Chapter 2

Deposition and Characterization Techniques for Thin Films
An understanding of the fundamental properties of the thin films at various stages of preparation is an important prerequisite for the production of device quality films and those properties depend strongly on synthesis conditions. Film properties rely on its thickness, composition and structure and on how the film interacts with its environment: light, electric and magnetic fields, chemicals, mechanical force, heat etc. The deposition technique employed for the growth of thin films and various characterisation tools used for the study of the films during deposition and after film formation is given in this chapter.
1 Introduction

The research in photovoltaics is focused on making solar cells cheaper and/or more efficient, so that they can more effectively compete with other energy sources. One way of doing this is to develop cheaper methods of obtaining sufficiently pure material that is apt for photovoltaic conversion. Another approach is to significantly reduce the amount of raw material used in the manufacture of solar cells. The various thin-film technologies currently being developed make use of this approach to reduce the cost of electricity from solar cells. Thin film solar cells use less than 1% of the raw material compared to wafer based solar cells, leading to a significant price drop per k Wh.

2.2 Thin Film Deposition

Thin-film deposition is any technique for depositing a thin film of material onto a substrate or onto previously deposited layers. Thin is a relative term, but most deposition techniques allow layer thickness to be controlled within a few tens of nanometres, and some like molecular beam epitaxy allows single layers of atoms to be deposited at a time.

Deposition techniques fall into two broad categories, depending on whether the process is primarily chemical or physical.

2.3 Chemical Deposition

Here, a fluid precursor undergoes a chemical change at a solid surface, leaving a solid layer. An everyday example is the formation of soot on a cool object when it is placed inside a flame. Since the fluid surrounds the solid object, deposition happens on every surface, with little regard to direction; thin films from chemical deposition techniques tend to be conformal, rather than directional.
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Chemical deposition is further categorized by the phase of the precursor. Plating relies on liquid precursors, often a solution of water with a salt of the metal to be deposited. The most commercially important process is electroplating. It was not commonly used in semiconductor processing for many years, but has seen revival with more widespread use of chemical-mechanical polishing techniques. Chemical vapor deposition (CVD) generally uses a gas-phase precursor, often a halide or hydride of the element to be deposited. In the case of metal organic chemical vapor deposition (MOCVD), an organometallic gas is used. Commercial techniques often use very low pressures of precursor gas. Plasma enhanced CVD uses an ionized vapor, or plasma, as a precursor.

2.4 Physical Deposition

Physical deposition uses mechanical or thermodynamic means to produce a thin film of solid. An everyday example is the formation of frost. Since most engineering materials are held together by relatively high energies, commercial physical deposition systems require a low-pressure vapor environment to function properly and most can be classified as physical vapor deposition.

2.4.1 Physical Vapor Deposition

Physical vapor deposition (PVD) is a technique used to deposit thin films of various materials onto various surfaces. The material to be deposited is placed in an energetic, entropic environment. A cooler surface is kept facing this source which draws energy from the particles those escape from the material surface, as they arrive, allowing them to form a solid layer. The whole system is kept in a vacuum deposition chamber, to allow the particles to travel as freely as possible. Since particles tend to follow a straight path, films deposited by physical means are commonly directional, rather than
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conformal. Physical vapor deposition methods are clean, dry vacuum deposition methods in which the coating is deposited over the entire object simultaneously, rather than in localized areas.

Various physical depositions include sputtering, pulsed laser deposition (PLD) and evaporation. Sputtering relies on a plasma to knock material from a target. Noble gases like argon are usually used for plasma. The target can be kept at a relatively low temperature, since the process is not one of evaporation. This makes sputtering one of the most flexible deposition techniques. It is especially useful for compounds or mixtures, where different components would otherwise tend to evaporate at different rates. PLD systems work by an ablation process. Pulses of focused laser light vaporize the surface of the target material and convert it to plasma; this plasma usually reverts to a gas before it reaches the substrate. Evaporation is a very simple and convenient method and is the most widely used technique. Sufficient amount of heat is given to the evaporant to attain the vapour pressure necessary for evaporation. Then the evaporated material is allowed to condense on a substrate kept at a suitable temperature.

The preparation of the precursor films described in this thesis work were performed by thermal evaporation by resistive heating,

2.5 Thermal Evaporation

Evaporation involves two basic processes: evaporation and condensation. A hot source material evaporates and condenses on the substrate.

