

**SYNTHESIS AND CHARACTERIZATION
OF LOW THICKNESS PtSe₂ FILM**

DISSERTATION

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By

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Declaration

I declare that the work presented in the Dissertation entitled '**TO SYNTHESIZE AND CHARACTERIZE LOW THICKNESS PtSe₂ FILM**' being submitted to the Department of Physics, School of Physical Sciences, Doon University, Dehradun, for the award of Masters in Physics is my original research work.

The Dissertation embodies the results of investigations, observations, and experiments carried out by me. I have neither plagiarized any part of the dissertation nor have submitted the same work for the award of any other degree/diploma anywhere.

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Certificate

This is to certify that the Dissertation entitled “**To Synthesize and characterize low thickness PtSe₂Film**”, submitted by **Deeksha Panwar** in partial fulfillment for the award of postgraduate degree in Physics, department of Physics, School Of Physical Sciences, Doon University. This work has been carried out at Doon University, Dehradun and IIT Roorkee during the period of Jan 2022 to June 2022 under the supervision and guidance of Dr. Archana Mishra, “Assistant Professor”, Doon University, Dehradun. It is also certified that the work in this dissertation embodies original research and hard work of the candidate.

Dr. Archana Mishra

Supervisor/Guide

Dr. Himani Sharma

Head of Department

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ABSTRACT

The material Platinum Diselenide is a member of the 2D group 10 noble Transition Metal Dichalcogenide family (TMDC's). TMDC's are a group of materials with formula MX_2 where M a transition metal lies between two planes of X a chalcogen atom. $PtSe_2$ transits from bulk exhibiting semi metallic character to monolayer exhibiting semi-conductor character with a band gap of 1.2 eV.

In this report we are studying the thickness dependent properties in $PtSe_2$ thin films using Pulsed laser deposition Technique. During PLD, many experimental parameters can be altered, which creates a strong influence on film properties. The laser parameters such as wavelength, pulse duration etc. can be altered. The preparation conditions, including target-to-substrate distance, substrate Temperature, background gas and pressure, may be varied, which impacts the thin films growth. X-ray diffraction (XRD) is used for the characterization and verification of thin film. $PtSe_2$ has received a lot of attention in the recent years because of its interesting physical properties for both fundamental research and potential application in nanoelectronics, optoelectronics, spintronics etc.

Keywords: pulsed laser deposition, Platinum diselenide, X-ray diffraction and scanning-electron microscopy

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CHAPTER 1 INTRODUCTION

In this dissertation we briefly study about the synthesis and characterization of low thickness PtSe₂ Film and its deposition techniques using pulse laser deposition that is a technique of pulse vapor deposition PVD. The advantages of pulse laser deposition are intimated in accordance with phases of PtSe₂. A brief discussion about the material used for deposition is also done.

1.1 Overview

While demanding more reliability with reduced cost the aspects of electronic devices constantly decreasing in size and because of the crystalline structure, distinctive properties, weak interlayer Van der waals interaction, varying composition, band alignment and band alignment in organic layered solids appeared as a flourishing research field. To keep layers together permitting TMDC have distinctive electronic, optical and chemical properties the structure of material maintained by van der waals interaction. Layered metal dichalcogenide electronic properties rely on crystalline polytype and precisely depend on d-electron that give these material (like semiconductors, metal) unique electronics Behaviour.

Since past century TMDC's are being studied, when in 2004 the synthesis of graphene bring on the cleared ways for 2 dimensional research. The intrinsic graphene came up as zero gap semiconductors, along with high electron mobility at room temperature with resistivity/monolayer and this defined resistivity substance is the lowest at room temperature. However, In spite of all the emerged properties of graphene the need to explore more 2D materials is created by the lack of band gap. To precisely ensure the reproducible and reliable utilization for some material on electronic devices the growth of high standard LMDs is lately a defining factor. After reading different standard methods such as chemical vapor deposition, mechanical exfoliation, laser thinning, liquid exfoliation, chemical bath deposition and

molecular beam epitaxy for synthesis and generating high quality of thin films it is being observed that PVD has more potential to be studied in detail and that proper picking of parameters in this might conclude in generation of high quality thin film because in other methods we were facing different issues and restraints such as unrestrained geometry, size, increased roughness, expensive and dangerous precursors. In the process of PLD high reaction rate can be achieved by kinetics that allows to produce good quality thin film, also in process of PLD the growth rate can be only limited through repetition rate of laser that permits control of thickness by regulating the number of pulses. Also one of the most

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common comparison of PVD with CVD and Molecular beam Epitaxy is that PVD process is way cheaper than these and non-dangerous.

