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Effects of CdCl₂ Treatment on Physical Properties of CdTe/CdS Thin Film Solar Cell

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Abstract

We report CdTe, CdS, and ITO thin films on glass substrates for solar cell fabrication by closed space sublimation and chemical bath deposition. CdTe and CdS thin films were sublimated to chemical treatment at 25°C in a saturated CdCl₂ solution (1.04 g/100 ml methanol) and heat treated at 400°C for 30 minutes. Indium tin oxide and tellurium films were analyzed by spectrophotometer and scanning electron microscopy. It has been observed that solar cell performance can be improved by depositing a CdCl₂ layer on the CdTe/CdS layers. The optical, structural, and morphological changes of CdTe and CdS surfaces on CdTe/CdS/ITO/glass solar cells due to CdCl₂ solution treatment followed by annealing for 400°C were studied. Optical analysis showed about 15% decrease in transmittance after CdCl₂ heat treatment in case of CdTe thin film, whereas CdS thin film demonstrated an increase of about 10–15% transmittance after CdCl₂ heat treatment. Similarly, a decrease in band gap values was found for both CdTe and CdS thin films after CdCl₂ heat treatment. XRD and SEM results of CdCl₂ heat-treated CdTe and CdS samples showed recrystallization, reorientation, and progressive increase in grain size. The grain sizes of CdTe and CdS samples demonstrated an increase of about 0.2 μm.

Keywords: CdCl₂, cadmium sulfide, cadmium telluride, heat treatment, morphological, optical, structural

1. Introduction

Considering the high absorption coefficient, near optimum band gap, and manufacturability of cadmium telluride (CdTe), it can quite easily be regarded as one of the most favorable photovoltaic materials realizable for use as high-efficiency and low-cost thin film solar cell. Typically, a



CdTe solar cell structure consists of Au/CdTe/CdS/ITO/glass. It has a direct band gap of 1.45 eV and only 1 μ m CdTe can absorb more than 90% of the photons having energy greater than 1.45 eV that is why it is well suited for solar cell applications. CdTe is stable up to 500°C. CdTe has lattice constant of 0.68 nm at 300 K. The thermal conductivity of CdTe is 6.2 W m/m² K at 293 K. The specific heat capacity is about 210 J/kg K at 293 K. In solar cell fabrication CdTe is used as a p-type semiconductor which has a junction with cadmium sulfide (CdS) as an n-type semiconductor. Infrared detector material (HgCdTe) is manufactured when CdTe is alloyed with mercury. An important application of CdTe is as a radiation detector that is used to detect X-ray and alpha, beta, and gamma rays, and for this purpose, CdTe is doped with chlorine. CdTe has electron affinity of 4.3 eV. The work function is 5.5 eV for p-type CdTe. It can be doped both as p-type as well as n-type semiconductors [1–3].

A II-VI semiconductor compound cadmium sulfide (CdS) has much importance because of its many applications in several heterojunction photovoltaic thin film devices of CdTe, copper indium gallium diselenide (Cu(In_xGa_{1-x})Se₂), or copper indium gallium sulfide (CIGS) and solar cells. Many other devices in the fields of electronics, optics, and infrared are also fabricated by using CdS [1–3]. The CdS is a suitable n-type material, which can be fabricated by a variety of fabrication techniques like sol-gel technique [4], close spaced sublimation (CSS), chemical bath deposition (CBD), thermal evaporation, chemical vapor deposition, molecular beam epitaxy, and spray pyrolysis. Each and every deposition process provides different optical, structural, electrical, and morphological properties [5–10].

First solar cell has reported CdS deposited by high rate vapor transport deposition (HRVTD). CdS is called window layer due to its higher rate of light transmission [11–14]. CdS thin films are very suitable for so many other semiconductor devices and radiation detectors. CdS thin films with wide band gap are highly used for photovoltaic devices. Since CdS is used as window layer in solar cell, it should be fabricated very thin and high amount of light should pass through CdS and be absorbed in CdTe. The CdS thin film must be continuous to reduce the effect of short circuit in the cell. The CdS thin films deposited by CBD can fulfill these requirements. So the thickness of CdS has much importance for the high-efficiency of solar cell [15–17]. CdS (CBD) thin films are grown by cadmium chloride, ammonium nitrate, and potassium hydroxide. After heating to a specific temperature, thiourea is added to start the fabrication process [18–20].

CBD is a very easy process for the fabrication of CdS thin films on ITO glass. For the fabrication of thin film solar cell, one needs a very thin film up to 60–80 nm. It is a very suitable process especially for solar cell point of view. It is a process in which substrate is placed in a hot chemical solution stirring vigorously for a specific time, positive and negative ions will reach and meet on the substrate and thin film is grown. The advantage of this technique is that neither vacuum and nor very high temperature is required for CBD [5, 21].

CdS thin films fabricated by the CSS are also another moderate temperature fabrication technique in a vacuum chamber. More than 20% efficiency has been achieved by using CdTe/CdS heterojunction thin film solar cell by this technique. CSS is a moderate temperature procedure so it provides better results in some cases [22, 23]. The CSS technique is one of the techniques that have produced encouraging results [15] mainly because it is a

simple deposition apparatus and high transport efficiency and deposition can be done in low vacuum at moderate temperatures. There is minimum use of material in the CSS system as compared to other methods. As the substrate is close to the source materials in the CSS technique, roughness increases in the thin films and has high absorption, which makes it a suitable material for solar cell applications. The CSS has a disadvantage as one cannot introduce a thickness monitor.

The general working properties of solar cells can be best described by three parameters: short circuit current (I_{SC}), open circuit voltage (V_{OC}), and fill factor (FF). Postdeposition processing of polycrystalline CdTe/CdS heterojunction thin film solar cells with cadmium chloride (CdCl₂) heat treatment has been demonstrated to improve the short circuit current and open circuit voltage of CdTe/CdS thin film solar cell, by recrystallization, reorientation, and grain enhancement of films for photovoltaic operation [24–26]. Heat treatment with CdCl, has been known to be a key step in high quality CdTe/CdS solar cells preparation [27–31]. Irrespective of the method being used for deposition of CdTe and CdS layer, CdCl, treatment has become a customary and vital process in fabrication of high-efficiency CdTe/CdS-based photovoltaic devices. Three different methods of CdCl, treatment are widely known to be used; solution CdCl₂ treatment, evaporated CdCl₂ treatment, and vapor CdCl₃ treatment [12]. CdCl₂ treatment is basically known to activate a chemical reaction between CdTe and CdS, which is the driving force for the bulk and grain-boundary inter-diffusion of CdTe and CdS [13]. However, regardless of the CdCl, treatment method being used, the basic mechanism by which CdCl, effects CdTe and CdS can be expected as a similar process [12–15].

The optical, structural, and morphological properties of CdTe and CdS thin films are mainly subjective to the preparation route. Hence, a variety of methods have been applied for the synthesis of such materials, e.g., thermal evaporation [16], chemical pyrolysis deposition (CPD) [17], metal organic chemical-vapor deposition (MOCVD) [18-20], closed space sublimation (CSS) [21] and chemical bath deposition (CBD) [22-25]. CSS and CBD are known to produce optimal and encouraging results for CdTe/CdS based solar cells. Both the techniques have many advantages for production of photovoltaic devices under controlled conditions, such as exceptional uniformity and reproducibility of film thickness even for a large-scale module [26, 27].

