STUDIES ON CHEMICAL SYNTHESIS AND PHYSICAL PROPERTIES OF CADMIUM BASED CHALCOGENIDE MATERIAL ON THIN FILMS

REPORT SUBMITTED

TO

DEPARTMENT OF PHYSICS

SWAMI MUKTANAND COLLEGE OF SCIENCE, YEOLA (NASHIK), INDIA

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE

DEGREE OF BACHLOR OF SCIENCE

IN PHYSICS

By

BHAGAT PARAS (T.Y.B.Sc.)

VAVAYARE JITENDRA (T.Y.B.Sc.)

BHATE DEEPAK (T.Y.B.Sc.)

PINGAT ANIKET (T.Y.B.Sc.)

DEPARTMENT OF PHYSICS

SWAMI MUKTANAND COLLEGE OF SCIENCE, YEOLA (NASHIK), INDIA

Project Supervisor

DR.S. V. DHANWATE

HEAD,DEPARTMENT OF PHYSICS

SWAMI MUKTANAND COLLEGE OF SCIENCE, YEOLA (NASHIK), INDIA

CERTIFICATE

This is to certify that the report entitled "Studies On Chemical Synthesis And Physical Properties Of Cadmium Based Chalcogenide Material On Thin Films" submitted by Mr. *Bhagat Paras ,Vavayare Jitendra ,Bhate Deepak ,Pingat Aniket Of T. Y.B.Sc class* of project work done under my guidance and supervision and the report has not formed the basis for the award to the scholar for any Degree, Diploma, Associateship, Fellowship or any other similar title and I also certify that the report represents an independent work on the part of the candidates.

Place :- Yeola **Dr. Dhanwate S V**

Date : 30/ 01/2020 **(Project Supervisor)**

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PART -I

INTRODUCTION TO THIN FILM

INTRODUCTION

The technology and understanding of films less than 1 micron thick have made tremendous advances in the last decades, primarily because of the industrial demand for reliable thin-film microelectronic devices to fulfill the urgent needs of the sputnik era. This progress has brought maturity and much scientific confidence in the use of thin films for basic and applied research. In addition to major contributions to a variety of new and future scientifically based technologies, thin films studies have directly or indirectly advanced many new areas of research in solid-state physics and chemistry, which are based on characteristic such as the thickness, geometry and structure of films.

The thin film technology has wide spread applications as it is easy to reproduce the film. Very few material is required to deposit the film on given substrate. With the help of thin film various properties of the bulk materials can be studied; which prevents the wastage of material and is economically cheap. Thin film technology has important role in the development of various fields such as microelectronics, optical coatings, integrated optics, thin film metallurgical coating, amorphous materials, surface engineering and solar energy conversion devices. New fields are also emerging for the thin films. The materials with macrometric and nanometric and the industrial applications of micro science and micro technology for the development of synthetic materials of tailored properties of VLSI / GSI, communication, informatics and solar energy conversion with decreasing size of active electronic devices, a higher packing density, higher speed performance and lower cost are obtained .

Thin films play very important role in conversion of solar energy into other forms of energy in several directions. The optical properties of specifically designed thin film structure can be used as reflection or antireflection coatings and as selective absorbers. Electrodeposited semiconductors have found applications in solar selective coatings, solid-state solar cells, Schottky barrier devices and photoelectrochemical (PEC) solar cells.

Thus semiconductor forms the backbone of all modern microelectronics and opto-electronics devices. Semiconductor characterization is an integral and indispensable component of these technologies. A good description of material characterization considers it as an integral part of process development and manufacturing.

Characterization measurements address quality assurance of materials, control and monitoring of equipment and manufacturing processes, diagnostic and failure analysis, and end device performance in light on intended design and function.The restricted third dimension is termed as thickness of the film. Thickness may vary from two to several wavelengths of the light but always remains much less than the other two dimensions. A material is said to be in thin form, if its thickness is in between 100 to 100,000 A^o (less than one micron).

The thickness ‘t’ of the film can be changed from two to several times the wavelength of light but always remains much less than the other two dimensions. According to the nature of distribution of the deposited material on the substrate, thin film can be classified mainly in to two groups :

1) Continuous thin films

2) Discontinuous thin films.

