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# STRUCTURAL AND OPTICAL CHARACTERIZATION OF NANOCRYSTALLINE CDS THIN FILMS SYNTHESIZED BY SILAR

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## ABSTRACT

Cadmium sulfide (CdS) is one of the most important II–VI semiconductor materials extensively investigated for optoelectronic devices, such as solar cells, photodetectors, and sensors. It is used particularly as a window or buffer layer for thin film photovoltaic devices due to its direct band gap, absorption in visible radiation and proper electronic properties. Successive ionic layer adsorption and reaction (SILAR) is regarded as one of the simple, low cost, low temperature and scalable chemical methods for preparation of nanocrystalline CdS thin films. The paper discusses the literature-based research of structural and optical characterization of CdS thin films prepared by SILAR technique. There are several techniques for examining structural properties, such as X-ray diffraction, scanning electron microscopy, atomic force microscopy, and energy-dispersive X-ray spectroscopy. Typically, the optical properties are studied by UV–visible spectroscopy and Tauc plot analysis. In reported work, it is found that the CdS films deposited by SILAR are generally polycrystalline, which may be cubic, hexagonal or mixed. The number of cycles, the precursor concentration, the dipping time, the rinsing time, the pH of the solution and the temperature of annealing affect their crystallite size, morphology, thickness, optical absorption and band gap. The CDs films prepared by SILAR process have been concluded as a promising technique in the production of thin films for low cost photovoltaic and optoelectronic applications.

**KEYWORDS:** CdS Thin Films, SILAR, Nanocrystalline Semiconductor, XRD, UV–Vi's Spectroscopy and Optical Band Gap

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## INTRODUCTION

Nanocrystalline semiconductor thin films are highly interesting for research because their structural, optical and electrical characteristics can be modified by different deposition parameters and preparation methods. Cadmium sulfide is a well-studied II–VI compound semiconductor having a direct band gap in the vicinity of the visible region, and high optical absorption. In CdTe and CIGS solar cells, CdS can be used for the window or buffer layer as it can pass a large fraction of the visible light and can enable the formation of a junction with the absorber layers (Yücel et al., 2016). It is also employed in photoconductors, visible-light detectors, gas sensors and photoelectrochemical devices (Mukherjee et al. 2015).

CdS thin films have been synthesized by many techniques such as chemical bath deposition, spray pyrolysis, thermal evaporation, sputtering, electrodeposition, spin coating and SILAR method. Of these, SILAR is especially appealing to academic and low-cost research laboratories as it avoids the need for vacuum systems or costly instrumentation. The technique involves repeated dipping of a substrate in the cationic and anionic precursor solutions and rinses. For CdS, the $Cd^{2+}$ ions are first the adsorbed on the substrate and then reacted with $S^{2-}$ ions to form CdS on the surface (Sankapal et al., 2000).

There are several benefits to SILAR. It enables deposition at room temperature or at low temperature, allows a control of the thickness by number of cycles, helps to prevent homogeneous precipitation, and allows to coat large or irregular surfaces. These benefits make it advantageous for countries where high-cost thin film deposition systems are not as readily available. In the teaching and/or research laboratory of Bangladesh, India and other developing settings, SILAR can be utilized to prepare semiconductor thin films with relatively simple glassware and chemical solutions. Cadmium compounds are however toxic and therefore proper laboratory safety and disposal of waste is essential.

The structure and optical features of the CdS films prepared by SILAR technique are sensitive to the conditions of preparation. The thickness of the film, and the growth of the grains depend on the number of SILAR cycles. Nucleation and compactness are affected by precursor concentration. Time control for dipping and rinsing to adsorb and remove loosely attached species. While annealing can enhance crystallinity, it can also bring about defects or modification of stoichiometry if not optimized (Mukherjee et al., 2015). Hence characterization

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is the requirement to understand if the prepared CdS film is suitable for device applications or not.

## SILAR SYNTHESIS PROCESS

A typical SILAR process involves the dip of a cleaned glass substrate into a solution of cadmium salt (cadmium acetate, cadmium chloride or cadmium nitrate). The $Cd^{2+}$ ions tend to adhere to the surface of the substrate. The rinsing is then made with deionized water to wash out the weakly adsorbed ions in the substrate. The substrate is then dipped into a solution of a sulfide source like sodium sulfide or ammonium sulfide, in which $S^{2-}$ ions react with $Cd^{2+}$ ions which are adsorbed to form CdS. The substrate is washed again to get rid of the excess ions and unwanted particles. This is a complete sequence of one cycle of the SILAR (Sankapal et al., 2000).