There are different materials being used for the manufacturing of thin film solar cells. The silicon photovoltaic cells are covering more than 70% of the world market. Silicon solar cells containing amorphous, polycrystalline, crystalline, and now a days silicon thin film solar cells are being used. Thin film solar cells (TFSC) containing (II-VI) and (III-V) semiconductor materials have high efficiencies. These include copper indium gallium sulfide (CIGS), GaAs, Cu (InGa)Se₂, and CdTe/CdS. The selection of material depends upon the band gap, absorbing ability of material, and cost of fabrication process.

CdTe is very suitable for thin film solar cells because it has direct band gap at room temperature. In the world PV market, the CdTe based solar cells have attained 16.5% efficiency. It has very good match with CdS on ITO glass substrate. CdS is used as a window layer; it means more than 70% of light will pass through the first n-type layer of solar cells. Different techniques can be used for the manufacturing of thin film CdTe based solar cells [19–26].

It has some native defect cation vacancies (Vcd) that can behave as double accepters and anion vacancies (VTe) can behave as double donors. These vacancies can be formed with other extrinsic impurities [5, 6]. A problem of CdTe is that it is toxic if ingested and if its powder is inhaled. If properly processed, manufactured, and encapsulated, then it may be harmless. It is observed that elemental cadmium is more toxic than CdTe. It is also studied that CdTe quantum dots causes extensive reactive oxygen damage to cell membrane, mitochondria, and cell nucleus.

If it is to be used at large scale commercialization of solar panels then exposure and long term safety of CdTe will be serious issue. So attempts are being made to overcome all these issues. The BNL has given the research that CdTe large-scale PV modules have neither any health risks nor any threats for environment. These modules can be recycled. These modules do not produce any pollutants during their working for long term [7, 8]. Major issue for the CdTe PV modules is the availability of tellurium. Actually, tellurium is an extremely rare element (1–5) parts per billion in the earth's crust. Manufacturing of CdTe PV solar panels at large-commercial scale will cause depletion of tellurium [9].

In the present work, effects of CdCl₂ thermal treatment on physical properties of CdTe/CdS heterojunction solar cells, fabricated using CSS and CBD, have been investigated. Correspondingly, the results have demonstrated that performance of solar cell can be improved significantly after this treatment. In this regard, optical properties such as film thickness, refractive index, absorption, and optical band gap, crystallographic properties such as crystallite size and plane orientation, and morphological properties such as grain size, have been investigated using UV-VIS-IR spectrophotometer, X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively, at room temperature (25°C) [28–31].

2. Why thin films

Thin film technology is well known technology for physics and engineering applications. It is projected to be one of the major processing techniques to fabricate electronic, optical, and magnetic data storage devices, solar cells, light emitting diodes (LED), etc.

Thin film science and technology play a crucial role in the high-tech industries that will bear the main burden of future, while the major exploitation of thin films are in microelectronics, communications, optical electronics, coatings of all kinds, and energy generation. Thin films of various materials are currently in use as protective and optical coatings, electronics, antireflection films, polarizers, radiation detectors, and solar energy converters.

It is not only thickness that is defining a film but also the way of fabrication which is responsible for the uniformity, structure, and other properties of the film. The optical, structural, and electrical properties of thin film are different from the bulk materials; it is the reason these are being used. Characterization and fabrication of thin films is very easy. The structural, chemical and physical properties depend on the deposition parameters and thickness of the thin film. The electrical, optical, and mechanical behavior of thin film also depends on microstructure,

surface morphology, purity, and homogeneity. These things are strongly dependent on the fabrication methods and selected parameters and post depositions treatments also.

Stable, accurate, efficient, reliable low-cost electronic, insulating, sensors, and many other industrial devices with minimum use of material are possible due to thin film technology. Thin film devices are easy to manufacture, especially suitable for flexible solar cells.

Thin films are layers of ferromagnetic, semiconductor or ceramic materials. It ranges from fractions of a nanometer to several micrometers in thickness (usually 100 A-1 µm). It is a microscopically thin layer of material that is deposited onto a metal, ceramic, semiconductor or plastic base. Thin Films can be conductive or dielectric (non-conductive) and are used in myriad applications. Thin films have almost a two dimensional structure, so they give a better insight into the structural properties of the material. The earliest of what might be called latest thin film optics was discovered by Robert Boyle and Robert Hooke, independently of the phenomenon now known of older material even without any visible signs of tarnish, was too low. One possible explanation which he suggested was the formation on the surface of the thin layer from the underlying material.

Dennis Taylor, in 1891, published the first edition of his famous book on the adjustment and testing of telescopic objectives. In fact Taylor developed method of artificially producing the tarnish by chemical etching. Kollmorgen followed this work and developed the chemical process for different types of glass. In Nineteenth century at the same time a great deal of progress was made in the field of interferometry. The most significant development from thin film point of view was the Fabry-Perot inferometer described in 1899 which became one of the basic structures of thin film filters. Development became more rapid in the 1930s, an indeed it is in this period that we can recognize the beginning of the modern thin film optical coating. In 1932, Rouard observed that very thin metallic film reduced the internal reflectance of the glass plate, although the external reflectance was increased. In 1934, Bauer in the course of fundamental investigations of optical properties of halides produced reflection-reducing coatings and pfund evaporated zinc sulfide layers to make low loss beam splitters for Michelson interferometers. Noting incidentally that titanium dioxide could be a better material. In 1936, John Strong produced anti-reflection coatings by evaporation of fluorite to give inhomogeneous films which reduced the reflectance of glass to visible light to as much as 89% a most impressive figure. Then in 1939, Geffcken constructed the first thin film metaldielectric interference filters. The most important factor in this sudden expansion of thin film coatings was the manufacturing process. Although sputtering was discovered by the middle of nineteenth century and vacuum evaporation in the beginning of twentieth century, these were not considered as useful manufacturing processes. The main difficulty was the lack of suitable pumps and it was not until the early 1930s that the works of CR Burch on diffusion pump oil made it possible for this process to be used satisfactorily. Since the tremendous trades have been made particularly in last few years, filters with greater than 100 layers are not uncommon and uses have been found for them in almost every branch of science and technology.

Much research has been done on the mechanism of thin film growth with evaporated films. It has been found by observation of films evaporated directly in the viewing field of an electron microscope that film growth may be divided into certain stages. Pashley et al. distinguished four stages of growth process: nucleation and island structure, coalescence of islands, channel filling, and formation of continuous film. Nucleation is the initial stage of a film. The particles which have been evaporated from the evaporation source and have reached the substrate, on which a thin film is to deposit, generally lose part of their energy on impingement. Therefore, the mobility of the atoms or molecules on the surface decreases as the atoms give up their energy to the substrate. The effect of elevated substrate temperature is to permit the atoms to retain sufficient energy to make the movements necessary for the accommodation on the substrate and among them. Most of the flux arrives at the substrate in atomic form. These ad-atoms diffuse around the substrate and their diffusivities are dependent on the interaction between the ad-atoms (adsorbed atoms) and the substrate and the temperature of the substrate. Any defects or crystallographic variations on the substrate surface acts as a potential well and the atoms have to overcome this potential barrier to keep moving around. Occasionally, they succeed so well that they are re-evaporated. In diffusing randomly they come across other atoms and join them to form doublets, which have lower diffusivities. Beyond a critical nucleus size (on the order of 10–100 Å), the larger nuclei grow at the expense of the smaller ones, and so the number of nuclei on the substrate is continually decreasing during growth. The initial nucleation is enhanced by the presence of defects on the substrate. At this stage the individual crystallites (about 100 Å in diameter) are quite perfect structurally. As more flux arrives at the surface, the nuclei sizes grow and eventually islands are formed. During this stage of film growth some islands come into mutual contact and coalescence ensues. The coalescence phase is critical for the formation of grain boundaries and dislocations. As the larger nuclei (perhaps several thousand atoms in size) combine, the amount of disorder at the merging boundary depends on the orientation of each nucleus before contact. If the nuclei are aligned (as in epitaxial growth on a well-matched substrate lattice), either a twin boundary or none at all is formed. If the islands are not aligned before contact (as in growth on an amorphous substrate or growth at low temperature), a grain boundary is formed. As the nuclei become large, the energy of the aggregate becomes smaller so the larger nuclei have less ability to hold to each other as they combine.

