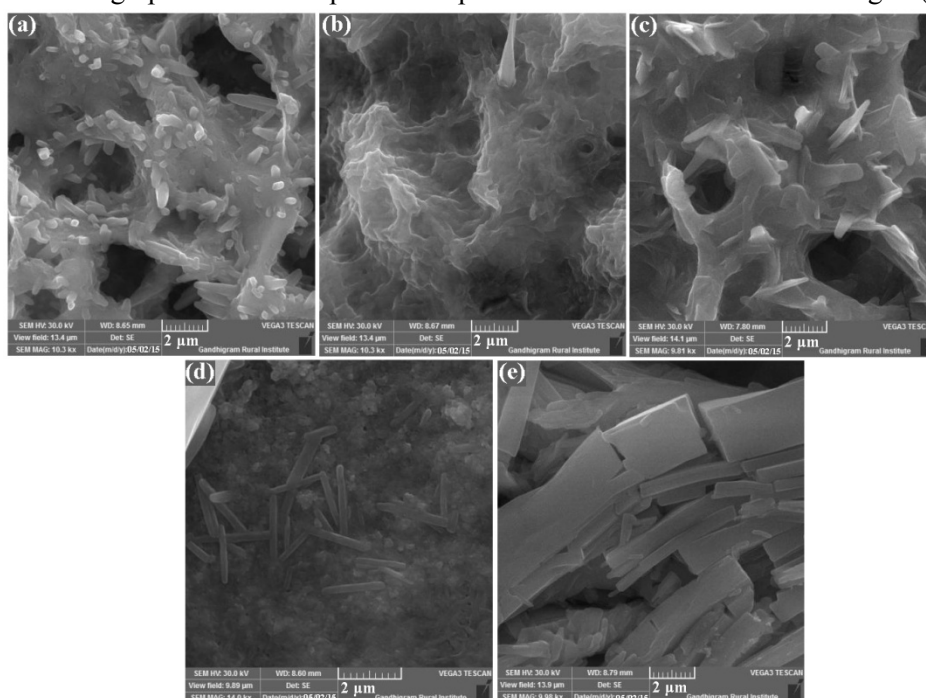

Fig. 2   SEM images of a) 0 wt.%, b) 1 wt.%, c) 2 wt.%, d) 3 wt.% and e) 4 w.% Mg doped CdS thin films

The SEM micrograph of the undoped CdS thin film (Fig. 2 (a)) reveal island like surface embedded with few nano sized grains. With Mg doping, no island surface is visible and no nanosized grains are evitable for the film coated with 1 wt.% Mg concentration (Fig. 2 (b)). As the doping concentration is increased to 2 wt.%, nano needles appeared to emerge throughout the film surface (Fig. 2(c)). For 3 wt.% Mg doping, the film surface is covered fully with equally sized nanograins (Fig. 2(d)). No creaks or holes are visible in the surface confirming the improved crystallinity of this film. As the doping concentration is increased to 4 wt.% Mg concentration, the surface is fully composed of well grown nano needles with different sizes and shapes (Fig. 2(e)). These results infer that the surface of CdS thin film modifies from island growth to nano needle like structures with Mg doping.

Chemical composition of CdS and doped CdS thin films were analysed by energy dispersive X-ray analysis (EDAX). Fig. 3(a, b) shows the EDAX spectra of as deposited CdS and CdS film coated with 3 wt.% Mg concentration respectively.
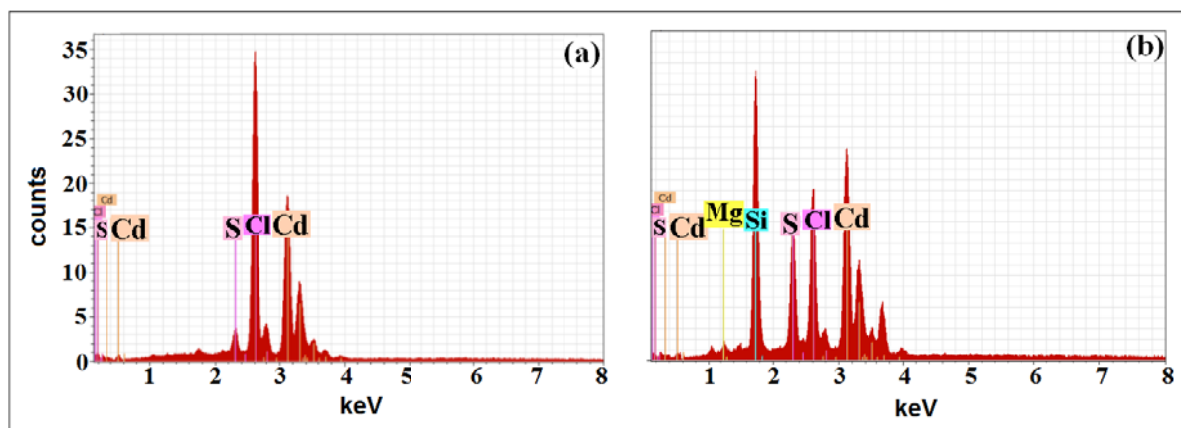