**Figure 1. A representative deposition cycle of CdS thin films is shown.**

This illustrates the steps of cleaning the substrate, $Cd^{2+}$ adsorption, rinsing, reacting with $S^{2-}$, rinsing, and forming the CdS film, after repeated cycles.

The formation reaction can be written as Cd2+ + S2- → CdS. The cycle is repeated, which gradually increases the thickness of the film. Note that the method is called “successive” because the cationic and anionic reactions do not occur concurrently in the same solution, but rather sequentially. This helps prevent any unwanted precipitation in the bulk solution and aids in forming the layers in the substrate under control.

It is very important to clean the substrate. Surface contamination, grease and dust decrease film adhesion and uniformity. The standard cleaning procedure for glass substrates is with detergent, distilled water, ethanol/acetone and possibly ultrasonic cleaning. Films can be dried in air and then annealed at desired temperatures after deposition. It is possible to increase crystallinity by letting atoms move to a more ordered structure during the annealing process, but too much heat can lead to sulfur deficiency or surface oxidation (Mukherjee et al., 2015).

## CHARACTERIZATION TECHNIQUES

The main method for structural characterization of CdS thin film is X-ray diffraction. XRD can be used to determine the amorphous/crystalline nature of the film and determine if the film is cubic, hexagonal or both. Peak shape broadening is related to nanoscale crystallite size and the peak positions give the phase information. A Debye–Scherrer equation is often used to calculate the average crystallite size of the material based on the width at half maximum of the diffraction peaks (Cullity and Stock, 2001).

Surface morphology is usually studied using scanning electron microscopy or atomic force microscopy. The information that is given by SEM is related to the shape, compactness, porosity, presence of cracks and agglomeration of the grain. AFM gives surface roughness and topographical information. The elemental composition of the film is analysed by energy-dispersive X-ray spectroscopy, which is used to support the presence of cadmium and sulfur, and to infer whether the film is close to being stoichiometric or not.

A uv–visible absorption spectrometer is typically used for optical characterization. A knowledge of the absorption edge, transparency and optical response of the film can be obtained from the absorbance and transmittance spectra. CdS is a direct band gap semiconductor, and the average value of the optical band gap is typically determined from Tauc plots (Tauc et al., 1966). Plotting a graph of ((\alpha h\nu) ^2) against photon energy (h\nu) and using the linear portion of the curve as an extrapolation to the energy axis, the band gap was calculated for direct transitions.

**The structural properties of SILAR CdS thin films were studied.**

Generally, XRD pattern of the CdS thin films deposited by SILAR technique indicates the nanocrystalline and polycrystalline nature of the films. Depending on the deposition conditions and annealing treatment, CdS can be deposited in cubic zinc blende, hexagonal wurtzite, or mixed phases (Sankapal et al., 2000). The (111) and (220) and (311) planes are the common diffraction peaks that occur in cubic CdS. In hexagonal CdS, peaks may correspond to the (100), (002), and (101) planes. Nanocrystalline thin films exhibit broad diffraction peaks, due to peak broadening caused by small crystallites.

**Figure 2. This is the representative XRD Pattern of Nanocrystalline CdS Thin Film.**

The representative pattern presents large CdS peaks which can be indexed to cubic CdS planes like (111), (220) and (311). Peak broadening is a sign of nanocrystallite formation.

One of the most significant structure parameters is the crystallite size. Smaller crystallites mean that the domains are nanoscales and that they contain more grain boundaries, but they can also lead to a higher surface area. The larger the crystallites the more crystallines present, but if the grain grows too much, the roughness will start to increase. The crystallite size in a SILAR CdS film tends to increase with the increasing number of deposition cycles or with an increase in the annealing temperature; as the cycles of ion adsorption and annealing provide additional heat energy to the film, more grains grow and coalesce, increasing the size of the crystallites (Mukherjee et al., 2015).