Since the thickness limitation is rather arbitary ,even some what thicker films may also come with in the scop of the above definition. With in the connotation of thin film, often a further sub division of thickness is made under the categories,

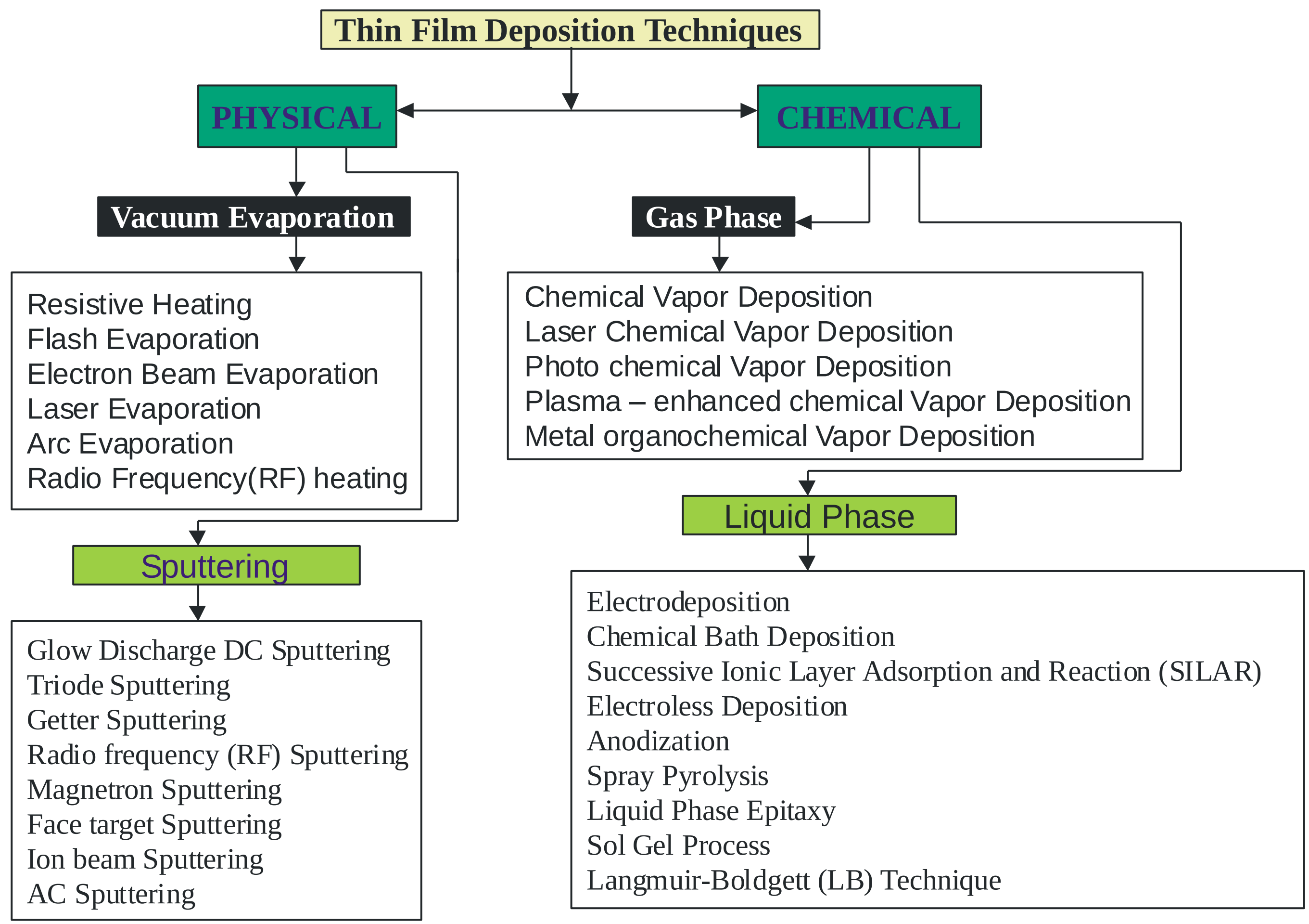
1) Ultra thin : Its ranging from few to about 50-100 A^{o}

2) Thin (very thin) : ranging from 100 – 1000 A^{o}

3) Comparatively thicker : grater than 1000 A^{o}

Nanocrystalline materials are novel materials which are not only scientifically interesting but also hold great potential for varied applications. Their properties are different and often superior to those of conventional coarse - grained polycrystalline materials and also amorphous alloys of the same composition. Nanocrystalline materials are polycrystalline materials with grain sizes of up to 1000 A$^{\circ}$. Because of the extremely small dimensions, a large fraction of the atoms in these materials is located in the grain boundaries and this confirms special attributes. Nanocrystalline materials can be prepared by inert gas condensation, mechanical alloying plasma deposition, spray conversion processing and many other methods. Nanocrystalline materials exhibit increased strength / hardness, enhance diffusivity, improved quality / toughness, reduced elastic density, reduced elastic modulus, higher electrical resistivity, increased specific heat, higher thermal expansion coefficient, lower thermal conductivity and superior soft magnetic properties in comparison to conventional coarse - grained materials. Preparation of such nanocrystalline films are quite practicable.

Deposition Techniques



- **Fig. Broad Classification of Thin Film Deposition Techniques**

Physical vapour deposition

Thermal evaporation

In thermal evaporation material is created in a vapour form by means of resistive or RF heating. The vapour atoms thus created are transported through vacuum to get deposited on substrate.