Fig 3   EDX spectra of a) Undoped and b) CdMgS thin film coated with 3 wt.% Mg concentration

The spectra confirm the presence of cadmium and sulfur in both the samples. The presence of Mg in the doped sample is evinced from Fig. 3(b). Impurity element Si detected by EDAX is attributed to the chemical component of glass substrate. Comparison of the EDAX data on the uncoated glass substrate and on the doped films confirms the inclusion of alkali dopants in the doped films. The composition of the elements present in the films is compiled in Table 2.

Table 2 Elemental composition of CdMgS thin films

Mg doping concentration (wt.%)	Elemental composition (at.%)			
	Cd	S	Mg	S/Cd
0	48.46	47.54	---	0.98
1	49.9	48.12	0.98	0.96
2	51.21	46.13	1.34	0.90
3	52.01	45.61	1.62	0.87
4	51.87	44.77	1.96	0.86

Optical studies

The optical properties such as transmittance, absorption coefficient and band gap energy of CdMgS thin films are of great important to identify their suitability as window layer for solar cell applications. Fig. 4 shows plots of the optical transmittance spectra of the CdS films deposited with different Mg concentrations while the inset Fig. 4 shows the absorbance spectra of the films. As it can be seen, the Mg-doped CdS films are found relatively to be highly transparent of about 83% in the visible and near infra-red regions which makes them suitable as transparent window in solar cells. A blue shift in the absorption edge towards higher wavelength side is noticed for the CdMgS film coated with 1 wt.% Mg concentration, whereas a red shift in the absorption edge is noticed for the other doped samples.

The absorption coefficients for all the films are in the order of 10^4 cm^{-1}. It is observed that the absorption edge of the films shift towards shorter wavelength with increasing Mg concentration an effect that is representative of a quantum confinement. All the films show low absorption in the longer

wavelength region. Using transmittance data, the absorption coefficient α was calculated and was then used to determine the band gap of the films as shown in Fig. 5.
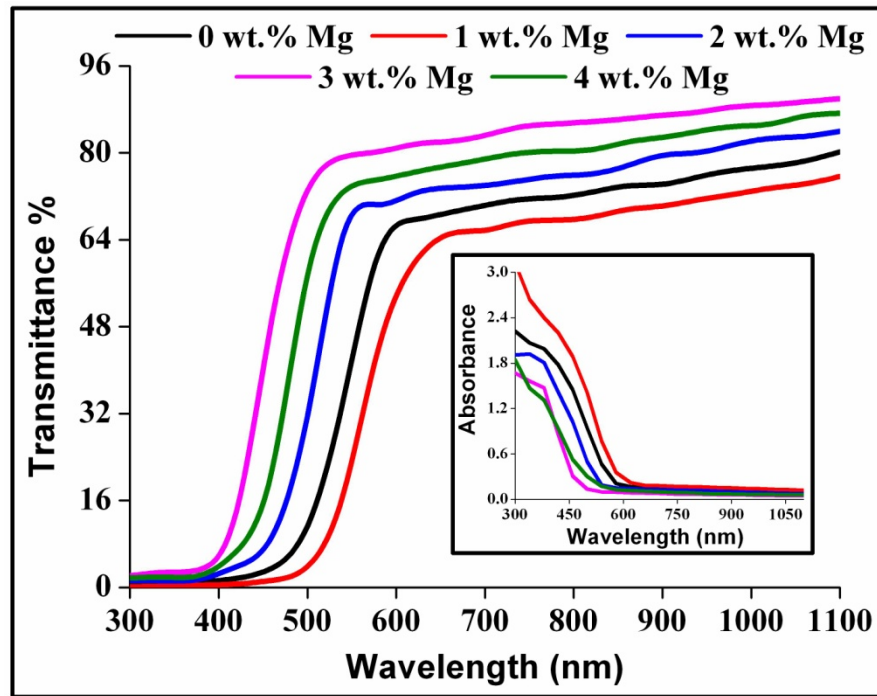