It is believed that certain energy is liberated by coalescence, which is sufficient to affect a temporary melting of the crystallites in contact. After coalescence, the temperature drops and newborn island occurs again. It has been established that when two islands which are of different sizes and crystallographic orientation coalescence, the resultant crystallite assumes the orientation of larger one. As the islands grow, there is a decreasing tendency for them to become completely rounded after coalescence. Large shape changes still occur, but these are confined mainly to the regions in the immediate vicinity of junction of the islands. Consequently, the islands become elongated and join to form a continuous network structure in which the deposit material is separated by long, irregular, and narrow channels of width 50–200 Å. As deposition continues, secondary nucleation occurs in these channels, and the nuclei are incorporated into the bulk of the film as they grow and touch the sides of the channel. At the same time, channels are bridged at some points and fill in rapidly in liquid like manner. Eventually, most of the channels are eliminated and the film is continuous but contains many small irregular holes. Secondary nucleation takes place on substrate with in holes,

which are produced during channel filling stage. The hole contains many secondary nuclei which coalesce with each other to form secondary islands which then touch edge of hole and coalesce with the main film to leave a clean hole. Further, secondary nuclei then form, and the process is repeated until the hole finally fills. The liquid like behavior of the deposit persists until a complete film is obtained. These processes are substantially complete before appreciable growth in thickness occurs.

3. Deposition techniques for thin films

The process by which thin film is deposited onto a substrate or onto a previously deposited layer is called thin film deposition. The process will be followed according to the requirements and economic conditions. There are some major techniques used for thin film fabrication such as physical vapor deposition (PVD), closed space sublimation (CSS), chemical vapor deposition (CVD), chemical bath deposition (CBD), pulsed laser deposition (PLD), electro-deposition (ED), sputtering technique, and atomic layer epitaxy (ALE) [16-18]. So many techniques can be used for the CdTe. It is the versatility of the CdTe because it provides many ways for its deposition. The method of deposition should be economical, easily scalable, and easy to handle and can give good conversion efficiency of the device. The method should be easily applicable at large industrial level. The deposition technique will be preferred which have maximum conversion efficiency, low cost, and high deposition rate. Many techniques have efficiency up to 12% in laboratory but not at the industrial level. There should be such kind of deposition techniques that will give good conversion efficiency in laboratory as well as at industrial level [10].

3.1. Physical vapor deposition (PVD)

It is the process in which material is sublimated from solid or liquid source and condensed upon the substrate; mostly the whole process is done in a vacuum. The deposition rate for physical vapor deposition is approximately from 1 to 10 nm per second. This deposition technique is used for the deposition of alloys, elements, and compounds by the reactive deposition. In reactive deposition, a gas environment is used which has a reaction with the source material (depositing material) to form a compound, the gas environment may be a nitrogen gas or some other gas.

The physical vapor deposition can be categorized as:

(i) vacuum evaporation, (ii) sputter deposition, and (iii) molecular beam epitaxy.

Physical vapor deposition is a technique whereby physical processes, such as evaporation, sublimation or ionic impingement on a target, facilitate the transfer of atoms from a solid or molten source onto a substrate. Evaporation and sputtering are the two most widely used processes and PVD method is used for depositing films. Important factors in controlling the structure of a growing film are growth flux or deposition rate, substrate temperature, source temperature, and evaporation time.

The ratio of the substrate temperature to the melting temperature of the film material is an important factor in determining the structure of a polycrystalline film. These factors determine the degree to which ad-atoms are able to seek out minimum energy positions and grain boundaries are able to adopt morphologies of minimum energy. The driving force for grain growth is the reduction in the total grain boundary surface area and the attendant reduction in the total energy associated with grain boundary surfaces. Another mechanism for the development of structure in polycrystalline films involves recrystallization, the driving force for which is the minimization of energy associated with defects, such as pre-existing dislocations, in addition to grain boundary energy. Minimization of stored elastic energy arising from intrinsic and mismatch strains in the film can also serve as an additional factor contributing to grain growth and recrystallization.

The factors that control the very early stages of growth of a thin film on a substrate are described in atomistic terms. The process begins with a clean surface of the substrate material, which is at substrate temperature Ts, exposed to a vapor of a chemically compatible film material, which is at the vapor temperature TV.

To form a single crystal film, atoms of the film material in the vapor must arrive at the substrate surface, adhere to it, and settle into possible equilibrium positions before structural defects are left behind the growth front.

To form an amorphous film, on the other hand, atoms must be prevented from seeking stable equilibrium positions once they arrive at the growth surface. In either case, this must happen in more or less the same way over a very large area of the substrate surface for the structure to develop. At first sight, this outcome might seem unlikely, but such films are produced routinely.

Atoms in the vapor come into contact with the substrate surface where they form chemical bonds with atoms in the substrate. The temperature of the substrate must be low enough so that the vapor phase is supersaturated in some sense with respect to the substrate, an idea that will be made more concrete below. There is a reduction in energy due to formation of the bonds during attachment. Some fraction of the attached atoms, which are called ad-atoms, may return to the vapor by evaporation if their energies due to thermal fluctuations are sufficient to occasionally overcome the energy of attachment. High energy ad-atoms stick on the growth surface where they arrive, and the film tends to grow with an amorphous or very finegrained polycrystalline structure. The growth surface invariably has some distribution of surface defects crystallographic steps, grain boundary traces, and dislocation line terminations, for example which provide sites of relatively easy attachment for ad-atoms. For semiconductor films, the epitaxial structure is essential to the electronic performance of the material. For metal films, on the other hand, the electrical conductivity of a polycrystalline film is nearly as large as that of a single crystal film.

In the vacuum evaporation process, source material is evaporated thermally inside the vacuum and its vapors are condensed at the substrate. The vacuum has a benefit of controlling the contaminations and reducing the melting temperature of the source material. Vacuum also increases the mean free path for the motion of deposited species. The vacuum required for deposition is from 10^{-5} to 10^{-10} Torr. Thermal evaporation is obtained by thermal heating

sources such as tungsten coils. The vacuum evaporation technique is used for fabrication of decorative coating, corrosion protective coatings, and electrically conducting films [13]. The sputter deposition is done by physical sputtering. It is the process in which material is not heated thermally so it is a non-vaporizing process. Material from target is ejected and then deposits onto a substrate. When energetic particles like ions are bombarded on the sputtering target, a plume of material is released and deposits onto a substrate just like a shower of sand when a golf ball lands in the bunker. The bombarding particles are usually gaseous ions accelerated from plasma. The sputtering gas is often an inert gas like argon gas. The spacing between source and substrate is less than vacuum deposition.

The plasma pressure for sputter deposition is from 5 to 30 mTorr. The sputtered particles are thermalized by gas phase collision before they reach substrate surface. The ions are generated from local plasma (diode or planar magnetron sputtering) or a separate ion beam source (ion beam deposition) [13, 14], as shown in **Figure 1**.

In molecular beam epitaxial (MBE) growth, the fabrication of crystalline thin film can be grown epitaxial above the other crystalline substrate with the beam of molecules or atoms.