Electron beam evaporation

In electron beam evaporation, an electron beam is accelerated through a potential of 5 to 10 KV & focused on a material. The electrons loose their kinetic energy mostly as the heat and temperature at the focused spot can become as high as 3000ºC.At such a high temperature most pf the refractory metals and compounds can be evaporated.

Molecular beam epitaxy (MBE)

The deposition of single crystal (epitaxial) film by condensation of one or more beam of atoms &/or molecules from the Kundsen (effusion) source under UHV condition is called MBE.

Ion platting

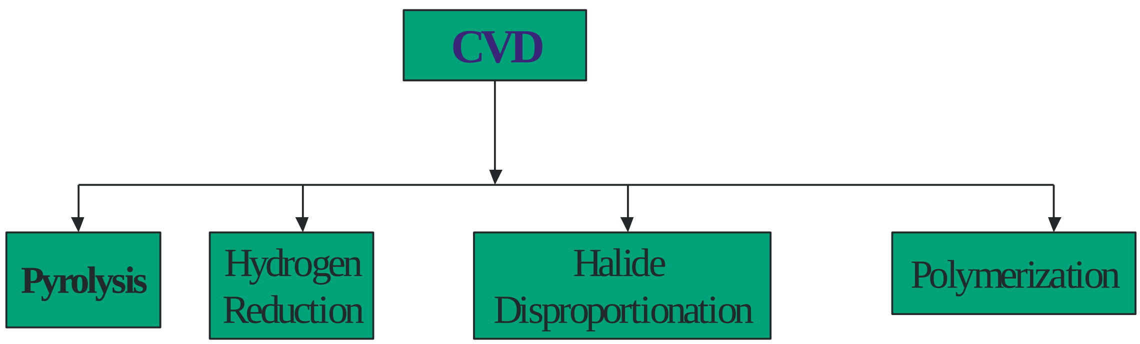
Ion platting refers to process in which the substrate and films are exposed to a flux of high energy ions during the deposition.

Sputtering

When a solid surface is bombarded with energetic particles, the surface is eroded & surface atoms are removed due to collision between surface atom and energetic particles .This phenomenon is known as sputtering.

Chemical Vapor Deposition (CVD)

Chemical vapor deposition is the decomposition of vapors of the volatile compound of the substance to be deposited by chemical reaction. It is a versatile and flexible technique since a variety of chemicals reactions are available.



Pyrolysis

It is the thermal decomposition of a compound to yield deposit of a stable residue. Organometallic compound decompose at temperature around 900K.But metallic halides decompose above 1000K. Pyrolysis of Silane (SiH_4) and Germane (GeH_4) is employed to produce the epitaxial layers of Si and Ge. Many of the organic silicate can be thermally decompose to give pure SiO_2 thin films.

The spray techniques involve spraying a solution (usually aqueous) containing soluble salts of the constituent atoms of the desired compound on a substrate maintained at elevated temperatures. The sprayed droplets on reaching the hot substrate undergo pyrolytic decomposition and form a single crystal or cluster of crystallite of the product.

Anodizing

Anodizing is an electrolytic process in which the metal is made the anode at suitable electrolyte. The cathode is metal or graphite. When an electric current is passed, the surface of metal is converted to a form of its oxide having different properties.

Electrodeposition Techniques

Elctrodeposition is more than 150 year old thin film technique. This has largely been used to deposit metal films. In addition, during the last 25 years electrodeposition has been more intensively studied in preparing compound semiconductors. In comparison with other techniques electrodeposition is relatively easily scalable & cost effective as it is a non-vacuum room temperature method. In addition substrates with various sizes & shapes may be used & toxic gases precursors are not needed unlike in many gas phase techniques. The choice of the particular method depends on several factors like material to be deposited, nature of substrate, required film thickness, structure of the film, application of the film etc. Among the methods mentioned above, electrodeposition method is most popular today because it is easier, attractive & less expensive.

In recent years, materials (metal, semiconductors, ceramic, superconductors, conducting polymers etc.) in thin films form have been prepared by electodepostion technique. This technique has following features:

1. It is an isothermal process in which, the thickness & morphology of the films can be easily controlled by electrochemical parameters such as electrode potential and current (charge).

2. Relatively uniform films can be obtained on substrates of complex shapes.

3. The deposition rate is higher than other physical and chemical methods.

4. The equipment needed is not expensive and does not require sophisticated instrumentations and vacuum.

5. Electrodeposition usually has low operating temperature.

a) Fundamentals of electrodeposition

1. <u>Electrolyte</u> The electrolyte or bath is a conducting medium in which the flow of electric current takes place by migration of ions. It can be aqueous, non-aqueous or molten and it must contain suitable metal salts.