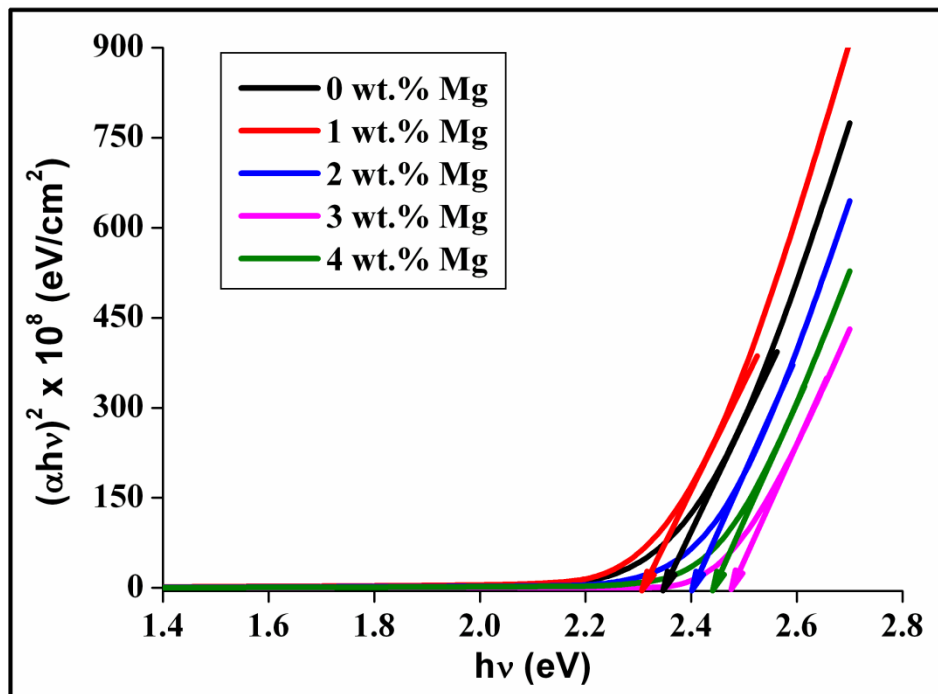Fig. 4 Transmittance spectra. Inset Fig. Absorbance spectra of CdMgS thin films

Fig. 5   Plots of $(\alpha h\nu)^2$ vs. hν of CdMgS thin films

It is observed that the undoped film has a band gap of 2.35 eV which agrees well with the band gap value obtained by Nagarethinam et al. [16] for CdS thin films fabricated by an automated trigger

enhanced spray technique. The band gap values for the CdS films coated with 1, 2, 3 and 4 wt.% Mg concentrations were found to be equal to 2.31, 2.4, 2.48 and 2.44 eV respectively. Except for the film coated with 1 wt.% Mg concentration, the optical band gap of the other doped films was blue shifted with Mg concentration. A similar blue shift phenomenon of E_g was also reported by Rajashree et al. [17] for Cd-doped PbS thin films prepared by spray pyrolysis technique. Thus, the observed modification in the E_g confirms the formation of ternary CdMgS alloy and the existence of strong quantum confinement in this system. Owing to quantum confinement effect, the band gap increases due to reduced crystallite size, which result in the shift of absorption threshold to shorter wavelength due to individual confinement of electrons and holes. It may be denoted that the optical band gap of CdMgS thin film with 0 and 1 wt.% Mg concentrations is found to be lower compared to the bulk value (2.42 eV) which may be related to microstructure imperfection, surface and lattice defects of the CdS thin films [18].