This method required an ultra-high vacuum up to (10⁻⁸) pa for the film deposition. The rate of deposition is very slow which is 1 µm/h. The materials are then evaporated and reach the substrate individually on the wafer and reaction takes place between these vapors. This method can give high-purity epitaxial layers of compound semiconductors. The word "beam" shows that sublimated atoms of the material do not interact with each other and with vacuum chamber until reach the wafer, due to long mean free path of atoms. The crystalline film is fabricated on the substrate of the same material which is called as homoepitaxy. So this epitaxy is done with only single material. Heteroepitaxy is performed with different materials. In heteroepitaxy, crystalline thin film is fabricated on the substrate of a different material. The disadvantage of this method is that it is very expensive. In MBE technique; vapor phase, liquid phase, and solid phase methods can be used [16].

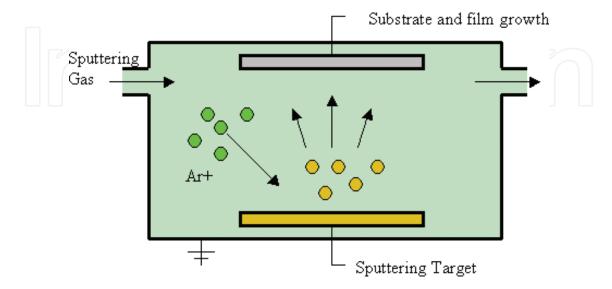


Figure 1. Sputtering apparatus.

3.2. Close spaced sublimation (CSS) technique for thin film deposition

Close space sublimation is a type of thermal evaporation technique. Advantage of CSS process is simplified deposition and high transport efficiency conducted under low vacuum conditions at moderate temperatures. In CSS technique, desired source material is placed in powder form in a graphite boat which is being heated by halogen lamps [5]. The substrate is placed in a mica sheet, which acts as a thermal gradient between the source and the substrate. The material starts to sublimate and deposit on substrate. The source is maintained at higher temperature than substrate. The deposited film presents a high crystallographic orientation and adequate opto-electrical properties for photovoltaic applications.

The CSS is a process for a thin film deposition of materials in a vacuum as shown in **Figure 2**. The material is sublimated by heating and its vapors condensed onto a substrate which is placed above the source material. The basic phenomenon of thin film deposition based on dissociation at high temperature.

Before the fabrication of thin film by close spaced sublimation, the substrate needs a proper cleaning by these methods like acetone, isopropyl alcohol, a rinse with distilled water, and ultrasonic cleaning.

The quality of thin films, material transport and deposition rate depends upon the fabrication parameters [17, 18].

It is observed that high substrate temperature of CdTe provides good performance of solar cell devices. Resistivity of CdTe decreases by increasing the substrate temperature and grain size reduces by increasing substrate temperature. Deposition rate also improved at high substrate

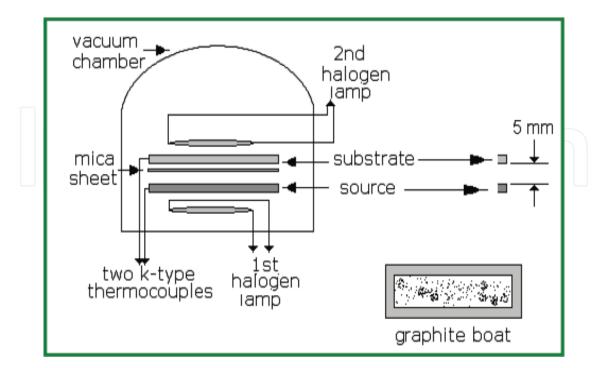


Figure 2. Schematic diagram of CSS designed at CIIT.

temperature [19]. The pacing between source and substrate is inversely proportional to the rate of deposition. Vacuum level for CdS and CdTe deposition is from 10⁻³ to 10⁻⁵ mbar.

Annealing of the films provides improvement of surface morphology and it reduces roughness of surface of CdS and CdTe films. Recombination centers reduce by annealing. The crystallinity of film also improves by the annealing process. Open circuit voltage also increased by increasing annealing temperature. Spectral response especially in the range of 500-600 nm is also improved by annealing temperature. Hence efficiency of solar cell can be improved [20–22].

The particles movement from higher concentration to lower concentration is called diffusion. The distance particles can travel without any collision is called mean free path. Diffusion of one kind of particles into other kind of material can change its characteristics like a semiconductor material can be converted into an n-type or a p-type. The advantages of CSS are as follows: (1) the evaporation source and substrate are heated directly by halogen lambs and their temperature is controlled using temperature controllers; (2) the source and substrate are separated by a mica sheet of about 1–3 mm. In this way, the source vapors are confined to closed space, leading to less wastage of evaporated material as compared to other methods. The mica sheet maintains the source and substrate at different temperatures, due to which the evaporating materials will always have better access to the substrate; (3) the films deposited by this method present a high crystallographic orientation and adequate opto-electrical properties for photo voltaic applications; (4) the system is very simple and easy to use; and (5) it has high transport efficiency conducted under low vacuum conditions at moderate temperature. There are also some limitations: (1) the main limitation of CSS deposition system is that there is no quartz crystal to monitor the growth rate and thickness of the film deposited; and (2) this method can only be used for limited number of materials, i.e., materials which can be sublimated at moderate temperatures.

Thermal evaporation method for preparing thin films is becoming very popular since 30 years or so. In this method the pressure of gas in chamber is reduced to value as low as possible, this is called creating vacuum. Generating vacuum properly is very important because when evaporation is performed in poor vacuum or close to the atmospheric pressure, the resulting deposition is generally non-uniform. The purity of deposited films also depends on the quality of vacuum and on the purity of source material. Vacuum is created by different pumps for example we have rotary vane pump and oil diffusion pump. In our vacuum coating unit, these pumps create different vacuum ranges, 10⁻³ to 10⁻⁶ mbar. In this vacuum chamber, source material is evaporated by heating at suitable temperature for particular time. In this method one must make sure that in order to deposit a material, the evaporation system must be able to sublimate it and also pressure of gas in chamber is low enough so that mean free path of the atoms of evaporated material is larger than source-substrate distance.

3.3. Chemical vapor deposition (CVD)

CVD is a chemical process used to produce high-purity and high-performance solid materials. The process is often used in the semiconductor industry to produce thin films. In a typical CVD process, the wafer (substrate) is exposed to one or more volatile precursors, which react and/or decompose on the substrate surface to produce the desired deposit. Frequently, volatile by-products are also produced, which are removed by gas flow through the reaction chamber. Micro fabrication processes widely use CVD to deposit materials in various forms, including: nanocrystalline, polycrystalline, amorphous, and epitaxial. These materials include: silicon, carbon fiber, carbon nanofibers, filaments, carbon nanotubes, SiO₂, silicon-germanium, tungsten, silicon carbide, silicon nitride, silicon oxynitride, titanium nitride, and various high-k dielectrics. The CVD process is also used to produce synthetic diamonds. It is a chemical reaction which transforms gas molecules into the solid material and then into thin film or powder form, on the substrate surface. CVD is widely used to fabricate semiconductor devices.