Evaporation takes place in a vacuum. Vapours other than the source material are almost entirely removed before the process begins. In high vacuum with a long mean free path, evaporated particles can travel directly to the substrate without colliding with the background gas. At a typical pressure of \(10^{-4}\) Pa, a 0.4 nm particle has a mean free path of 60 m.
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Hot objects in the evaporation chamber, such as heating filaments, produce unwanted vapours that limit the quality of the vacuum. Evaporated atoms that collide with foreign particles may react with them; for example, if aluminium is deposited in the presence of oxygen, it will form aluminium oxide. They also reduce the amount of vapor that reaches the substrate.

Evaporated materials deposit non-uniformly if the substrate has a rough surface. Because the evaporated material falls on the substrate mostly from a single direction, protruding features block the evaporated material from some areas. This phenomenon is called shadowing or step coverage. When evaporation is performed in poor vacuum or close to atmospheric pressure, the resulting deposition is generally non-uniform and may not be continuous or smooth film. Rather, the deposition will appear fuzzy or cloudy. Only materials with a much higher vapor pressure than the heating element can be deposited without contamination of the film.

The evaporation system includes an energy source besides vacuum pump which evaporates the material to be deposited.

Many different energy sources exist. In the thermal method, the source material is placed in a crucible, which is radially heated by an electric filament, while in the electron-beam method, the source is heated by an electron beam with energy up to 15 keV. In flash evaporation, a fine wire of source material is fed continuously onto a hot ceramic bar, and evaporates on contact. Resistive evaporation is carried out by passing a large current through a wire or foil of the material that is to be deposited.

2.5.1 Comparison to Other Deposition Methods

Evaporation has a better step coverage than the other alternative methods, such as sputtering and chemical vapor deposition. This may be an advantage or disadvantage, depending on the desired result. Deposition by sputtering is
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slower than evaporation. Sputtering uses plasma, which produces many high-speed atoms that bombard the substrate and may damage it. Evaporated atoms have a Maxwellian energy distribution, determined by the temperature of the source, which reduces the number of high-speed atoms.

2.6 Other Deposition Processes

There are some other methods outside these two categories, based on a mixture of chemical and physical means:

- In reactive sputtering, a small amount of some non-noble gas such as oxygen or nitrogen is mixed with the plasma-forming gas. When material is sputtered from the target, it reacts with this gas and a different material is deposited on the substrate. i.e. an oxide or nitride of the target material.

- In topotaxy, a specialized technique similar to epitaxy, thin film crystal growth occurs in three dimensions due to the crystal structure similarities (either heterotopotaxy or homotopotaxy) between the substrate crystal and the growing thin film material.

- Two stage process, a cost effective technique consisting of the preparation of precursor film by any of the method like sputtering, thermal evaporation, followed by the chalcogenisation of these samples.

2.7 Two Stage Process

The major concerns related to the preparation of thin films in high production level are poor material utilization and the difficulty of obtaining uniform material fluxes over large area substrates. Attention will therefore be focused on the development of a relatively easily scalable two stage
deposition technique, to produce uniform coatings of thin films on large area substrates.

Two stage processes is a simple method for the preparation of chalcogenide and selenide thin films. This method has been effectively employed to produce high efficiency solar cells.

As the name indicates the two stage processes consists of two steps; 1) preparation of the precursor, 2) chalcogenisation of the precursor. Strength of two-stage approaches arises from the fact that they can utilize various deposition techniques (sputtering, thermal evaporation, screen printing and so on) in the precursor stage.

We have used the two stage processes to prepare copper indium selenide (CulnSe$_2$), copper indium sulfur selenide (Culn(Se$_{1-x}$S$_x$)$_2$), and indium sulfide thin films. During the first step of the two stage process, a metal film containing (Cu and In and/or Ga) or In only, are sequentially deposited, according to the final compound to be obtained are prepared by vacuum processes (thermal evaporation). During the second step of the process, the precursors are reacted with selenium or sulfur or in a mixture of sulfur and selenium atmosphere in order to form the ternary compound semiconductors. The final quality of the films formed depends on this chalcogenisation process as well as the structural properties of the precursor film before the chalcogenisation stage.