Optical constants (i.e. refractive index and extinction coefficient) are the parameters which characterize how a material responds to an electromagnetic field excitation at a given frequency. The influence of Mg doping concentration on the refractive index and extinction coefficient of CdS thin films are shown in Fig. 6 and 7 respectively.
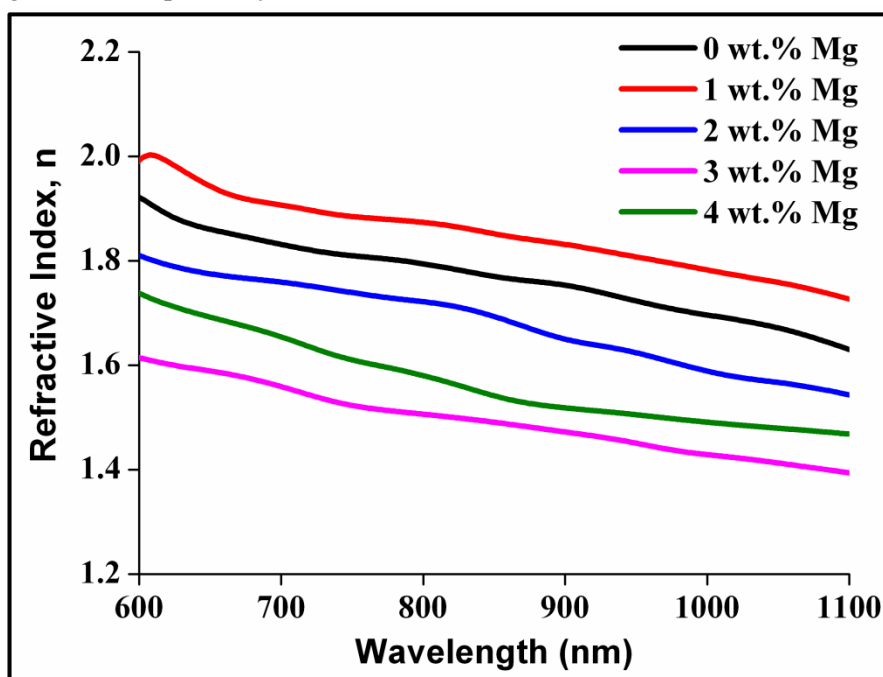

Fig. 6   Variation of refractive index with wavelength of CdMgS thin films

The average refractive index values of the CdMgS films with 0, 1, 2, 3 and 4 wt.% Mg concentrations were found to be equal to 1.67, 1.72, 1.56, 1.40 and 1.46 respectively. It is observed that except for the CdMgS film coated with 1 wt.% Mg concentration, the n values for the doped samples decreases as the doping concentration increases. The variation of the refractive index is an indicative of some interactions between photon and electrons in the material. Fig. 7 shows the dependence of extinction coefficient of the CdMgS films on wavelength.

The extinction coefficient of the Mg-doped CdS films is found to vary between 23×10^{-3} to 78×10^{-3} and is fairly constant in the long wavelengths. Extinction coefficient reflects the absorption of electromagnetic waves in semiconductors due to inelastic scattering events. The average extinction coefficient values for the CdMgS films with 0, 1, 2, 3 and 4 wt.% Mg concentrations were found to be

