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# Structural Characterization of PbS and Cd-doped PbS Nanocrystalline Thin Film Fabricated By Chemical Bath Deposition Technique

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**ABSTRACT :** The PbS and Cd doped PbS nanocrystalline thin films were fabricated by chemical bath deposition technique on glass substrates at 70°C. The X-ray analysis shows the nanocrystalline nature of PbS and Cd-doped PbS thin films. The crystallite size of pure PbS thin film was found to be 72.52 nm and that of Cd-doped PbS thin film as 27.24 nm. The morphology of the films was examined by Scanning Electron Microscopy (SEM) and it shows the contrasting microstructures.

**KEYWORDS :** Thin Film, Chemical Bath Deposition, X-Ray Diffraction, Scanning Elctron Microscopy

### I. INNTRDUCTION

Lead sulfide (PbS) having narrow bands belongs to IV-VI group f compound semiconductor and they are widely used as infrared sensor due to presence of direct band gap of 0.4eV [1]. They possess a large excitation Bohr radius of PbS (i.e.18 nm) [2]. As a result, a strong quantum confinement of both electrons and holes is observed in nano sized structure and consequently, the value of the energy band gap can be controlled according to the effective mass model with the modification of size of the particle [3]. This semiconductor materials finds wide applications in photography [4], Selective sensors for Pb<sup>2+</sup> [5] and solar radiation absorption [6]. Moreover, PbS has been utilized as diode lasers, humidity, photo resistance, temperature sensors, solar control and decorative coatings [7,8]. However, these properties depend on the condition of crystal growth and the nature of substrates. Because of these reasons, many researchers have been attracted a great interest in the study of this material with different deposition technique. The commonly employed techniques are electro deposition [9,10], spraypyrolysis [11], microwave heating [12,13] and chemical bath deposition [14-18]. The photo conductivity of PbS nanocrystalline thin films has been investigated [19]. The size of the particle of this material decreases when metal ions are added to it, which in turns increases its energy gap. For these reasons, it can be chosen as the excellent candidate for opto-electronic applications in many fields such as IR detectors, photography, solar cells and light emitting [20,21]. The doping of Ni and Cd in PbS thin films produces a significant shift in the forbidden band gap with energy range, 1.4 to 2.4 and 0.15 to 0.5eV [22]. Because of these significant effect in energy band gap due to doping of metal ions, it is used for solar and detector applications. Amongst the various methods for preparation of PbS nanocrystalline thin films, the chemical bath deposition method is a good and convenient one because of its advantages such as low cost, low temperature requirement, the ability to deposit thin films on different types of substrates suitable for large scale deposition areas and controllable properties of thin film through adjustable deposition parameters [23]. In the present investigations also, the chemical bath deposition

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technique is employed because of its advantages for preparation of PbS and Cd doped PbS thin films and the films were characterized by XRD and SEM.

## II. EXPERIMENTAL DETAILS

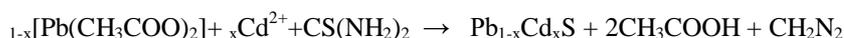
The pure and Cd-doped PbS thin films were deposited on glass substrate by Chemical Bath Deposition Technique. The cleaning of substrate surface has an important role because of its effect on adherent thin film properties in Chemical Bath Deposition method. Before deposition of PbS thin films, the glass substrates were first washed with liquid detergent and rinsed thoroughly in double distilled water and then in boiling water. Finally, they were immersed into acetone for 15 minutes and then dried in an oven.

For deposition of pure PbS thin film, lead acetate was used as source material of lead ion and thiourea as the sulphur ion source. The lead acetate solution of 0.1M was prepared with double distilled water and the same was stirred separately at 50 ° C until a clear solution is obtained. The ammonia solution is added to the solution drop by drop slowly in order to maintain its  $p^H$  at around 10. Then the equimolar solution of Thiourea (0.1M) was added to the solutions. The resulting mixture solution is further stirred slowly at the same temperature until its colour is turned in to dark brown. Using a suitable substrate holder, four ultrasonically cleaned glass substrates were vertically immersed in the solution for 24 h for deposition of PbS thin films at room temperature. After deposition of PbS thin films, the samples were taken out and washed thoroughly in distilled water several times and dried in air and then put in a desiccator.

The reaction mechanism which is responsible for deposition of PbS thin film by CBD method is as follows.



Similarly, the Cd-doped PbS thin films (0.1M) were deposited on the glass substrate by adding an aqueous solution of cadmium chloride (1wt%) to the precursor mixture solution of lead acetate and thiourea maintaining the same  $p^H$  value. The overall chemical reaction taking place is as follows.