In our work of preparing CulnSe$_2$, copper and indium layers are deposited on glass substrates by thermal evaporation. The Cu-In bi-layer is annealed in vacuum to form the copper indium alloy (Cu$_{11}$In$_9$). This alloy was then selenized at various temperatures to form CulnSe$_2$ thin films. For post sulfurisation, the CulnSe$_2$ obtained as above were allowed to remain in sulfur atmosphere for some time. The Culn(Se$_{1-x}$S$_x$)$_2$ thin films were
prepared by annealing the Cu\textsubscript{11}In\textsubscript{9} alloy in a combined atmosphere of sulfur and selenium. For indium sulfide preparation indium is first deposited by thermal evaporation and then this metallic film is exposed to H\textsubscript{2}S atmosphere to get In\textsubscript{2}S\textsubscript{3} thin films of desired property. In the preparation of CuInSe\textsubscript{2} and CuIn(Se\textsubscript{1-x}S\textsubscript{x})\textsubscript{2} thin films a very thin layer of gallium was coated prior to indium coating in the first stage of two stage process (the precursor preparation), for better adhesion of the final film to the substrate. Details of thermal cycles used for chalcogenisation and the conditions are described in the respective sections of film growth.

### 2.7.1 Sulfurisation Set up

Sulfurisation was carried out in a specially designed set up as shown in figure. The set up consists of a reaction vessel made of quartz, temperature controlled heater and the sulfurisation source. H\textsubscript{2}S was used as the sulfurising agent, which was prepared using a Kipp’s apparatus by the reaction between dilute hydrochloric acid and ferrous sulfide. The sulfurisation temperature was varied from 250\degree C to 400\degree C. The schematic diagram of the set up used for sulfurisation is shown in figure 2.1.
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2.7.2 Selenization Set up

Selenization was carried out in a specially designed apparatus consisting of a double walled quartz tube and a split furnace which allowed rapid heating and cooling of the samples. Selenium shots were used as the selenium source since H$_2$Se gas is highly toxic. The unreacted selenium is condensed on the water cooled end of the quartz tube. Any trace of Se escaping the furnace is dissolved in carbon di sulfide. The nitrogen gas was flowed from the selenium source region to the substrate region to carry the selenium vapor. The selenization temperature was varied from 250$^\circ$ C to 400$^\circ$ C. A Proportional Integral Derivative (PID) controller was used to maintain different thermal cycles. The photograph of the selenization set up fabricated in our lab is shown in figure (Fig. 2.2).

For co chalcogenisation and post sulfurisation a mixture of selenium and sulfur atmosphere were used. Selenium atmosphere was created as explained previously. With this arrangement H$_2$S is allowed to pass during the selenization for co chalcogenisation. To carry out the post sulfurisation the CuInSe$_2$ samples prepared by selenization process were allowed to sulfurise for different duration.
Figure 2.2 The photograph of selenization set up
2.7.3 Substrate Cleaning

The chemical cleanliness of the glass substrate, prior to growth, directly influences the material properties of the deposited films. Scratches on the glass have an adverse effect on the structural properties of the thin films, while the presence of contaminants normally results in films with poor adhesion properties. Commercially available microscopic glass slides (7.5 cm x 2.5 cm) and quartz substrates were used as substrate. To eliminate visible impurities the slides were washed with a commercially available soap solution and then with distilled water. To eliminate organic impurities they were kept in freshly prepared chromic acid for 20 minutes. Then these slides were washed in a current of distilled water and dried.

2.8 Characterisation of the Thin Films Prepared

The optimization of the preparation conditions is the main task in order to get device quality films. This has to be carried out on the basis of detailed structural, compositional, morphological, optical and electrical properties of the films obtained at different growth conditions. In this work, the analysis of the thin films prepared was done during deposition and after film formation. The following section deals with the various characterisation tools used for the study.

2.8.1 Thin film thickness

i) Quartz Crystal Microbalance

A quartz crystal microbalance (QCM) measures mass by measuring the change in frequency of a piezoelectric quartz crystal when it is disturbed by the addition of a small mass of any other tiny object intended to be measured. Frequency measurements are easily made to high precision:
hence, it is easy to measure small masses. Correlation between mass and frequency is achieved by means of the Sauerbrey equation [1].

In this in-situ method, thickness measurement depends on the oscillation of a quartz crystal when excited and the frequency of its oscillation depends on its thickness as given by the relation [2],

\[ f = \frac{v}{2d} = \frac{N}{t} \quad \text{(nm/kc/sec)} \]

where, \( v \) is the velocity of the transverse elastic waves normal to the crystal plate, \( d \) is the thickness of the crystal and \( N \) is the frequency constant depending on the nature of the crystal.