equal to 46.24, 56.66, 36, 22.34 and 28.71 x 10^{-3} respectively. It is well known that the refractive index and extinction coefficient of the films vary inversely with transmission. The high (low) transmittance spectra have low (high) optical constants.
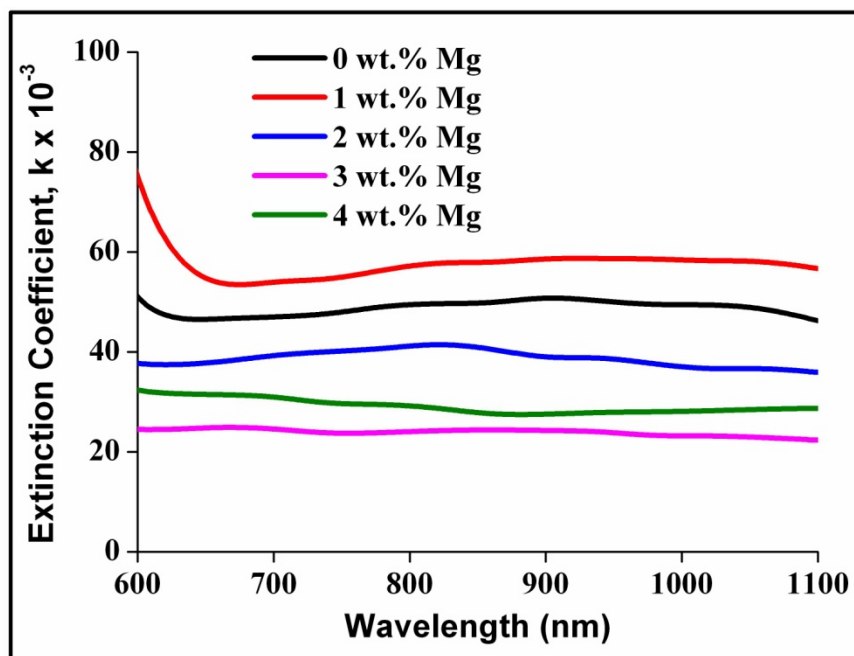

Fig. 7   Variation of extinction coefficient with wavelength of CdMgS thin films

Electrical studies

The resistivity (ρ) of the CdMgS thin films was calculated by the formula: $\rho = \left(\dfrac{\pi}{\ln 2}\right) R_s \cdot t$

where t is the film thickness and R_s is the sheet resistance. Fig. 8 shows the variation of electrical resistivity of the CdMgS films as a function of Mg doping concentration.

The undoped CdS film has a resistivity of 2.67 x 10^1 ohm-m which exactly matches with the value reported by Kim et al. [19]. The resistivity of the CdS thin films should not exceed 10^3 order ohm-cm to be used as window layer for heterojuction solar cells [20]. The resistivity values obtained for the CdMgS thin films are well within this range and hence these films can be used as window material for solar cell applications. Except for the film coated with 1 wt.% Mg concentration, all the doped films have low resistivity compared to that of the undoped samples. The reduction in the resistivity of the doped films is expected due to substitutional incorporation of Mg^{2+} ions in the CdS structure. This reduction of the resistivity can be attributed to the rise of carriers due to sulfur deficiencies [21], which agrees with the compositional analysis. The decrease in resistivity might also be due to the decrease of residual defects.