Other measures used for assessing the quality of a film are microstrain and dislocation density. Microstrain is associated with lattice distortion from defects, grain boundaries and film-substrate mismatch. Dislocation density is a measure of the number of defects. The lower the strain and the lower the dislocation density, the higher the crystallinity. The annealing can eliminate the structural disorders, but if the annealing is too high, the sulfur vacancies or defects caused by oxidation may exist.

The amount of SILAR cycles has a significant impact on the surface morphology. The film might not be continuous at low cycles due to nuclei separation. As more cycles are repeated, the particles will grow and fuse together, forming a more continuous film. But excessive cycles can result in agglomeration and rough surfaces. For applications such as solar cells, only a small and uniform CdS layer is desirable as the presence of pinholes and rough interfaces will enhance the recombination losses (Yücel et al., 2016).

## SILAR CdS Thin Films Optical Properties

Because of the applications, which are mainly associated with light absorption and transmission, the optical properties of CdS thin films are important. Typical UV–visible absorbance spectra exhibit strong absorbance in the visible region. The absorption edge is typically at wavelengths between 500 and 520 nm, which are the spectral values of the band gap of CdS. Generally, the thicker the film the greater the absorbance will be since more material is available to absorb the light.

**Figure 3. Representative uv-visible Absorbance of silar CdS Thin Film.**
The representative spectra indicates that absorbance increases with the number of SILAR cycles since the thickness of the film increases and the amount of CdS loading increases.

There is also a need for transmittance in PV window layer applications. Ideal CdS window layer should be thin enough to allow the light to reach the absorber layer and thick enough to form a continuous junction. Thick CdS layers can absorb a portion of the short wavelength photons, which can decrease the current of the solar cell. It may contain pinholes if it is too thin that it will lead to instability of the device (Rahman et al., 2020).

The most frequently used method to estimate the optical band gap of CdS thin films is known as the Tauc method. Typical band gap reported in the literature is around 2.3–2.5 eV depending on the crystallite size, thickness, strain and defect concentration (Mukherjee et al., 2015). In very small crystallites, due to quantum confinement, one can expect a higher band gap. A reduced band gap can be observed as crystallite size increases, as thickness of the film increases, or when defect states are created in the vicinity of the band edges.

**Figure 4. Representative Plots of Direct Band Gap CdS.**
The extrapolated linear region shows an illustrative optical band gap of approximately 2.42 eV, in generally similar range of CdS thin films.

The optical response of CdS is closely related to the quality of its structure. A sharp absorption edge occurs in a well-crystallized, compact film. In a disordered film the absorption tails are wider due to the appearance of localized states caused by defects and grain boundaries. Optical and structural properties should always be considered together, therefore.

**Table 1: Summary of Typical Structural and Optical Features of SILAR-Deposited CdS Thin Films**

| Parameter | Typical Observation for SILAR CdS | Characterization Method |
|---|---|---|
| Crystal phase | Cubic, hexagonal, or mixed CdS depending on conditions | XRD |
| Major diffraction peaks | Cubic: (111), (220), (311); Hexagonal: (100), (002), (101) | XRD |
| Crystallite size | Nanocrystalline; often increases with cycles or annealing | XRD/Scherrer analysis |
| Morphology | Granular, compact, or agglomerated depending on deposition cycles | SEM/AFM |
| Composition | Cd and S present; sulfur deficiency may occur | EDS |
| Absorption edge | Usually near visible region around 500–520 nm | UV–Vi's spectroscopy |
| Optical band gap | Usually near 2.3–2.5 eV | Tauc plot |

## APPLICATIONS

The CdS thin films deposited by SILAR method are also very promising in PV and optoelectronic applications. They are most used as a window or buffer layer in thin film solar cells. CdS makes a junction with absorber materials like CdTe and CIGS which facilitate charge separation. SILAR is low cost and thus can be used in research laboratories or pilot scale studies where expensive vacuum deposition systems are not available (Yücel et al., 2016).

Also, CdS thin films can be used in photodetectors due to their visible-light sensitivity. Nanocrystalline CdS has a high surface to volume ratio, which can be beneficial for interactions with light or gases. Hence, CdS can be employed in gas sensors, photoelectrochemical cells, and hybrid nanostructures. A composite of CdS and other materials like ZnO, $TiO_2$ or graphene-based layers can enhance the charge transport and light response.