Atomic layer epitaxy (ALE), or atomic layer chemical vapor deposition (ALCVD), now a days is known as atomic layer deposition (ALD) technique used for the production of high quality and thin solid films of specific crystal structures or orientations. This method provides very fine control of film thicknesses to one atomic layer. It has a wide range of applications in areas such as thin film ceramics, gas sensors, radiation detectors, optical/infrared filters, surface hardening, and fiber optical materials. The term *epitaxy* comes from the Greek roots *epi*, meaning "above", and taxis, meaning "in ordered manner". It can be translated "to arrange upon". It is a method that is based on sequential use of gas phase chemical process. Mostly in (ALE) two chemicals are used called precursors. Chemical reaction takes place between these precursors with surface one at a time in sequential manner. Using ALE alternating monolayers of two different elements can be deposited onto a substrate. In this technique material amount deposited in each cycle is constant. This method provides crystalline and uniform films especially if very thin film is required. In the laboratory of advanced energy systems, ALE used to grow both CdS and CdTe layers in a single process. ALE reactor with four individually controlled sources to control inside solid reactants, and has number of sources for external inert gas flow and liquid reactants. The spacing between two substrates is 1–3 mm.

The deposition of a substance on an electrode by the process of electrolyses is another CVD method. It is a technique in which electric current is passed through a chemical solution and ionization occurs then these ions deposit on the substrate.

Electrolytic deposition-cathodic film is also a versatile method of depositing film on an electrode (cathode) of the cell in which electrode is placed in a solution and the ions in that solution are impelled to the electrodes by an electric anodization was used as electrolyte in the middle of nineteenth century. A few years later, sulfuric acid bath was used in same process. This technique can be related to that of electrolytic deposition method. Same apparatus is used for both, but in this case, the thin film is formed at the anode or positive electrode rather than at cathode.

4. Fabrication of CdS thin film by chemical bath deposition (CBD) and (CSS) techniques

A II–VI semiconductor compound cadmium sulfide (CdS) has much importance because of its so many applications in several heterojunction photovoltaic thin film devices of CdTe, copper indium gallium diselenide (Cu(In $_x$ Ga $_{1-x}$)Se $_2$), or copper Indium Gallium sulfide (CIGS), and solar cells. Many other devices in the fields of electronics, optics, and infrared are also fabricated by using CdS [1–3]. The CdS is a suitable n-type material, can be fabricated by a

variety of fabrication techniques like sol-gel technique [4], close spaced sublimation (CSS), chemical bath deposition (CBD), thermal evaporation, chemical vapor deposition, molecular beam epitaxy and spray pyrolysis. Each and every deposition process provides different optical, structural, electrical, and morphological properties [5–10].

First, solar cell has reported CdS deposited by high rate vapor transport deposition (HRVTD). CdS is called window layer due to its higher rate of light transmission [11–14]. CdS thin films are very suitable for so many other semiconductor devices and radiation detectors. CdS thin films with wide band gap are highly used for photovoltaic devices. Since CdS used as window layer in solar cell so it should be fabricated very thin, high amount of light should pass through CdS and absorb in CdTe. The CdS thin film must be continuous to reduce the effect of short circuit in the cell. The CdS thin films deposited by CBD can fulfill these requirements. So the thickness of CdS has much importance for the high efficiency of solar cell [15–17]. CdS (CBD) thin films are grown by cadmium chloride, ammonium nitrate, and potassium hydroxide. After heating to a specific temperature, thiourea is added to start the fabrication process [18–20]. CBD is very easy process for the fabrication of CdS thin films on ITO glass. For the fabrication of thin film solar cell, one needs a very thin film up to (60-80) nm. It is very suitable process especially from solar cell point of view. It is a process in which substrate is placed in a hot chemical solution stirring vigorously for specific time, positive and negative ions will reach and meet on the substrate and thin film is grown. The advantage of this technique is that neither vacuum and nor very high temperature is required for CBD [5, 21].

Thin films of CdS were deposited on microscope glass sides and ITO glass sides. Two fabrication procedures were used for CdS deposition for the first time in our lab. Firstly, CdS thin films were fabricated by CBD technique. For fabrication of thin film for solar cells, we need a very thin film up to (60-80) nm. In this process substrate was placed in a hot chemical solution stirring vigorously for 10-30 minutes with magnetic stirring at 3 Hz at 75°C by combining positive and negative ions on the substrate thin film was grown. The advantage was that neither vacuum nor very high temperature is required for CBD. The chemical solutions of (0.02 M) about 80 ml solution of cadmium chloride (CdCl₂), (1.5 M) about 80 ml solution of ammonium nitrate (NH₄NO₃), and (0.5 M) about 200 ml solution of potassium hydroxide (KOH) were made in a beaker. When temperature reached at 75°C, thiourea of (0.2 M) about 80 ml solution was added to start fabrication. The films were fabricated with magnetic stirring of 3 Hz for 10-30 min at 75°C as described in [5]. It is a process in which substrate is placed in a hot chemical solution stirring vigorously for specific time, positive and negative ions will reach and meet on the substrate and thin film is grown. The advantage of this technique is that neither vacuum and nor very high temperature is required for CBD [24]. Substrate was cleaned by using detergent then rinsed in distilled water then again clean with acetone and rinsed in distilled water then clean with isopropyl alcohol (IPA).

These three solutions are added to the beaker. The substrates are fixed inside the solution with the help of a substrate holder. The beaker is placed on the hot magnetic plate with magnetic stirrer inside. A pH meter and thermometer is also dipped in the solution to measure pH and temperature, respectively. Provide heat to solution up to temperature of 75°C. The solution is provided by stirring continuously throughout the experiment.

When temperature reached at 75°C add the thiourea about 0.2 M (80 ml).

As thiourea is added to the solution, the reaction starts suddenly. So thiourea is the last component which is added to the solution. The CdS deposition starts when thiourea is added. Use stop watch now and substrates will be taken out after the 10, 20, and 30 min, respectively.

Films are retired from the solution according to the specific deposition time and rinsed immediately with distilled water into an ultrasonic cleaner. The CdS deposited films with pale yellow color are obtained. The CdS film is deposited on both sides of the ITO glass. So film from glass side is cleaned by using 10% hydrochloric acid (HCl) solution. It needs much care when using HCl acid on glass side for cleaning because drops of HCl can remove film from ITO sides as well. After deposition, these CdS films will be annealed at 400°C for 30 min [25–27].

CdS thin films fabricated by the CSS are also another moderate temperature fabrication technique in a vacuum chamber. More than 20% efficiency has been achieved by using CdTe/CdS heterojunction thin film solar cell by this technique. CSS is moderate temperature procedure so it provides in some cases better results [22, 23]. The CSS technique is one of the techniques that have produces encouraging results [15] mainly because it is simple deposition apparatus and high transport efficiency and deposition can be done in low vacuum at moderate temperatures. There is minimum use of material in the CSS system as compared to other methods. As the substrate is close to the source materials in the CSS technique, roughness increases in the thin films and has high absorption, which makes it suitable material for solar cell applications. The CSS has a disadvantage that one cannot introduce thickness monitor.

