

MINOR REASERCH PROJECT
FINAL REPORT

TITLE

“Study and characteristics of CdSe and CdS binary and mixed ternary compound semiconductors in thin film form prepared by CBD technique.”

Submitted by :-

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LINE OUT OF THE WORK DONE UNDER THE PROJECT

1. Library work

- i) Central library North Maharashtra University, Jalgaon.
 - ii) Central library Pratap College, Amalner.
 - iii) Central library Nanasaheb Y.N. Chavan A.S.C. College, Chalisgaon.
2. Experimental survey of the problem and search of published work on internet.
3. Actual experimental work at Thin Films Laboratory in physics Nanasaheb Y.N. Chavan Arts, Science, Commerce. College, Chalisgaon.

Synthesis of CdSe Thin Film by CBD Technique:-

Number of trails are taken and after many attempts are made to deposite the CdSe thin films by varying the concentration of $(CH_3 COO)_2 Cd$ also by changing the PH of solution and temperature of bath. The optimum conditions are determined as,

$(CH_3 COO)_2 Cd , 2H_2$ - 0.2 m - 20 ml

$NH_4 OH$ - - 04 ml

Distilled water - - 50 ml

$NH_4 OH$ - - 50 ml

$Na_2 Se So_3$ - 0.1 m - 20 ml

Final PH - 10.4

Bath Temperature - $35^{\circ} C$

In one beaker four slides are dipped and slides are taken out one by one, after the Interval of 30 minutes in order to have different thicknesses.

CdSe Films: ($\rho = 5.816 \text{ gm/cm}^3$)

Slide Number	Initial Weight	wt. after Deposition	Weight Diff. Δw gm	Area (A)= cm^2	$t = \frac{\Delta w}{\rho X A}$
11	5.375	5.380	0.005	10.8	450 \AA^0
04	5.125	5.314	0.009	10.8	800 \AA^0
10	5.415	5.426	0.011	7.56	1050 \AA^0
12	5.510	5.525	0.015	10.8	1300 \AA^0
13	5.405	5.425	0.020	10.8	1500 \AA^0

i) Preparation of samples of pure CdSe (Cadmium Selenide thin films.) :-

These films were deposited on glass slide substrates using the chemical bath deposition (CBD) Technique. The films are obtained in the reaction bath at temperature in the range $(30 - 50)^\circ\text{C}$. The Structure and surface morphology of the films were studied by EDAX and SEM. Also the Electrical properties were determined by four probe resistivity method and thermoelectric power apparatus in the lab of physics Nanasheb.Y.N.Chavan. A.S.C College, Chalisgaon.

Also the optical properties were determined from spectroscopy transmission (T) and reflection (R) in the wavelength range of 200 nm to 2600nm. Many attempts are made to optimize the optimum condition for deposition of samples of CdSe. Following sets of the samples of different thicknesses are prepared.

- 1) Set 1 - 450 \AA^0
- 2) Set 2 - 800 \AA^0
- 3) Set 3 - 1050 \AA^0
- 4) Set 4 - 1300 \AA^0
- 5) Set 5 - 1500 \AA^0

ii) Electrical Characterization: - Measurement of resistivity by four probe method and measurement of Thermo electric power by TEP instruments is performed in the T.F. physics laboratory of Nanasheb Y.N. Chavan A.S.C. College Chalisgaon.

iii) Optical characterization:- Measurement of transmission (T) and Reflectance (R) in the Wavelength range (200-2600) nm is performed in the 'COSIST LAB' Department of physics University of Pune.

iv) Determination of EDAX & SEM:-

Also the EDAX & SEM (Scanning Electron Microscope) is also determine in the scanning electron microscope lab Department of physics University of Pune.

v) Synthesis of CdS Thin Films by CBD Technique :-

Number of trails are taken and after many attempts are made to deposite the CdS thin films by varying the concentration of $CdSo_4$, $Cdcl_2$ and Cadmium acetate and the PH is varied from 9.5 to 10.5 and the temperature is varied from $(50 - 60)^0 C$. The optimized concentrations and PH values and different steps are as follows.

$(CH_3 COO)_2 Cd$	-	0.1 m	-	25 ml
EDTA	-		-	10 ml
Distilled water	-		-	10 ml
Thiourea	-	0.1 m	-	25 ml
$NH_4 OH$	-		-	08 ml

Final PH - 10 and Bath Temperature - $55^0 C$

In one beaker four slides are dipped and slides are taken out one by one, at the Interval of 45 minutes for different thicknesses.