PL Studies

Photoluminescence (PL) is a process in which an electron, excited by monochromatic photon beam of certain energy undergoes radiative recombination either at valence band or at traps/surface states within the forbidden gap [22]. The room temperature PL spectra of CdMgS thin films with 0, 3, 4 wt.% Mg concentrations excited at λ = 230 nm are shown in Fig. 9.
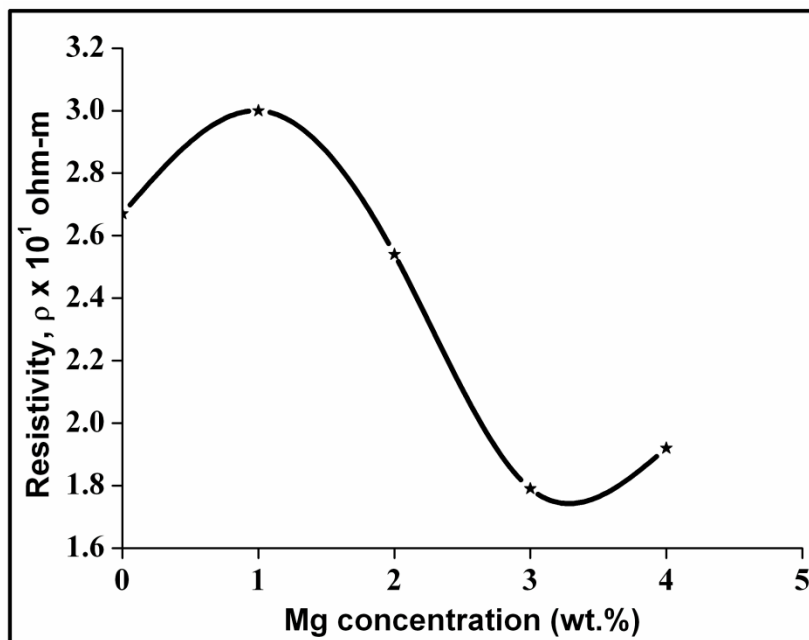
Fig. 8   Variation of electricity resistivity as a function of Mg doping concentration
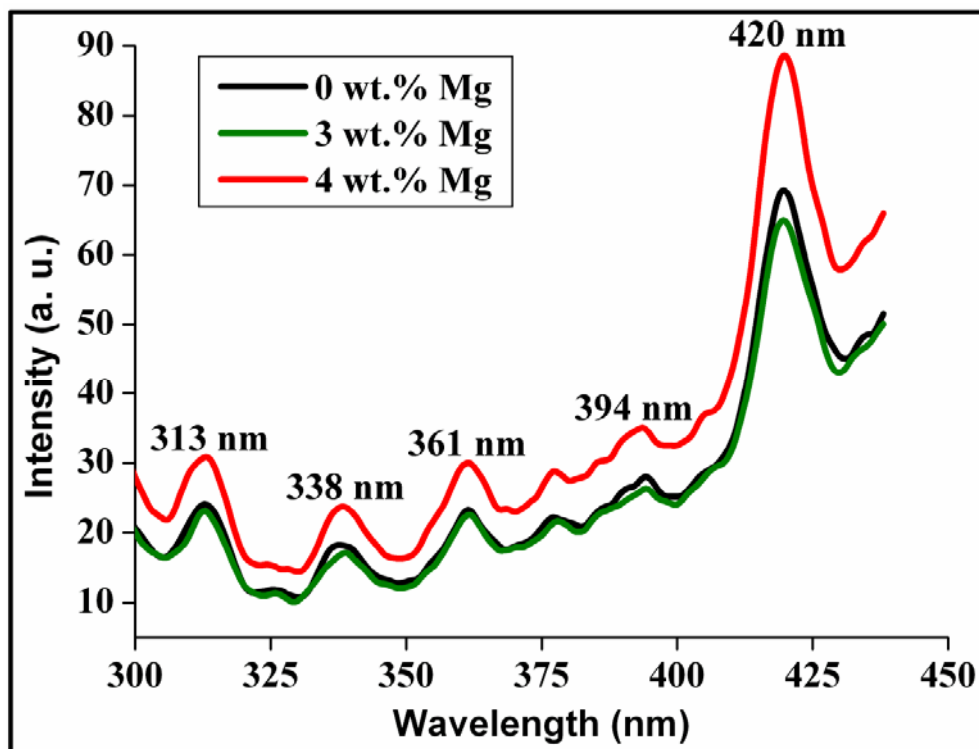

Fig. 9   PL spectra of CdMgS thin films

For 3 wt.% Mg doped CdS, there is a decrease in PL intensity as compared to that of the undoped film whereas for the CdS film coated with 4 wt.% Mg concentration, PL intensity is found to be increased. Emission peaks observed at 313 nm and 338 nm may be attributed to the transitions due to excitons at higher energy levels. S vacancy states are most likely responsible for the PL peak observed at

361 nm [23]. The S vacancies act as deep level trap for electrons and they can exothermically extract electrons from the valence band [24]. The emission peak at 394 nm (3.15 eV) is a high energy band known as the violet band. The peak located at 420 nm is due to an exciton bound to a donor level.