2. <u>Elctrode</u> an electrode is a conductor through which an electric current enters or leaves an electrolyte. An electrode connected to the terminal is effected as an anode where as another is referred as cathode. At anode, negative ions are discharge or the ions are formed or oxidizing reaction occurs.

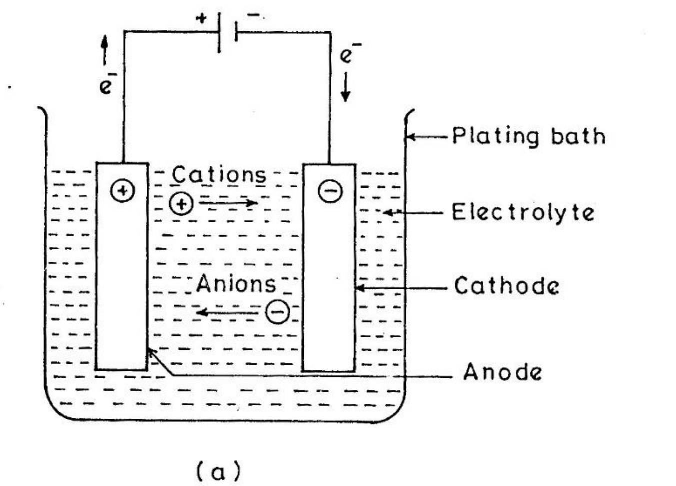
3. <u>Elctrode potential</u> an electrode potential is a difference in potential between electrode and the immediately adjacent electrolyte.

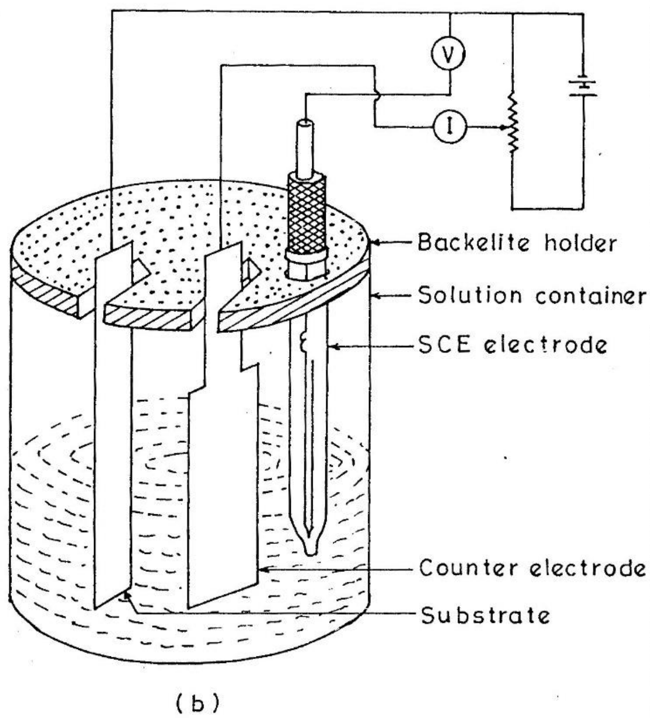
4. <u>Standard electrode potential</u> A standard electrode potential is equilibrium potential, for an electrode in contact with an electrolyte, in which all the compounds of a specified electrochemical reaction are in their standard state.

b) Basic concepts of electrodepositon

Elctrodepositon is a process of depositing a substance upon an electrode by electrolysis (i.e. the production of chemical charges by the passage of current through an electrolyte). The phenomenon of electrolysis is governed by Faraday's laws of electrolysis, a relationship between the quality of electricity passing through an electrolyte and the amount of any material liberated or deposited at the cathode was discovered by Michel Faraday known as Faraday laws of electrolysis.

<u>First law of electrolysis</u> The amount of any substance i.e. liberated or deposited on an electrode during electrolysis is directly proportional to the quantity of electricity passed through electrolyte.





(a) SCHEMATIC DIAGRAM OF THE MECHANISM OF ELECTRODEPOSITION.
(b) SCHEMATIC DIAGRAM OF DEPOSITION CELL.