CdS (CSS) fabrication was carried out in a vacuum chamber of approximately 4×10^{-1} mbar. The CdS sigma Aldrich powder 99.999% pure was used. Substrate temperature was maintained at 400°C and source temperature was in between (500-600)°C. Source material was heated by 1000 W halogen lamp and substrate was heated by a 500 W halogen lamp. Time of deposition was about (3–5) min as shown in Figure 2. The film was fabricated after collected after cooling. It is very easy process for the fabrication of CdS thin films on ITO glass. The disadvantage of closed spaced sublimation is that thickness cannot be controlled. For the fabrication of thin film solar cell we need a very thin film up to 60–80 nm, which is not easily possible by using close spaced sublimation process. It is very suitable process especially from solar cell point of view. CdS thin films were deposited on ITO glass substrate by CBD technique as shown in Figure 3 using CdCl₂, ammonium nitrate (NH₄NO₃), potassium hydroxide (KOH), and thiourea as starting materials. ITO coated glass was used as available from sigma-Aldrich. Deposition was carried out with magnetic stirring at 3 Hz for 30 min at 75°C. The resulting CdS layer was annealed at 400° C for 30 min in a vacuum chamber at 4×10^{-2} mbar, and its thickness was found to be 800 nm. The CdTe layer was deposited on CdS by CSS technique under vacuum of 4×10^{-1} mbar. Deposition time was kept as 3–5 min. The substrate and source temperature were maintained at 400 and 530°C, respectively. A 500 W halogen lamp was used to heat the CdS/ITO/glass substrate, whereas a graphite boat was used to heat the source material with a 1000 W halogen lamp. The source and substrate spacing was measured as 4-5 mm. CdTe film thickness was measured as 5 μm. To enhance the p-type properties of CdTe, tellurium (Te) layer (400 nm) was deposited on CdTe by CSS technique under vacuum of 4 × 10⁻² mbar. Deposition time was kept as 5 min, whereas, source and substrate temperatures were kept as 350 and 200°C, respectively. Resulting Te layer was annealed at 200°C for 1 h.

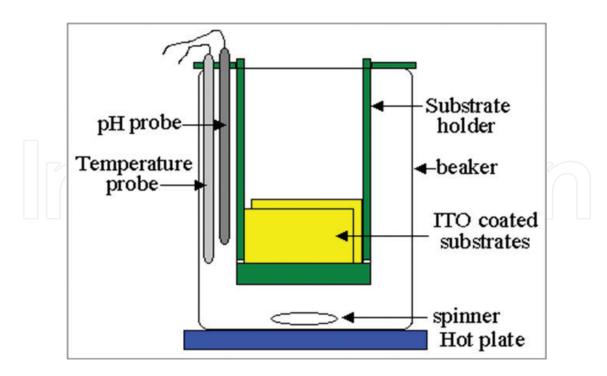


Figure 3. Chemical bath deposition apparatus.

1.04 g CdCl, was dissolved in 50 ml methanol at 65°C with constant magnetic stirring at 3 Hz for 10 min CdCl₂/methanol solution was then allowed to cool down at room temperature (25°C). The CdS/ITO/glass and the CdTe/CdS/ITO/glass structures were soaked in the CdCl₂ solution for 7 s. After drying at room temperature, the structures were annealed in a tube furnace for 30 min at 400°C with constant flow of argon gas (30 ml/min).

Transmission spectra, X-ray diffraction (XRD), and scanning electron microscope (SEM) with energy dispersive X-ray (EDX) investigations were carried out in order to understand the optical, crystallographic, and morphological effects of CdCl, treatment on CdTe and CdS films. Optical analysis using UV-VIS-NIR spectrophotometer was also used to study the thickness of films. Rutherford back scattering (RBS) analysis was used to identify the elemental composition of CdS thin films and further confirm their thickness. Rutherford back scattering spectrometry with an accuracy of 7% was used. High energy alpha particles were bombarded on the CdS film. Backscattered He²⁺ ion energy distribution and yield at a given angle were measured. Backscattered ions were detected by using a surface barrier detector with 17 keV resolution kept at a scattering angle of 160°. Since cross section of backscattering for each element is known, it is possible to get a quantitative compositional analysis from the RBS spectrum. Silver (Ag) paste was used for ohmic contact formation in the ITO/CdS/CdTe solar cells structure.

5. Comparative analysis of optical properties of thin films

(UV-VIS-IR) spectrophotometer was used to study optical properties of CdS (CBD) and CdS (CSS) thin films. The transmission for (CBD) films starts after 300 nm wavelength but in case of (CSS) transmission starts after 500 nm. So in CdS (CSS), blue portion of light was absorbed. Transmission increased especially above 550 nm wavelength region. In the region of 600–800 nm the transmission was more than 70% for both (CBD) and (CSS), so light transmission through (CBD) films was higher than (CSS). Due to this high transmission characteristic which showed that CdS a good window layer for the thin film solar cells of many kinds, fabricated by both of the techniques. The Swanepoel model provided calculations about thickness and refractive index. Energy values were calculated by plotting a graph between energy and $(\alpha hv)^2$. Formula for refractive index (n) is given in Eqs. (1)–(3).

$$d = \frac{M \lambda_{\text{max}} \lambda_{\text{min}}}{4n(\lambda_{\text{max}} - \lambda_{\text{min}})} \tag{1}$$

where M is the number of oscillations between maximum and minimum transmission wavelengths λ_{\max} and λ_{\min} respectively. The obtained thickness of CdTe films was 5 μ m. n can be calculated using the relation:

$$n = \frac{\left[N + (N^2 - 4s^2)^{\frac{1}{2}}\right]}{2} \tag{2}$$

Where,
$$N = 1 + s^2 + 4s \left(\frac{T_{\rm M} - T_{\rm m}}{T_{\rm M} T_{\rm m}} \right)$$
 (3)

Here, the refractive index of glass, s = 1.52, T_{max} and T_{min} are the maximum and minimum transmissions, respectively. The values of d and n are calculated from Eqs. (1) and (2). The optical properties demonstrate a slight increase in absorption in IR region. Consequently, the transmittance is seen to slightly decrease after $CdCl_2$ heat-treatment. The band gap can be determined using the following relation:

$$\alpha h v = A (h v - E_g)^{N/2} \tag{4}$$

Here A is a constant, hv is the photon energy, E_g is the optical energy band gap. N depends on the nature of the transition (N = 1 for direct band gap, while N = 4 for indirect band gap transition). hv can be calculated by

$$hv(eV) = 1.24/\lambda(\mu m)$$
 (5)

The band gap can be obtained by extrapolating $(\alpha hv)^2$ versus the incident photon energy (hv) plot. CdCl₂ heat-treatment results showed a slight decrease in band gap values as well. The band gap values extracted from these plots are 1.50 eV for as-deposited and 1.49 eV for CdCl₂ heat-treated sample.

5.1. Optical properties of CdTe by closed space sublimation (CSS)

The optical measurements using the spectrophotometer can provide the information about the transmittance, thickness, refractive index, absorption coefficient, and energy band gap. The as-deposited CdTe films have a high absorption in the visible and near-infrared regions, which did not change significantly after the CdCl treatment. The transmission in the as-deposited sample is about 80% at higher wavelength. The same is improved to above 85% in the heat-treated sample while the CdCl2 treated sample has about 80% transmission in higher wavelength region as shown in **Figure 4**.

The crystallographic orientation of CdTe samples was investigated by X-ray diffraction. The main reflections of the samples are the same and can be indexed according to fcc CdTe lattice.

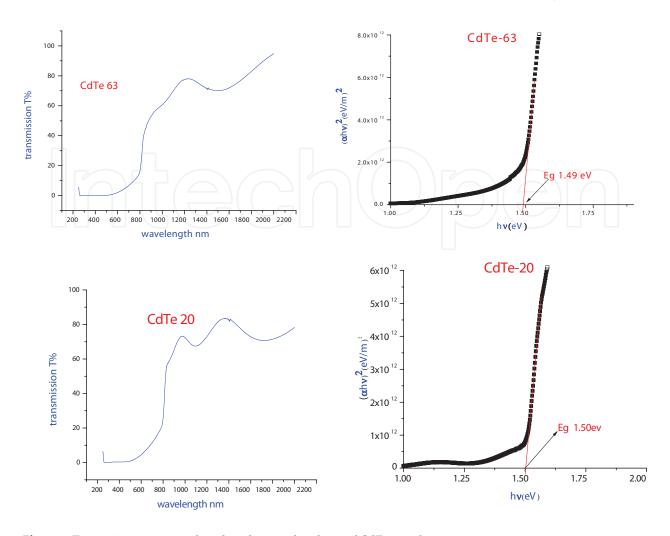


Figure 4. Transmittance vs. wavelength and energy band gap of CdTe samples.