SILAR's low cost and ease of construction can be useful in a university environment in developing nations, such as Bangladesh, where the development of semiconductors is a significant field of interest. A vacuum chamber is not needed for students to learn how to grow thin film, how to conduct XRD analysis, how to use UV–visible spectroscopy, and how to estimate the band gap. But cadmium toxicity should not be taken lightly. Gloves, use of fume hood, separation of wastes and proper disposal of chemicals are required.

## CHALLENGES AND FUTURE SCOPE

While SILAR is easy to use and effective, it has certain drawbacks. Manual dipping potentially can cause less reproducibility, particularly if the dipping and rinsing time and the concentration of the solution are not carefully controlled. Solution aging and substrate cleaning also can influence the uniformity of a film. Automated SILAR systems will increase cost and improve the reproducibility.

Cadmium toxicity is also a problem. CdS is beneficial but the use of cadmium containing materials demands careful handling. Further studies on cadmium waste reduction, recovering unused solution, and the use of alternative buffer layers that are safer are warranted. Meanwhile, CdS is still relevant due to its continued use in research of thin-film solar cells.

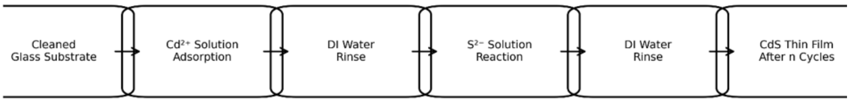
The further research should be carried out to optimize SILAR parameters by systematic experimental design. The influence of the number of cycles, concentration of the precursor, pH, annealing temperature, and substrate type should be examined simultaneously. Further improvements of CdS film performance may be obtained by further doping, composite formation, or heterostructure design. For deeper understanding of the defects, carrier transport and recombination, more advanced characterization like Photoluminescence, Raman spectroscopy, Hall measurement, and impedance spectroscopy can be used.

## CONCLUSION

Nanocrystalline CdS thin films synthesized by SILAR are important materials for photovoltaic and optoelectronic applications. SILAR is a simple, low cost, low temperature and controllable deposition technique. Typically, the structure of CdS films prepared by SILAR is found to be polycrystalline and has cubic, hexagonal, or mixed structure. The crystallite size, strain, shape, and composition are influenced greatly by the deposition cycle, precursor concentration, rinsing, pH, and annealing. Optical characterizations reveal high visible-region absorption, and a direct optical band gap, typically near 2.3-2.5 eV.

Structural properties are intimately tied with optical properties. In general, the higher the crystallinity and more compact the morphology, the more pronounced the absorption edges and the more constant the band gap values will be. While there are problems associated with CdS, such as cadmium toxicity, film reproducibility, and defect control, SILAR continues to be a viable technique for preparation of CdS thin films. Optimization and safety precautions are required to continue the solar cell, photodetector, sensor and semiconductor research applications of SILAR-deposited CdS.

Figure 1. Representative SILAR Deposition Cycle for CdS Thin Films



One SILAR cycle: cation adsorption → rinse → anion reaction → rinse; repeated cycles increase CdS thickness