Fig: Electrodeposition

The electrode gains a certain charge on itself which attracts oppositely charged ions, & water molecules holding them at the electrode / electrolyte interface by electrostatic forces. A double layer consisting of an inner layer of oriented water molecules interposed by perfectionlly adsorbed ions immediately on the electrode followed by a second layer of charge opposite to that of the electrode is formed. During deposition ions reach the electrode surface move to stable position on it, release their legands (water molecules or complexing agents) if solvated, release their charges and in the process undergo the stipulated electrochemical reaction. The rapid depletion of the depositing ions from the double layer region is compensated by a continuous supply of fresh ions from the depleted region occurs because of the following:

1. Diffusion owning to concentration gradient.

2. Migration owning to applied electric field &

3. Convection current in electrolyte.

The factors influencing electrodeposition process are:

1. pH of the electrolyte
2. Current density
3. Temperature of bath
4. Bath composition
5. Electrode shape
6. Agitation

Various metals like Cu, Ag, Au, Fe, Co, Ni, Pd which occupy the central part of the periodic table & alloys such as Cu-Sn, Co-Ni, Fe-Ni, Sn-Zn,Sn-Pb have been successfully electroplated from aqueous solutions. None of the so called refractory metals can be deposited in the pure state from aqueous solutions. Semiconductors such as CdSe have been deposited by co-deposition of the Cd & Se from aqueous solution. Semiconductor metals chalcogenides like CdS, HgS, CdSe, etc. have been deposited from organic solutions of metal salt & elemental chalcogenides

PART II

EXPERIMENTAL TECHNIQUES

Experimental Set up for Electro Deposition

The electrodeposition set up used for the preparation of CdSe thin film is schematically shown in Fig

It consists of following parts:

a. Bakelite holder

It is a dIsc-type in shape having thickness 1 cm and diameter 2.5 cm. It consists of two slots, one for substrate and another for counter electrode with attachment of screws to hold them. A hole is made to the bakelite holder near the substrate slot through which saturated calomel electrode (SCE) can be inserted in an electrolytic solution.

b. Solution container

It is a corning glass cell, cylindrical in shape of 40 c.c.

c. Counter electrode

It is highly pure graphite plate. The size of electrode was 2 x 4 x 0.3=2.4cm^3.