The data analysis gave the lattice constant as 6.395 Å, which agreed with the reported value of 6.410 Å for the as-deposited and $CdCl_2$ lattice constant (ASTM Cards 15-0770, 75-2086). Two values of the lattice constant are attributed to the recrystallized lattice. The strongest (1 1 1) reflection in the as-deposited sample indicates that a preferential orientation of (1 1 1) matches well with the observation earlier reported. The loss in the texture of CdTe is exhibited in $CdCl_2$ -treated sample. However, the intensity of the (1 1 1) peak is lower in $CdCl_2$ -treated sample CdTe-63, implying that the samples are losing (1 1 1) texture and at the same time reorienting themselves in the (2 2 0) direction. The structure of CdTe is Cubic. The nature of deposited film is polycrystalline as shown in **Figure 5**.

5.2. Scan electron microscopy (SEM) of CdTe samples CdTe-20 and CdTe-63, CdTe-20 and CdTe-63

The morphology of the as-deposited heat-treated and $CdCl_2$ -treated samples CdTe-20 and CdTe-63 show the change in the shape and size of the CdTe grains as in **Figure 6**. The average grain size of the as-deposited sample is under 01.7 μ m and 0.86 μ m in the heat-treated sample respectively while some of the bigger grains divide into smaller grains and reorient themselves, which results into an entirely different microstructure. The SEM images support the XRD results.

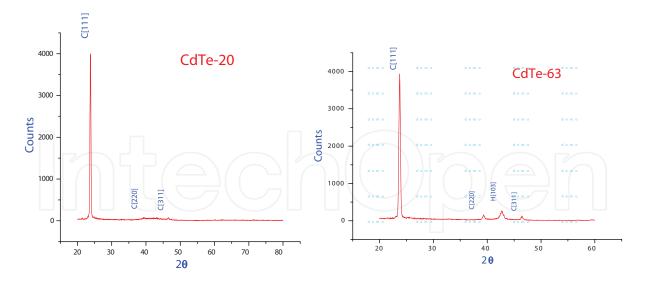


Figure 5. X-ray diffraction spectrum for CdTe samples.

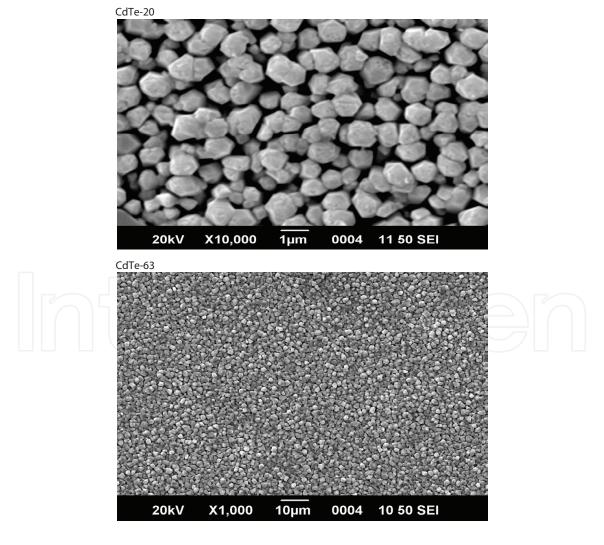


Figure 6. SEM micrographs of CdTe-20 and CdTe-63 samples.

5.3. Optical analysis of CdS thin film by chemical bath deposition

The CdS layer has good transmittance especially above 550 nm wavelength region as shown in **Figure 7**. Transmission starts after 300 nm by CBD process. In the region of 600–800 nm, the transmission is more than 70% in samples CdS 21 and CdS/CBD 23. This is the characteristic which shows that CdS is good window layer for the thin film solar cells of many kinds.

The band gap can be obtained by extrapolating $(\alpha hv)^2$ versus the incident photon energy (hv) plot. CdS 23 and CdS 24 samples fabricated by chemical bath deposition results showed a slight decrease in band gap values as well. The band gap values extracted from these plots are 2.36 eV and 2.33 eV.

5.4. Structural analysis of CdS by chemical bath deposition (CBD)

Structural properties were studied by X-ray diffraction using $CuK\alpha$ radiations of 1.5418 Å. The data analysis provided lattice constant as 6.50 Å for as-deposited sample. The lattice parameter can be calculated by using the relation for only cubic structure.

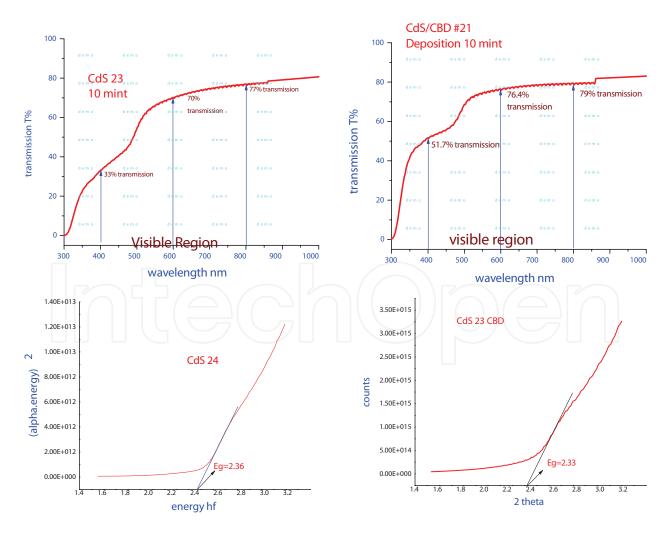


Figure 7. Transmittance vs. wavelength and energy band gap of CdS samples.

$$a = d (h^2 + k^{2+} l^2)^{1/2}$$
 (6)

where h, k, and l are Miller indices, for hexagonal structure formula for "d" is

$$1/d^2 = 4/3((h^2 + hk + k^2) + \frac{l^2}{c^2}$$
 (7)

By using the Scherer formula, crystalline size can be calculated

Crystalline size =
$$\frac{0.9\lambda}{\beta\cos\theta}$$
 (8)

 λ is X-rays wavelength, 0.9 is a constant shape factor, β is full width half maxima in radians, θ is Bragg's angle [25]. Peaks relating to CdS can be identified by using standard card JCPDS-00-041-1049. The d-spacing values were compared with standard values of ASTM cards to find the structure. It was observed in this experimental work that CdS thin films had a mixed structure of H (0 0 2) and C (1 1 1) for both techniques. So CdS has a polycrystalline behavior [20–22, 30–39]. Preferred orientation is (0 0 2) and structure is hexagonal as shown in **Figure 8**.

5.5. Optical analysis of CdS thin film by close sublimation technique (CSS)

The CdS deposited by close spaced sublimation have transmission for light above 550 nm then it again decreases for infrared region as shown in **Figure 9**. So if we consider only visible spectrum as for the solar cell point of view then it can be observed that from 600 to 800 nm, the transmission is more than 60% which shows that CdS is a good and efficient window layer for the visible spectrum or solar spectrum exclusively. The energy band gap is also showing the 2.44 and 2.38 eV for samples CdS 13 and CdS 318, respectively.