## III. RESULTS AND DISCUSSION

### Structural study.

The structural characterization was done by an X-ray diffractometer (Bruker D8 Advance) in the  $2\theta$  range, 20 – 70 using CuK $\alpha$  radiation of wavelength  $\lambda = 1.5406\text{\AA}$ . The XRD patterns of pure PbS (0.1 M) and Cd-doped PbS thin films (0.1M) are shown in figure 1 and 2. The XRD Pattern of PbS has face centered cubic structure as confirmed by JCPDS Data card and the peaks correspond to (111), (200) and (220) corrected crystal planes. The narrow peaks in the XRD Pattern of PbS and CdPbS indicate that the materials have good crystallinity and they are preferentially oriented along the (200) direction. It is also observed that a significant broadening occurs after doping of Cd for (111), (200), (220) orientations. This confirms the presence of a phase transformation due to doping of Cd into PbS lattice [24]. Further, a marginal shift in the diffraction peak is also produced due to the internal lattice strain arising when a large number of Cd<sup>2+</sup> ions occupies PbS<sup>2+</sup> ions in the host lattice. The lattice strain in turn generates structural disorder during crystal growth [25]. The lattice d-spacing is calculated by using Bragg's Law,

$$\lambda = 2d\sin\theta.$$

For (200) orientation, the lattice d-spacing of PbS is found to be 3.06930 Å and that of CdPbS is found to be 2.99946 Å respectively. Thus, there is a decrease in the value of lattice d-spacing due to doping of Cd which is in agreement with the literature in which similar results are reported [26]. Hence, this confirms the formation of CdPbS thin film.

The lattice constant is also calculated by using the relation

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$$a = d (h^2 + k^2 + l^2)^{1/2}$$

The lattice constants are found to be 6.1386 Å for PbS and 5.9989 Å for CdPbS for (200) orientation. The crystallite sizes of PbS and CdPbS are calculated by using Scherrer's formula,

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

where K is a constant (= 0.94),  $\beta$  is the full width at half maximum (FWHM) of the diffraction peak corresponding to a particular crystal plane. The size of pure PbS crystal for (200) orientation is found to be 72.52 nm and that of CdPbS for the same orientation is found to be 27.24 nm. Thus the crystallite size also decreases as the result of doping of Cd which is in line with reports of earlier workers [27].

### SEM analysis

The morphology of undoped PbS and Cd doped PbS (CdPbS) thin films deposited on glass substrate was examined by scanning electron microscopy (SEM). The SEM micrograph image of PbS and that CdPbS nanocrystalline thin films are shown in figures 2 and 3. The SEM image of the undoped PbS nanocrystalline thin film is discontinuous with separated but well developed crystallites. However, the SEM image of PbS doped with 1 wt% of cadmium (CdPbS) nanocrystalline thin film is continuous with uniform crystallite size.

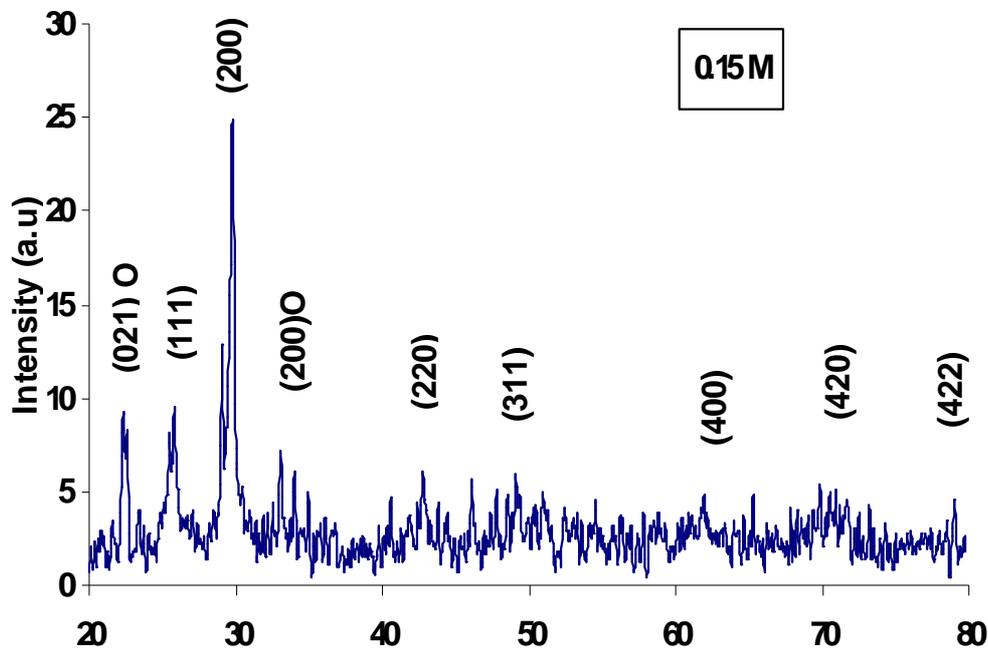


Figure 1. XRD pattern of PbS thin film prepared by CBD technique on glass substrate for 0.10 M.