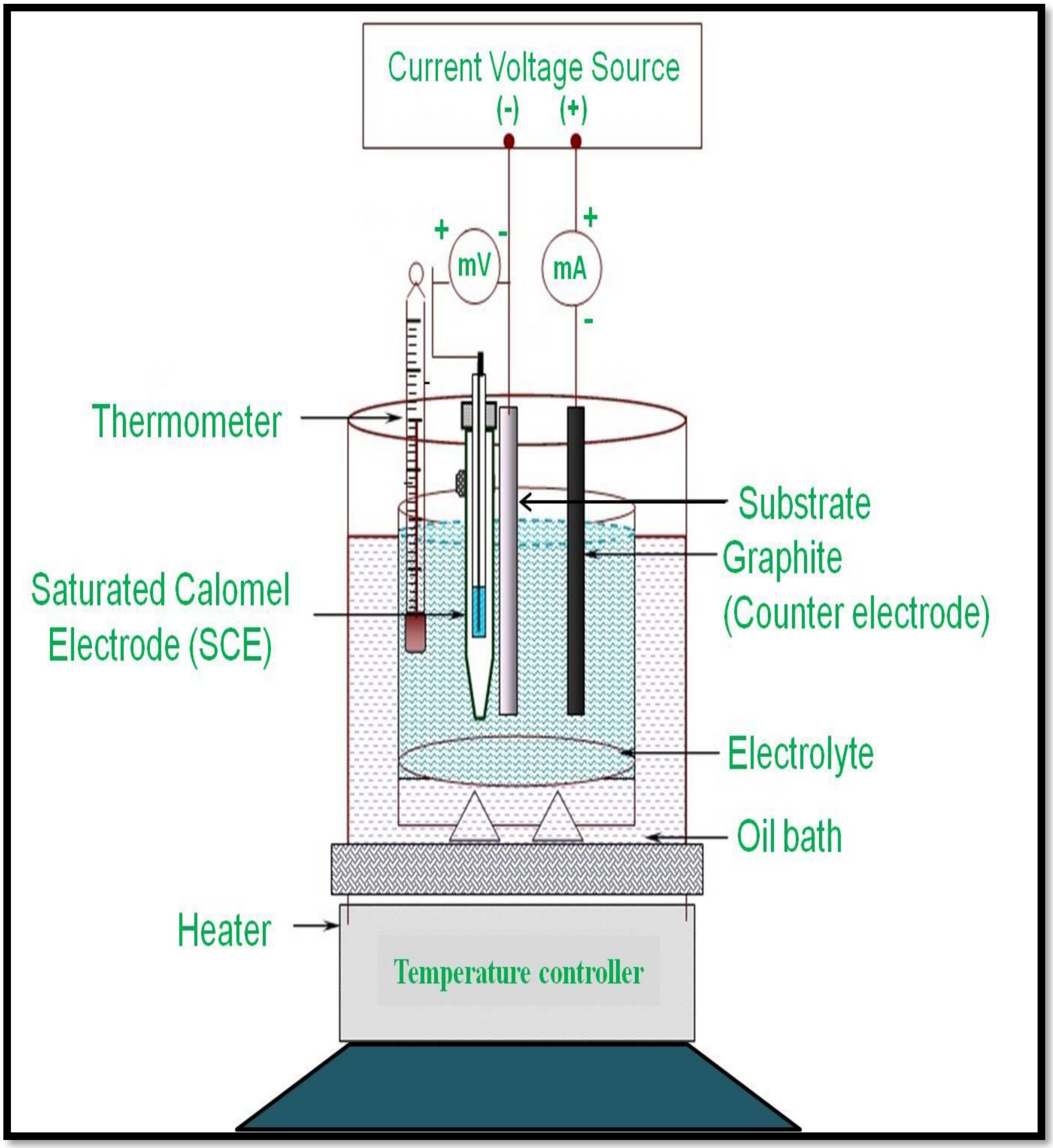


Figure Experimental setup for electrodeposition technique.

d. Substrates

Commercially available stainless steel strips of a size 1x 4=4 cm^2 and fluorine doped tin oxide (F.T.O.) covered glass substrates were used as substrates in an electrodeposition bath. The reference electrode employed was saturated calomel electrode (SCE).

Substrate Cleaning

Electrodeposition process needs electrically conducting substrates for thin film deposition. Usually, metallic substrates like copper (Cu), stainless steel (SS), Titanium (Ti) and FTO coated glasses are useful as substrates. The preparation of smooth, starch free substrate surface is highly essential in electrodeposition because inhomogenities tend to be amplified during the electrodeposition. Polishing is needed for the metallic substrates before using them as cathodes, however, FTO coated glass substrates doesn't require polishing .

The surface of metallic substrates is cleaned in the following way:

i. The metallic substrates are thoroughly polished to appear mirror like surface using fine grade polish paper.
ii. The substrates are then washed with detergent solutions (Labolene or some equivalent), with the help of cotton and flowing water.
iii. They are etched with 25% dilute HCl or for approx. 20 seconds and cleaned in ultrasonic cleaner.
iv. Finally they are dried in vapors of alcohol.

Preparation of Solutions

The constituents utilized to prepare CdSe thin films are described below.

i. Cadmium acetate
ii. Selenium dioxide
iii. Ethylene diamine tetra acetic acid (EDTA) Tetra sodium salt,
iv. Ethylene glycol

All the solutions were prepared in Ethylene glycol
An appropriate amount of salts were dissolved into Ethylene glycol to get desired concentration of the solution.

Thickness Measurement

The films were deposited for different time interval and thickness was calculated.

The formula used is,

Thickness = Mass deposited/ (density × area)

$t = \Delta M / (\rho \times A)$

Where, ΔM= mass deposited in gram.

For that, the mass of the film before and after the deposition was noted.

Deviation of Thickness with time of deposition on(a) SS substrates (b) FTO coated glass substrates.

Table Thickness measurement

Figure Deviation of Thickness with time of deposition on

Sr. No.	Deposition time (min.)	Thickness on SS (μm)	Thickness on FTO (μm)
1	10	1.91	1.10
2	15	3.45	2.54
3	20	5.78	2.49
4	25	4.45	3.07
5	30	3.87	3.45

PART III

RESULT AND CONCLUSION

In recent years the cadmium chalcogenide have received great attention by many scientists and researchers because of their semiconducting and other interesting physical and photoelectrochemical properties. In the present work the binary compound of CdSe thin film was prepared and characterized to study the above-mentioned properties. These films are prepared by electrodeposition technique, which is relatively inexpensive and short duration method.

The films were deposited by optimizing the preparative parameters such as bath temperatures, pH, deposition time, deposition potential

Before deposition of thin films substrate cleaning is done thoroughly .

The films deposited on the stainless steel substrate are more uniform and smooth as compared to those on FTO coated glass. For optimization of all preparative parameters are simple and suitable.

From these studies it is concluded that the growth of deposits can be controlled by controlling preparative parameters. The variation of thickness with deposition time is studied and it is noted that the film deposited for time 35 min. shows maximum thickness of 4.45 µm on stainless steel substrate and 3.07 µm on FTO coated glass substrate.

Departmental Research Activity

After the complete study of this project Mr. Jitesh Vavayare , Mr. Aniket Pingat , Mr. Paras Bhagat and Mr. Deepak Bhate were presented research paper in title of " *Physics and Technology with Thick and Thin Films*" in two days national conference on Synthesis , Characterization of promising Nano materials for energy and Environmental Application, which is jointly organized by Department of Physics, V. N. naik College , Nashik and BOD, SPPU, Pune
under the guidence of Dr. Dhanwate S V.