When a film of thickness \( t \) is deposited on the quartz plate, the mass of the crystal is changed. The corresponding change in the frequency of the crystal can be utilised to find the average thickness of the film deposited.

\[ t = \frac{\Delta f}{C \sigma} \]

where, \( \sigma \) is the density of the deposited film and \( C = \frac{f^2}{N \rho} \) (\( \rho \) is the density of the quartz crystal) is called the sensitivity for mass determination which is a constant of the crystal used. The QCM used in our lab was Model C200 in which the changes in the resonant frequency of the quartz crystal oscillator with the film deposition are calibrated to give the deposition rate and the thickness of the film. The quartz thickness monitor was used during the deposition of Ga, In and Cu layers.

**ii) Stylus Thickness Profiler**

The thickness of the films obtained after two stage process is measured using stylus thickness profiler (Vecco Dektak 6M Stylus Profiler). In principle this method consists of measuring the mechanical movement of a stylus as it
traverses a film-substrate step. The diamond stylus has a tip radius of 0.0001 inch, and bears on the specimen being measured with a force of about 0.1 gm. The stylus traverses a substrate film step, and the vertical motion of the stylus relative to a reference plane is converted to an electrical signal. This signal is amplified and recorded on rectilinear paper. Thus, a profile graph is produced which represent a cross section of film step as well as substrate surface irregularities. The schematic diagram illustrating the determination of thickness of thin films using stylus profiler is given in figure 2.3

![Diagram](image)

Figure 2.3 Schematic diagram illustrating the determination of the thin film thickness with Stylus Profiler

2.8.2 Structural Characterisations

i) X-Ray Diffraction (XRD) Technique

Solid matter can be described as 1) amorphous: The atoms are arranged in a random way 2) crystalline: The atoms are arranged in a regular pattern, and there is as smallest volume element that by repetition in three dimensions describes the crystal. This smallest volume element is called a unit cell. The dimensions of the unit cell are described by three axes: a, b, c and the angles between them alpha, beta, and gamma.
About 95% of all solid materials can be described as crystalline. When X-rays interact with a crystalline substance (Phase), one gets a diffraction pattern. This x-ray diffraction pattern is like a fingerprint of the substance. The powder diffraction method is thus ideally suited for characterization and identification of polycrystalline phases by a match procedure [3]. Furthermore, the areas under the peak are related to the amount of each phase present in the sample.

The basic law involved in the diffraction method of structural analysis is the Bragg’s law. When monochromatic X-rays impinge upon the atoms in a crystal lattice, each atom acts as a source of scattering. The crystal lattice acts as series of parallel reflecting planes. The intensity of the reflected beam at certain angles will be maximum when the path difference between two reflected waves from two different planes is an integral multiple of \( \lambda \). This condition is called Bragg’s law and is given by the relation,

\[
2d \sin \theta = n \lambda
\]

where \( n \) is the order of diffraction, \( \lambda \) is the wavelength of the X-rays, \( d \) is the spacing between consecutive parallel planes and \( \theta \) is the glancing angle [4].

X-ray diffraction studies give a whole range of information about the crystal structure, orientation, average crystalline size and stress in the films. Experimentally obtained diffraction patterns of the sample are compared with the standard Powder Diffraction Files published by the International Centre for Diffraction Data (ICDD). International Center Diffraction Data (ICDD) or formerly known as (JCPDS) Joint Committee on Powder Diffraction Standards is the organization that maintains the data base of inorganic and organic spectra.

The average grain size of the film can be calculated using the Scherrer’s formula [5],
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\[ d = \frac{0.9 \lambda}{\beta \cos \theta} \]  

where, \( \lambda \) is the wavelength of the X-ray and \( \beta \) is the full width at half maximum intensity in radians.

The lattice parameter values for different crystallographic systems can be calculated from the following equations using these hkl parameters and the interplanar spacing \( d \).