FTIR analysis

The FTIR spectra of undoped CdS and CdMgS thin film coated with 4 wt.% Mg concentration are shown in Fig. 10.
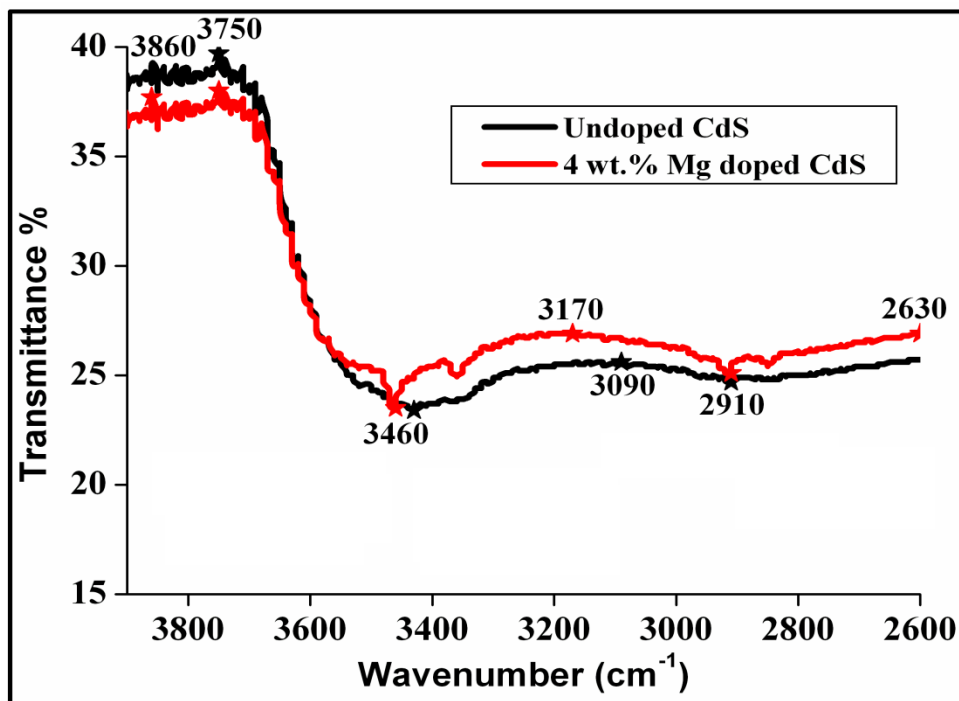

Fig. 10 FTIR spectra of CdMgS thin films

No significant differences between the spectra corresponding to both undoped and doped films are observed. The mid-infrared spectrum (4000 – 400 cm^{-1}) can be approximately divided into four regions and the nature of a group frequency may generally be determined by the region in which it is located. The regions are generalized as follows: the X–H stretching region (4000 – 2500 cm^{-1}), the triple-bond region (2500 – 2000 cm^{-1}), the double bond region (2000 – 1500 cm^{-1}) and the fingerprint region (1500 – 600 cm^{-1}). The fundamental vibrations in the 4000 – 2500 cm^{-1} region are generally due to O–H, C–H and N–H stretching. The presence of broad band range of 3000 – 3600 cm^{-1} is attributed to hydrated water and the hydroxyl group in the spectra results from the hygroscopic nature of CdS [25]. The peak of weak intensity at 2910 cm^{-1} is due to C–H stretching vibration [26] and this peak fully correlates with the observed frequency 2926 cm^{-1} which corresponds to the C–H_2 asymmetric stretch [27]. The weak infrared band observed at 2630 cm^{-1} corresponds to S–H stretching involving sulphur.

Conclusion

CdMgS thin films with different Mg concentrations (0, 1, 2, 3 and 4 wt.%) were prepared on glass substrates by spray pyrolysis technique using perfume atomizer. The effect of Mg concentration on the photovoltaic properties of CdS thin films was investigated by studying XRD diffractograms, SEM images, transmittance curves and resistivity measurements. The XRD patterns revealed hexagonal

structure with a (0 0 2) orientation for both undoped and doped samples. XRD measurements did not detect any new peaks due to Mg-doping indicating that incorporation of Mg^{2+} ions does not affect the fundamental crystal structure of CdS. SEM images showed that Mg-doping modified the morphology of the CdS film from island like structure to needle shaped nano structures. Film transparency increased with increase in Mg-doping concentration. Optical band is blue shifted from 2.31 eV to 2.48 eV with increase in Mg concentration. Multiple Cd and S defects were observed by employing photoluminescence method. Resistivity value decreased as Mg doping concentration increased and a minimum resistivity of 1.79×10^{1} ohm-m was observed for the film coated with 3 wt.% Mg dopant. High optical transmittance, widened band gap and low resistivity values observed for the Mg-doped CdS films make them a suitable material as window/buffer layer in solar cell applications.

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