In the above photo at center the chief geust of this conference Hon Dr. Mahendra More , Head, Department of Physics, Savitribai Phule Pune University,Pune, on his right side Hon.Dr. Sahebrao Dhanwate Head, Department of Physics, Swami Muktanand College of Science , Yeola Dist. Nashik, Hon. Dr. Arun Garde , Research Supervisor, and Associate Professor in Physics, MSG College Maalegaon Camp(Nashik), at left side Dr. Smt. Sardar , Head, Department of Physics, ASC College , Dindori (Nashik) along with Physics students

In the above photo at center the the Hon. Principal Dr. Dhanraj Goswami, Swami Muktanand college of Science, Yeola (Nashik), discussed with students and gave the best wishes for paper presentation to students , on this event Dr. Dhanwate S V , Head Department of Physics, Swami Muktanand college of Science, Yeola (Nashik),

Hon. Principal Dr. Dhanraj Gosawmi and Dr. Sahebrao Dhanwate , Head Department of Physics, Swami Muktanand College of Science, Yeola (Nashik) with their college students

Mr. Aniket Pingt, Mr. Paras Bhagat, Mr. Jitendra Vavyare and Mr. Deepak Bhate at the time of poster presentation

at

Mr. Aniket Pingt and his friends , explain their views about research work through poster presentation in the presence of Evaluation committee member with Principal Dr. Dhanraj Gosawmi and Dr. Sahebrao Dhanwate

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Tel. (0253) 2576692 Website: www.kvnacs.org.in

E-mail: vnnaik_col@rediffmail.com, seminarphysics.vgw@gmail.com

ABSTRACT BOOK

Organizing Chair
Dr. Shantaram P. Badgujar
Principal, Kr. V. N. Naik Shikshan Prasarak Sanstha's
Arts, Commerce and Science College, Nashik

Convener
Dr. Vasant G. Wagh
HOD, Department of Physics,
Kr. V. N. Naik Shikshan Prasarak Sanstha's
Arts, Commerce and Science College, Nashik

Physics and technology with Thick and Thin films

Vyavhare Jitesh, Bhagat Paras, Bhate Deepak, Pinget Aniket, Wagh Ajinkya
Deparment of Physics, Swami Muktanand College of Science, Yeola (Nashik), India -423401

Abstract:

The technologies thin and thick film less than one micron thick have made tremendous advances in the last decades, primarily because of the industrial demand for reliable thin film microelectronic device to fulfill the urgent needs of the era. This progress brought maturity and much scientific confidence in the use of thin film for basic and applied science with decreasing size of active electronic devices, a higher packing density, higher speed performance and lower cost are obtained. At the same time, as the size of active materials decreases below a certain length characteristic of the physical phenomenon being investigated in the material e.g. mean free path of conduction, penetration depth in superconductors, wave length of radiation etc, geometrical & quantum size effect manifest. These effects begin to dominate the physical phenomena as the size of material is reduced in one, two & three dimensions. This low dimensional materials exhibit quantization, non-equilibrium electron, phonon & photon transport processes, metastable & stable structures, surface dominated diffusion & chemical processes. The pivotal role of thin film technology in the development of such diverse and challenging frontiers as microelectronics, optical coating and integrated optics, thin film superconductivity and quantum engineering, surface science, micro-organism, metallurgical coating and amorphous and crystalline materials is now a part of nano science

Keywords: - Thin film, Thick film, Physical and Chemical parameters

Synthesis, characterization and properties of chemically deposited CuInSe2 and CdSe thin films

M. G. Dhanvate[1] and S. V. Dhanwate[2]

1Department of Physics, RNC Arts, JDB Commerce and NSC Science college, Nashik Road, Nashik, India

2Department Of Physics, Swami Muktanand College of Science Yeola – 423401 , India

Abstract:

For the last couple of decade's interests in the use of photoelectrochemical solar cells lead to large amount of research is going on in the search for thin film polycrystalline materials with acceptable efficiency; sometimes approaching that of single crystals. In recent years the cadmium chalcogenides and copper chalcopyrite thin films have received great attention by many scientists and researchers because of their semiconducting and other interesting physical and photo electrochemical properties. The main aim of this research work is study of various physical properties by different techniques such as, X-ray diffraction, Energy Dispersive Analysis by X-rays, Scanning Electron Microscopy and optical absorption techniques. In Future, from these optimum parameters were determined for production of completed solar cell devices.