- **Cubic system**, 
  \[ \frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} \]  

- **Tetragonal system**, 
  \[ \frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \]  

- **Hexagonal system**, 
  \[ \frac{1}{d^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \]

The particular advantage of X-ray diffraction analysis is that it discloses the presence of a substance and not in terms of its constituent chemical elements. Diffraction analysis is useful whenever it is necessary to know the state of chemical combination of the elements involved or the particular phase in which they are present. Compared with ordinary chemical analysis the diffraction method has the advantage that it is much faster, requires only very small sample and is non destructive.

X-ray diffraction measurements of the different films were done using Rigaku automated X-ray diffractometer. The filtered copper K\( \alpha \) (\( \lambda = 1.5418 \text{Å} \)) radiation was used for recording the diffraction pattern.
Scanning electron microscopy (SEM) is used for inspecting topographies of specimens at very high magnifications using equipment called the scanning electron microscope. There are many advantages in using the SEM instead of a light microscope. The SEM has a large depth of field which allows a large amount of the sample to be in focus at one time. The SEM also produces images of high resolution. The combination of higher magnification, larger depth of focus, greater resolution makes the SEM one of the most heavily used instrument in current research field. SEM inspection is often used in the analysis of cracks and fracture surfaces, bond failures, and physical defects on the die or package surface.

During SEM inspection, a beam of electrons is focused on a spot of the specimen, resulting in the transfer of energy to the spot (Fig. 2.4). These bombarding electrons, also referred to as primary electrons, dislodge electrons from the specimen itself. The dislodged electrons, also known as secondary electrons, are attracted and collected by a positively biased grid or detector, and then translated into a signal.

To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, analyzed, and translated into images of the topography being inspected. The electron beam typically has an energy ranging from a few hundred eV to 50 keV.
A SEM may be equipped with an Energy Dispersive X-ray (EDX) analysis system to enable it to perform compositional analysis on specimens.

**iii) Energy Dispersive X-ray Analysis**

EDX Analysis stands for Energy Dispersive X-ray analysis. It is a technique used for identifying materials and contaminants, as well as estimating their relative concentrations on the surface of the specimen. The EDX analysis system works as an integrated feature of SEM, and cannot operate on its own without the latter.

During EDX Analysis, the specimen is bombarded with an electron beam inside the scanning electron microscope. The energy of the beam is typically in the range 10-20keV. The bombarding electrons collide with the specimen electrons, knocking some of them off in the process. A position vacated by
an ejected inner shell electron is eventually occupied by a higher-energy electron from an outer shell giving up some of its energy as X-ray.

The amount of energy released by the transferring electron depends on which shell it is transferring from, as well as which shell it is transferring to. Furthermore, the atom of every element releases X-rays with unique amounts of energy during this transferring process. Thus, by measuring the amounts of energy present in the X-rays being released by a specimen during electron beam bombardment, the identity of the atom from which the X-ray was emitted can be established. The X-rays are generated in a region about 2 microns in depth, and thus EDX is not a surface science technique.

The output of an EDX analysis is an EDX spectrum. An EDX spectrum normally displays peaks corresponding to the energy levels for which the most X-rays had been received. Each of these peaks is unique to an atom, and therefore corresponds to a single element. The higher a peak in a spectrum, the more concentrated the element is in the specimen.

2.8.3 Optical Characterisations

The knowledge of the optical property of any type of PV sample either under collimated light incident at variable angles, or under diffuse light, nevertheless, would greatly contribute to the comprehension of its electrical performances when it is exposed outdoors to the solar irradiation.

The spectrophotometers enable the measurement of optical constants like the absorption coefficient, band gap, spectral reflectance and transmittance of any prototype sample, of small dimensions (few square centimetres), under a collimated light beam incident at a fixed angle.
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i) Absorption coefficient and Band gap

The absorption coefficient of a solar cell depends on two factors: the material making up the cell, and the wavelength or energy of the light being absorbed. Solar cell material should have an abrupt edge in its absorption coefficient. The reason is that light whose energy is below the material’s band gap cannot free an electron and it isn’t absorbed. A small absorption coefficient means that light is not readily absorbed by the material.

The band gap $E_g$ of a semiconductor material is the minimum energy needed to move an electron from its bound state within an atom to a free state, where the electron can be involved in conduction. The lower energy level of a semiconductor is called the valence band and the higher energy level where an electron is free to roam is called the conduction band. The band gap is the energy difference between the conduction band and valence band.