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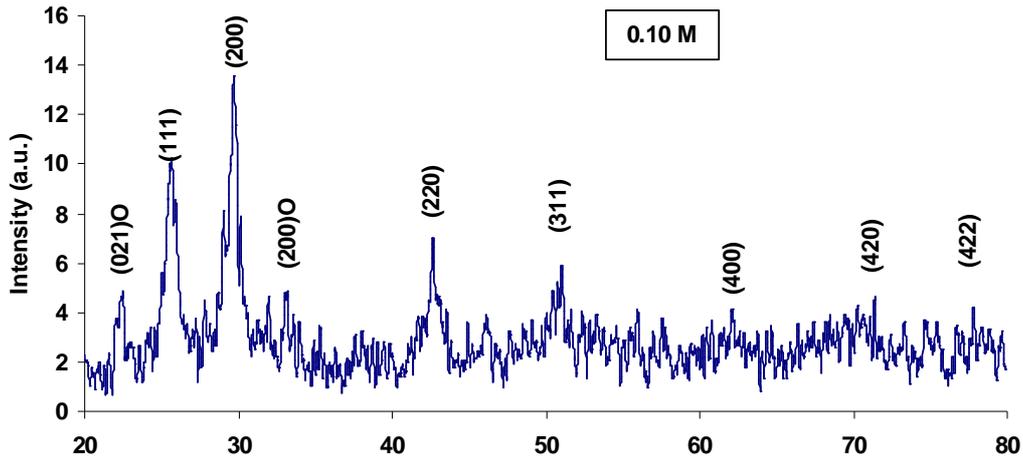


Figure 2. XRD pattern of CdPbS thin film prepared by CBD technique on glass substrate for 0.10M.

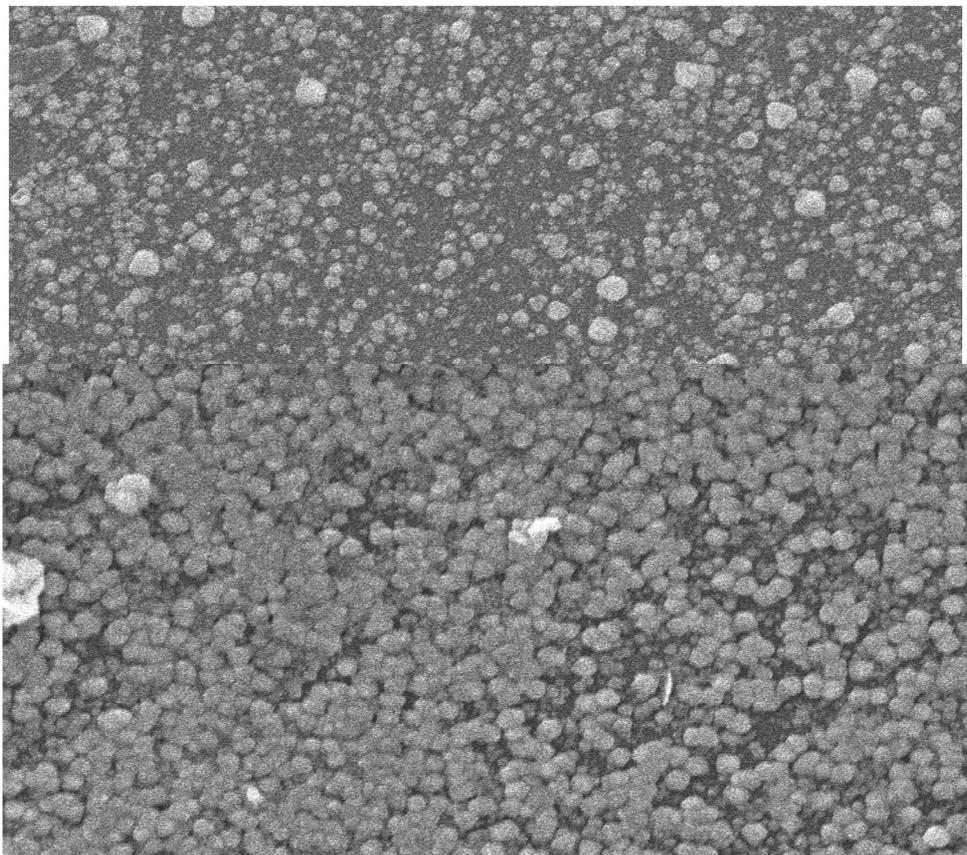


Figure 4. SEM micrograph of CdPbS nanocrystalline thin film (0.1 M)

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## IV. CONCLUSION

In this present study, the pure PbS and Cd doped PbS(CdPbS) nanocrystalline thin films are fabricated on glass substrate by employing the chemical bath deposition technique. From XRD analysis, the crystallite sizes are found as 72.52 nm for PbS and 27.24 nm for CdPbS and we can conclude the formation of PbS and CdPbS thin films with good crystallinity and the great adhesion of pure and Cd doped PbS thin films on glass substrate. The SEM images show a very contrasting microstructures when the pure PbS nanocrystalline thin films are doped with 1 wt% of cadmium.

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