CdS Films: ($\ell = 4.82 \text{ gm/cm}^3$)

Slide Number	Initial Weight	wt. after Deposition	Weight Diff. Δw gm	Area (A)= cm^2	$t = \frac{\Delta w}{\ell X A}$
34	5.350	5.140	0.005	9.36	$400 A^0$
31	5.230	5.237	0.007	9.36	$750 A^0$
32	5.500	5.510	0.010	9.00	$950 A^0$
29	5.660	6.675	0.015	9.00	$1200 A^0$
26	5.670	6.688	0.018	8.64	$1550 A^0$

2) Preparation of Thin film samples of pure CdS (Cadmium Sulfide)

Cadmium Sulfide thin films were deposited on glass substrates using chemical bath deposition (CBD) techniques. The films are also obtained in reaction bath at temperature range $(50 - 60)^{\circ}\text{C}$. The structure & surface morphology of films were studied by EDAX & SEM. Also the electrical properties were determined from resistivity & thermoelectric power apparatus in the T.F. laboratory of physics department Nanasaheb Y.N. Chavan A.S.C. College, Chalisgaon.

Also the optical properties were determined from spectro copy transmission (T) and reflectance (R) in wavelength range of (200 - 2600) nm.

After many attempts are made to optimize the optimum condition for deposition of sample of pure CdS Thin Films.

Following sets of samples of different thicknesses are prepared

- 1) Set 1 - 400Å°
- 2) Set 2 - 750Å°
- 3) Set 3 - 955Å°
- 4) Set 4 - 1200Å°
- 5) Set 5 - 1550Å°

i) Electrical Characterization:-

Measurement of resistivity by four probe method and measurement of thermo electric power by TEP instrument is performed in T.F. physics laboratory of Nanasaheb Y. N. Chavan A.S.C. College, Chalisgaon.

ii) Optical Characterization measurement of transmittance (T) and Reflectance (R) in the Wavelength range of (200-2600) nm is performed in 'COSIST LAB' Department of physics Pune University Pune.

iii) Determination of EDAX-SEM:-

Also the EDAX & SEM (scanning electron microscope) is also determine in the scanning electron microscope lab Department of physics university of Pune.

Synthesis of CdSeS Thin Film by CBD Technique:-

Many attempts are made to deposit CdSeS Thin Films by varying the concentration of solutions and also by varying the temperature and PH of the solutions , the steps are as follows

$(CH_3 COO)_2 Cd$	-	0.1 m	-	20 ml
EDTA	-		-	05 ml
Distilled water	-		-	10 ml
Thiourea	-	0.1 m	-	20 ml
$Na_2 Se So_3$	-	0.1 m	-	20 ml
$NH_4 OH$	-		-	08 ml

Final pH - 9.8 and Bath Temperature - 50⁰ C

1) Preparation of samples of different compositions such as $CdSe_x CdS_{1-x}$ (x varies from 0.1 to 0.9):- Many attempts are made to deposit the sample of different composition as above also many times tried to optimize the optimum conditions and prepared the samples of same composition and also tried to use these samples for different characteristics such as electrical & optical characterization, also for EDAX and SEM but the sample adhesion was not enough proper and stoichiometric.

EDAX of pure materials CdSe:- (Atomic %)

Thickness	Cd	Se	S	Total
450 A ⁰	53.03	20.40	26.57	100
800 A ⁰	55.16	31.82	13.2	100
1050 A ⁰	---	---	---	---
1300 A ⁰	52.0	37.08	10.93	100
1500 A ⁰	53.73	34.05	12.22	100

Table 1.1

All the samples of these compounds deposited on a glass substrate are cut in to (1X1 mm) in size and EDAX/SEM (Scanning electron microscope) is determined at the center for scanning electron microscope lab in department of physics, university of Pune. From these composition percent of Cd, Se and S are determined.

EDAX of pure materials (CdS) (Atomic %)

Thickness	Cd	S	Se	Total
400 Å	61.79	38.21	---	100
750 Å	64.39	35.61	---	100
950 Å	64.42	35.58	---	100
1200 Å	68.54	30.88	0.58	100
1550 Å	68.54	31.46	---	100

Table 2.1

RESULTS AND DISCUSSIONS

For CdSe (Cadmium Selenide) pure Binary Compound :-

i) Electrical Study:- From the electrical characterization we tried to study the resistivity and other thickness dependent parameters. Also tried to study the size effect, bulk resistivity.

The electrical resistivities of the different samples are in the range $(1 - 4) \times 10^4 \Omega \cdot \text{cm}$. Also this material shows properties of photoconductor and solar energy material. Hence these materials have a wide application in device fabrications i.e. electronic device such as thin film transistors and γ ray detectors.

ii) Thermo Electric measurements: - From these measurements the films are shown to be 'n' type in nature.

iii)Optical Study: - From the optical characteristics of CdSe for all the samples of thickness 400Å , 800Å , 1300Å , and 1500Å .