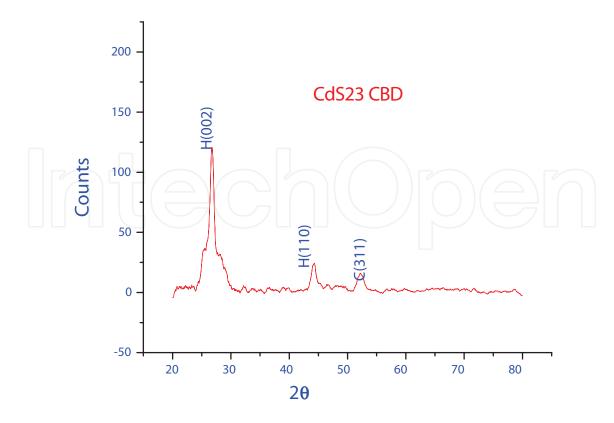


Figure 8. X-ray diffraction spectrum for CdTe samples.

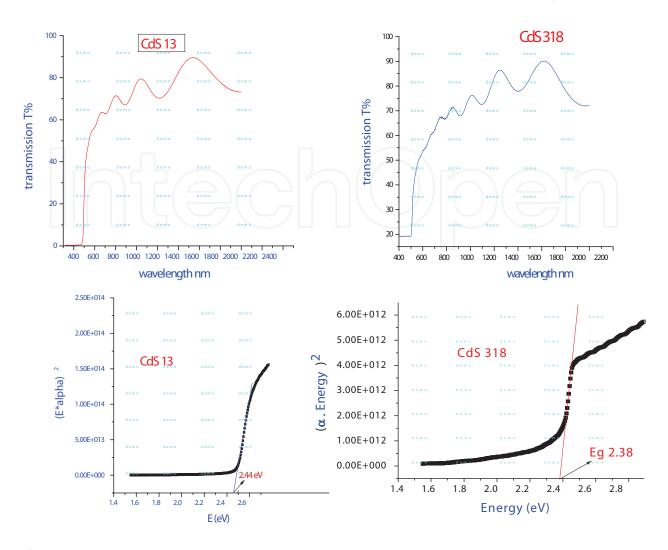


Figure 9. Transmittance vs. wavelength and energy band gap of CdS samples.

5.6. Structural analysis of CdS by close sublimation technique (CSS)

The XRD spectra were taken scanning the values of 2θ from 20 to 80° as shown in **Figure 10**. In CdS (CSS) high intensity planes were grown during the film growth due to high temperature. In CdS (CSS) strong peaks C (111), and H(002) were for two samples 317 and 318 as observed, which proved the polycrystalline behavior of CdS thin films. The height of peak/intensity for CdS (CBD) was only up to 200, 150, and 400, first strong peak was H (002) for two samples and C (111) for third sample at 2θ value of (26.49, 25.19, 26.74) degree of angle. In our research work for both CBD and CSS techniques, first hexagonal strong peak was dominant. The size of grains was different for different fabrication techniques this may be due to film thickness, temperature or different nucleation of CdS for different deposition methods.

5.7. SEM analysis of CdS (CBD) and CdS (CSS)

Surface morphology was studied by using scanning electron microscopy (SEM), and grain size for CdS (CSS) was measured to be 300–400 nm as shown in **Figure 11**.

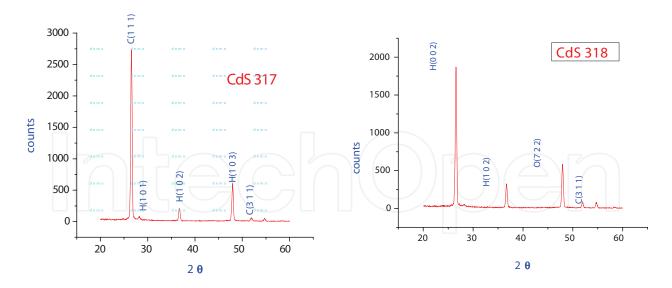


Figure 10. X-ray diffraction spectrum for CdS samples.

The surface of CBD films was slightly nonuniform but CdS (CSS) structure was fine with better crystallinity. SEM analysis showed that CBD-CdS thin films grain size approximately was found to be (50–100) nm and CSS-CdS films grain size was approximately (200–300) nm. It was also observed that at high source temperature, grain size was bigger in the CSS technique. It is reported in the literature that even less crystalline and non-uniform films of CBD process gave high-efficiency as compared to CSS [5, 40–42].

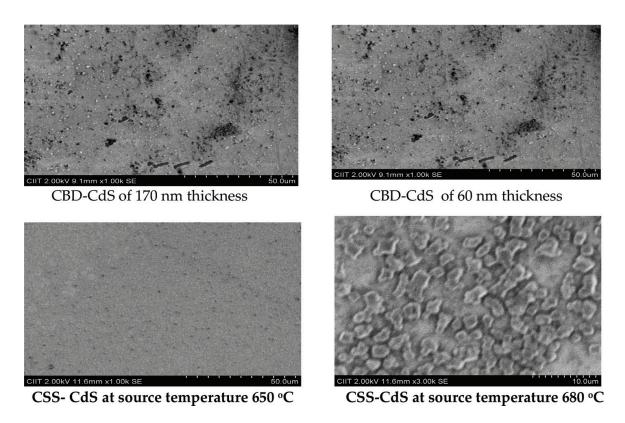


Figure 11. SEM micrographs of CBD-CdS and CSS-CdS samples.

The transmission spectra of ITO coated thin films and ITO/CdS films shows more than 90% light is passes in the visible as well as near infrared regions shown in **Figure 12**. Since a CdS thin film (100–200 nm) is used for solar cells, the CdS film is well suited as the TCS for CdTe solar cells.

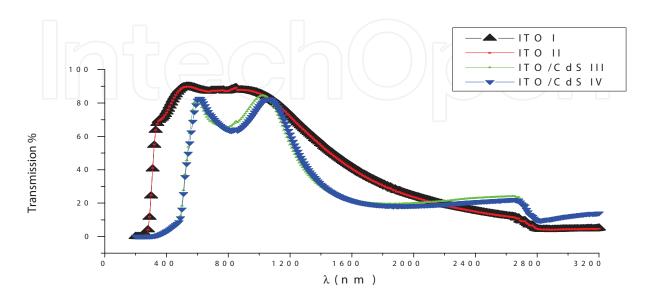


Figure 12. Transmission as a function of wavelength of ITO substrates and ITO/CdS thin films.

6. Conclusions

An ITO/CdTe/CdS/glass solar cell was successfully prepared using CSS and CBD techniques. Optical analysis of ITO demonstrated transmittance of over 85% for whole wavelength. The effects of CdCl, immersion and heating on the optical, structural, and morphological properties of CdTe and CdS surfaces were studied with an effort to promote recrystallization, reorientation, and progressive increase in grain size. Structural properties investigated by using XRD exhibited shift in 2θ diffraction angles of peaks, change in crystalline size, change in intensity of preferred orientation, and change in the total number of peaks, which indicates toward recrystallization and reorientation. The optical results after CdCl, heat-treatment showed a decrease in transmittance in the case of CdTe thin film and increase in the case of CdS thin film. Similarly, band gap values were also observed to decrease after the CdCl₂ heat-treatment. Surface morphology of CdTe thin films was affected by CdCl, heat-treatment as well. The SEM micrographs assisted in investigating the changes. A grain size of CdTe as-deposited sample was found to improve after the CdCl, heat-treatment. Similarly, a grain size of CdS as-deposited sample was found to improve after the CdCl, heat-treatment. In conclusion, the significant improvement to the CdTe/CdS films solar cell performance can be achieved when annealed at 400°C in the presence of CdCl, on the free surfaces of CdTe and CdS. These results can be used in turn to improve the short circuit current and open circuit voltage of solar cells.

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