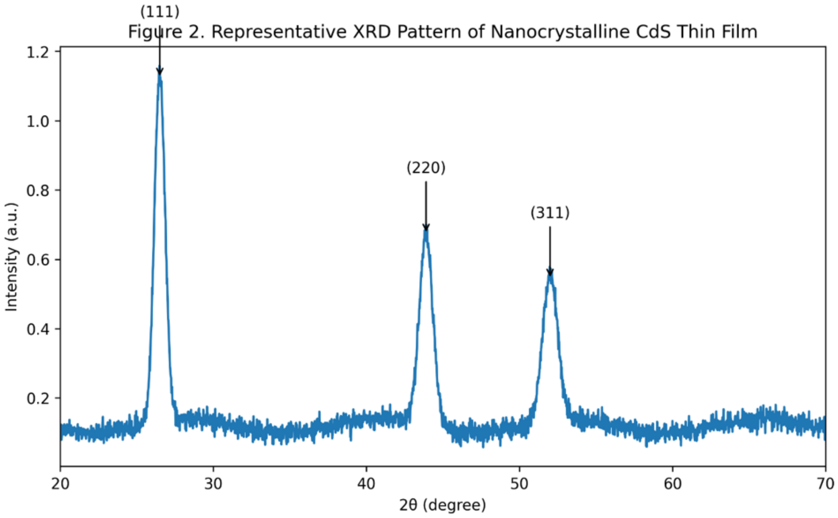


Figure 2. Representative XRD Pattern of Nanocrystalline CdS Thin Film

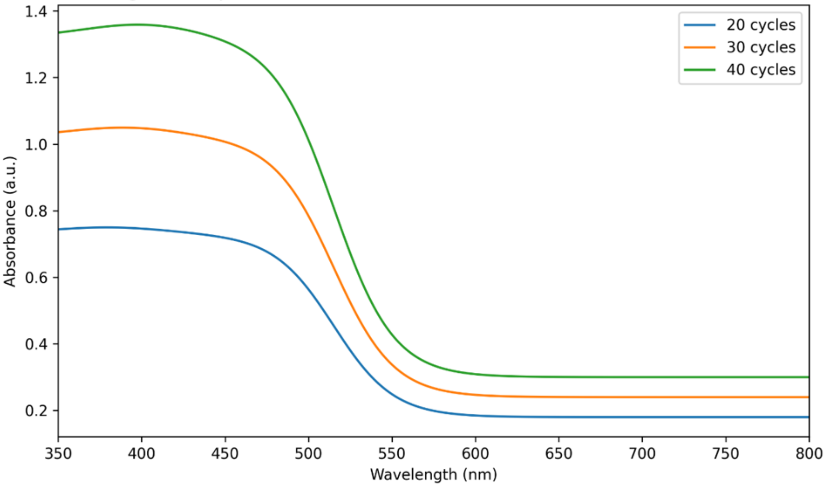
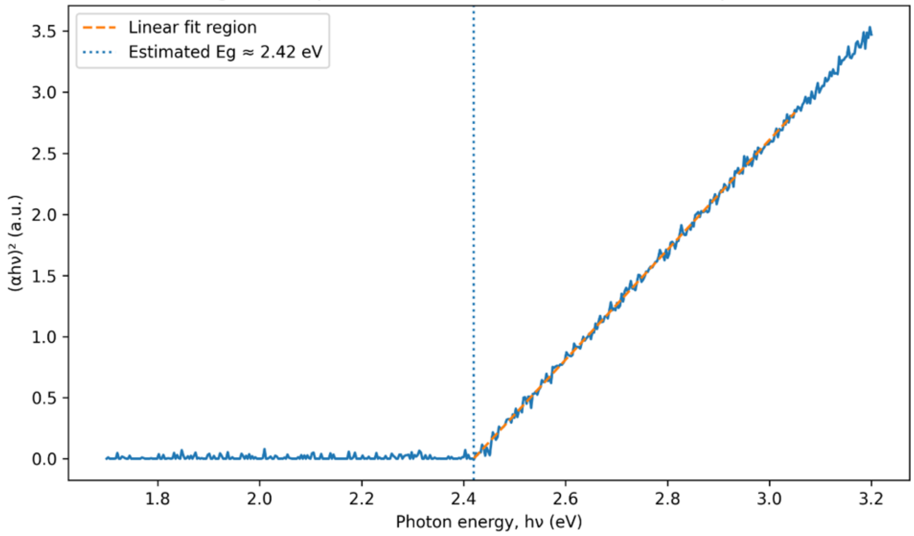


Figure 3. Representative UV–Visible Absorbance of SILAR CdS Thin Films

Figure 4. Representative Tauc Plot for Direct Band Gap CdS



| **Parameter** | **Typical observation for SILAR CdS** | **Relevant characterization** |
|---|---|---|
| Crystal phase | Cubic/hexagonal or mixed CdS depending on conditions | XRD |
| Main XRD peaks | (111), (220), (311) for cubic CdS; (100), (002), (101) for hexagonal CdS | XRD |
| Crystallite size | Generally, nanocrystalline; may increase with cycles/annealing | XRD/Scherrer analysis |
| Morphology | Granular, compact or agglomerated depending on cycles and rinsing | SEM/AFM |
| Optical absorption edge | Visible-region absorption edge near 500â€ “520 nm | UVâ€ “Vis |
| Band gap | Usually close to 2.3â€ “2.5 eV, depending on crystallite size, strain, defects, and thickness | Tauc plot |
| Key controlling factors | Cycle number, precursor concentration, pH, dipping/rinsing time, annealing | XRD, SEM, UVâ€ “Vis |

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