Band gap energies are in the range (1.4 - 1.9) eV. These values are in good agreement with the reported values.

Also we had tried to determine the refractive index (n) and Extinction coefficient (K) but these values are not in good agreement with the reported work.

iv)EDAX-SEM Study:-

SEM-EDAX study CdSe:-

For the samples of thicknesses 450, 800, 1300, 1500 Å Scanning electron micrographs are presented in figures (1.1, 1.3, 1.5, and 1.7) and EDAX spectra's are presented in figures (1.2, 1.4, 1.6 and 1.8)

As deposited CdSe films are **dark radish** in colour with good adhesion for the substrate. The as grown films are polycrystalline in nature with crystallites randomly scattered on the surface. Also sizes of the crystallites are visualized on the surface.

Following points are observed from SEM photographs:-

- 1) The films are fine and lustrous.
- 2) Films shows the presence of microgranules on the complete surface, however large numbers of granules have been gathered at some places.
- 3) Also films shows regular black figures on the elevated surface shows presence of voids.
- 4) Also there is dendrite growth on the some part of surface of films.
- 5) Finally the films are lustrous and shows the presence of microgranules on the complete surface.
- 6) SEM analysis indicates that the film is covered with large number of star shaped microgranules.

EDAX-Analysis: - The EDAX analysis shows the deviation from ideal stoichiometry of atomic weight and percentage. Ideally theoretically it should be 50:50 percentage but it much more deviates from these ideal values. The average ratio of atomic percentage is approximately 55:45. These films form **hexagonal** structure.

Table 1.1 shows the detail elemental analysis of the composition of starting basic ingredients and film composition comparisons.

The EDAX spectra for different thicknesses are shown in the figures (1.2, 1.4, 1.6, and 1.8).

RESULTS AND DISCUSSIONS: - (PbS):-

i) Electrical Study: - From the electrical characteristics we tried to study the resistivity and the thickness dependent parameters. Also tried to study the size effect and bulk resistivity the results are not in good agreement with the reported values.

The electrical resistivity of the CdS is of the order of $10^4 - 10^5$ (Ω cm).

Also these are the semiconducting materials in the conversion of solar energy into electrical energy by means of PEC process. Hence these materials have wide applications in device fabrication photoconductors, photo resistors, also in transistor image magnifications.

ii) Thermo electric measurements: - These measurements the films show 'n' type electrical conductivity.

iii) Optical Study: - from the optical characterization of CdS for all samples of thicknesses 400, 750, 950, 1200, 1550 \AA . Band gap energies are determined that are approximately in the range (2 – 3)ev. These values are in good agreement with the reported values.

Also we tried to determine the refractive index (n) and extinction coefficient (K) but these values are not in agreement with the reported values.

iv) SEM-EDAX study(CdS):- For the samples of thicknesses 400,750,950,1200,1550 \AA , the scanning electron micrograph are presented in the figures (2.1,2.3,2.5,2.7 and 2.9) and as the deposited CdS films are **dark yellowish** in color with good adhesion for the substrate. The films are dense smooth and homogeneous without visible pores.

Following points are observed from SEM photographs:-

1) The films show presence of grain boundaries.

2) Most of the grain boundaries are with the shape irregular triangle, rectangular, pentagons and hexagons some of them are approximately of the same area even though they are not oriented in the same direction.

3) Although all the grain boundaries are not well defined and randomly oriented.

4) Also lines of demarkation are clearly visible and well defined and the films are completely covered with grain boundaries.

EDAX- Analysis: - The EDAX Analysis is also shows the deviation from ideal stoichiometry of atomic weight and percentage. Ideally theoretically it should be 50:50 percentage but it much more deviates from the ideal values.

Table 2.1 shows the detail elemental analysis of the composition of starting basic ingredients and film compositions.

The EDAX- spectra for different thicknesses are shown in the figures (2.2, 2.4, 2.6, 2.8 and 2.10).

Conclusions:

- 1) The CdSe has shown great promises as a photoconductor and solar energy material. These films have the applications in manufacturing of electronic devices, thin film transistors and γ ray detectors, also used in IR optics, polarizer's, substrates detectors and source for vacuum deposition.
- 2) The electrical resistivity of CdS is of the order of $10^{+5} \Omega \text{ cm}$ with 'n' type electrical conductivity and is often used in optoelectric devices, especially incase of heterojunction solar cells. Also CdS is a promising semiconductors materials in the conversion of solar energy in to electrical energy by means of PCC process.