When the energy of the incident photon ($h \nu$) is larger than the band gap energy the excitation of electrons from the valence band to the empty states of the conduction band occurs. The light falling on the material is then absorbed. Electron hole pairs are created depending on the number of incident photons $S_0(\nu)$ (per unit area, unit time and unit energy). The frequency $\nu$ and wavelength $\lambda$ of the incident photon are related by the equation

$$\lambda \ [\mu \text{m}] = \frac{c}{\nu} = \frac{1.24}{\nu}$$  \hspace{1cm} (2.8)

where $c$ is the speed of light.

The photon flux $S(x, \nu)$ decreases exponentially inside the material according to the relation

$$S(x, \nu) = S_0(\nu) \exp(-\alpha x)$$  \hspace{1cm} (2.9)
where, the absorption coefficient, \( \alpha(v) = 4\pi kv/c \) is determined by the absorption process in semiconductors and \( k \) is the extinction coefficient [6].

The absorption coefficient \( \alpha(v) \) depends on the band structure of the semiconductor. In direct band gap semiconductors where the minimum of the conduction band and the maximum of the valence band occur for the same wave vector in the Brillouin zone, the absorption coefficient \( \alpha_d \) as a function of the frequency \( v \) is given by the relation [7],

\[
\alpha_d(v) = \frac{\alpha_0 (h v - E_g)^{1/2}}{h v}
\]  
2.10

where \( E_g \) is the band gap energy of the material and \( \alpha_0 \) is a constant.

In indirect band gap semiconductors, the absorption coefficient for allowed transition takes the form

\[
\alpha_{in}(v) = \frac{(h v - E_{gi} \pm E_p)^2}{\exp\left(\frac{E_p}{K T}\right) - 1}
\]  
2.11

where \( \pm E_p \) is the absorbed or emitted phonon energy, \( K \) is the Boltzmann constant, \( T \) is the temperature, \( E_{gi} \) is the indirect band gap energy.

So for direct transition (2.9), we have

\[
(\alpha hv)^2 = \alpha_0 hv - \alpha_0 E_g
\]  
2.12

Therefore, a plot of \((\alpha hv)^2\) vs \( hv \) will be a straight line for direct band gap materials. The intercept of the curve to the photon energy axis gives the band gap \( (E_g) \) of the material.

The absorption spectra of the samples were taken using JASCO V570 UV-VIS-NIR spectrophotometer. It measures the intensity of light passing through a sample \( (I) \), and compares it to the intensity of light before it passes through the sample \( (I_o) \). The ratio \( I / I_o \) is called the transmittance, and is
usually expressed as a percentage ($\%T$). The absorbance, $A$, is based on the transmittance:

$$A = -\log (\%T)$$

The absorption coefficient ($\alpha$) was calculated from the absorbance. Finally the band gap of the material is found out from the plot $(\alpha hv)^2$ vs $hv$.

2.8.4 Electrical Characterisations

i) Resistivity by Two Probe Method

The resistivity of the films was determined by the two-probe method with the electrodes in planar geometry. Evaporated indium layers or high conducting silver paste was used as the electrodes. The current voltage measurements were carried out using a Keithley’s source measure unit (Model SMU236). The resistivity ($\rho$) of the films is calculated applying ohm’s law, by the relation $\rho = RA/L$. Where $R$ is the resistance obtained from current- voltage characteristic curves. ‘$A$’ is the area of the film in planar geometry which is given by the product of the film thickness and the width of the film. $L$ is the spacing between the electrodes.

ii) Temperature Dependence of Conductivity

The temperature dependence of conductivity was measured by measuring the current voltage (I-V characteristics) varying temperature of the specimen from 20 K to 500 K. Keithley’s source measure unit (Model SMU236) and liquid helium cryostat having automated temperature controller (Model Lakeshore 321) was used to carry out the I-V characteristics. Liquid helium was used to cool the samples to 20 K. The specimen temperature was raised to 500 K using heaters. The voltage is kept constant and the variation of current with temperature is noted.
The activation energy is calculated from the arrhenious plot, which allows determining the time and temperature relationship of a process.

Thermally excited reactions are described by

$$\sigma = \sigma_0 \exp\left(-\frac{E_a}{kT}\right)$$  \hspace{1cm} 2.13

where \(E_a\) is the activation energy [8].

An arrhenious plot of this equation is a plot of \(\log \sigma\) over \(1/kT\) which gives a straight line. The slope of this line yields the activation energy.
References


Bibliography

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