Keywords:- Chemical Electro Deposition, XRD, SEM, E-DAX, Optical Properties,

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Design of nanostructures for optical and photoelectrochemical properties of CInSe

Dr. Dhanwate S. V.
Associate Professor and Head Department of Physics, Swami Muktanand College of Science, Yeola (Nashik), India

Abstract:

The morphology and size of nanostructures can be tuned by controlling the reaction condition such as temperature, pH, presence of additives such ions, solvent have been widely studied .CuInSe thin film are prepared by electro deposition technique over stainless steel substrates in an aqueous acidic bath containing CuS04, InCl3, and Seo2. The stainless steel plates were used as the cathode in electrodes cell with graphite as the counter electrode and saturated calomel electrode (SCE) was the reference electrode. The electrolyte was prepared by mixing solution of CuSo4 (0.1M), InCl3 (0.075M), and SeO2 (0.05M) The pH of electrolyte solution was varied by dilute HCL. Before deposition the substrate were thoroughly cleaned with double distilled water. The distance between the working electrode and counter electrode way kept constant as 1.5 cm during deposition. n-p junctions for the separation of photogenerated charge carriers makes good candidates for photocatalysis For visual – light observation shows structure of material The X-ray diffraction (XRD) analysis of the deposited film showed presence of polycrystalline nature. The surface morphology studies by scanning electron microscope (SEM) shows that the deposited film are well grains are uniformly distributed over the surface of stainless-steel plates

Keywords:- Nanoparticles, deposition process, CuInSe, room temperature, XRD analysis, optical and morphological property

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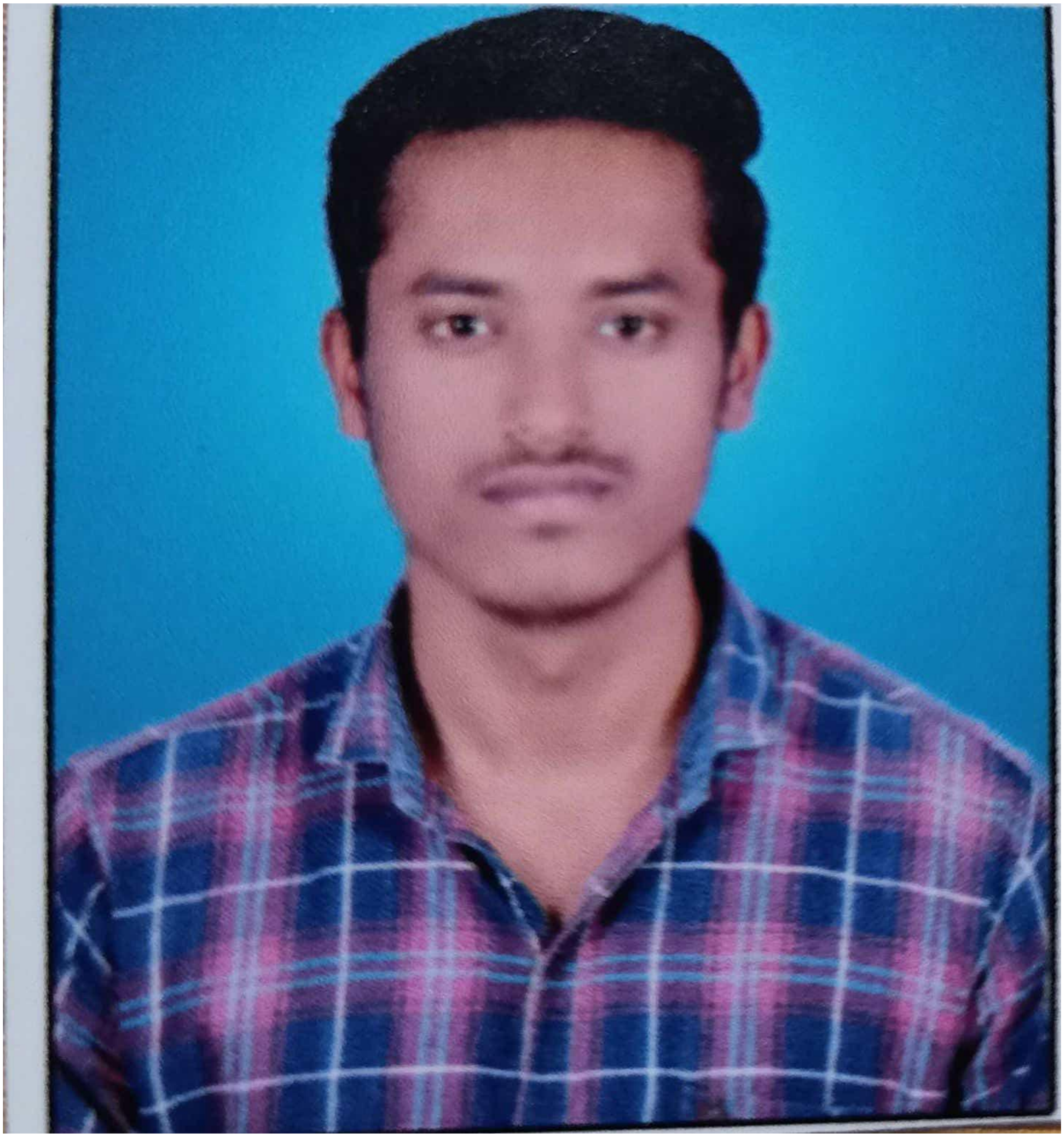

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