1.2 Thin Film

Thin film is a fine layer of material that ranges from fractions of nanometers to some micrometer in thickness. The underlying properties of the substrate influence the properties of the thin film, which can differ greatly depending on the thickness of the film and the deposition techniques used. In many application-controlled synthesis of thin film is a fundamental step. The advancement in thin film deposition technique in the course of 20th century has permitted a wide range of technological development in areas like Semiconductor Devices, Integrated passive devices, LED Optical coatings, For energy production (such as solar cell thin film) and storage (battery thin film). Further these films may broadly be classified in three subdivisions as per their thickness as - Ultra-thin [50-100] Å, Thin [100-1000] Å, Comparatively thick one [greater than 1000] Å.

Although, the thickness limit of this is arbitrary. A thin film can be defined as a multilayer. Thin film dependency is on thickness for properties and behavior. Thickness has an important role in thin film. Being a vital parameter, thickness influences the electrical, optical, structural and so on, properties of metals greatly. Duplicable characteristics are acquired by the choice of particular thickness and great combination of deposition parameters for a specific material.

There are some exceptional properties about these thin films that is the thin films can be distinguished on the basis of their Structure, Chemical Properties etc.

And despite this fact that there are numerous orders of magnitude fewer atoms obtainable in films. General testing and investigation of thin films can be accomplished with equipment and instrument that are diverse in character. Some allocation in these are given below:

Size may differ from the portable desktop interferometer to the 504 extensive accelerator and beamline of a Rutherford backscattering (RBS) facility.

- Cost ranges from that of test instruments needed to estimate electrical resistance of films to the approximate \$1 million price badge of a commercial SIMS spectrometer.
- Operating Environment differs from the encircling in the measurement of film thickness to 10^{-10} torr vacuum needed for the estimation of film surface composition.
- At one extreme is mixed bag of electron microscopes and at another extreme is manually done scotch-tape film peel test for adhesion and surface analytical equipment where operation, data assembly, proper analysis, and display are essentially computer controlled.

This has found everywhere that a film may have several impurities, imperfections, dislocations, grain boundaries and other various defects and also could be interrupted. To lessen some of these issues proper control on the situation and conditions of deposition is to be maintained. Thin films are bit ordinary now-a-days and can be sought more or less almost everywhere. These films are applied to enhance the surface of a material.

1.3. Thin Film Deposition and Growth

The thin film deposition process involves producing and depositing thin film coatings onto a substrate material. These coatings could be made of several materials, from metals to oxides to compounds. For the thin film growth and nucleation, there are three important processes. The first involves the synthesis of the deposition material, namely the substrate and the target material, followed by transfer from the target to the substrate using a deposition technique, and finally, the growth of the target on the substrate to produce the required thin film.

The atoms from the target infringe on the substrate, where they can either reflect instantly from the substrate or condense on the substrate surface, evaporating back into the gas phase after a certain residence period. Numerous factors influence this process, including activation energy, target-substrate binding energy, adhesion coefficient, and so on. During this process, the atoms lose energy and might not immediately react with the substrate. They will

has some mobility over the surface before condensation and these mobile atoms are referred to as adatoms which is a portmanteau for adsorbed atoms. The energy lost during condensation of the atom on the surface can either be by chemical reaction with the substrate atoms, the collision of the diffusing surface atoms, finding a preferential nucleation site or collision of the adsorbed surface species. If the surface mobility is low and the atom to atom interaction is strong, each atom can serve as nucleation site for growth but when the adatom-surface interaction is feeble, the surface mobility of the condensing adatom will be high and results in condensation at preferential nucleation sites where there is stronger bonding either due to an increase in the coordination number or a change in elemental or electronic chemistry. The condensing atoms react and bond with the atoms on the surface to form atom to atom chemical bonds. The chemical bond formed can be of any of these following bonding types: electrostatic attraction (van der Waals forces) due to the polarization of atoms, metallic (homopolar) bonding where the atoms share orbital electron or electrostatic (coulombic heteropolar) where the ions are formed due to electron loss or gain. The atom is said to have undergone chemisorptions if the resulting bonding from the reaction between the condensed atom and the surface atom is very strong. These adatoms transform into nuclei by a conglomeration of numerous adatoms and result in the continuous thin film. Due to the nature of the deposition, the resulting nuclei formed at the initial stage are often thermodynamically unstable and might desorb with time depending on the deposition parameters used. After a while, the clustered nuclei reach a critical safe size and become thermodynamically stable and the nucleation challenge is said to have been overcome. This process involving the transition from thermodynamically unstable condition to a thermodynamically stable condition where a stable, critical-sized nucleus formed is termed to the nucleation stage. As the deposition process continues, the stable critical nuclei grow in number as well as in size until a saturated nucleation density is attained. The nucleation density and the average nucleus quantity depends on number of deposition parameters such as the temperature of the substrate, working pressure, adhesion properties, binding energy between the target and the substrate,

energy of the impinging species, the activation energies of adsorption, desorption, thermal diffusion, the rate of impingement, topography, and chemical nature of the substrate. A nucleus can grow in two modes named parallel and perpendicular depending on the configuration of the substrate and target, and the two scenarios can be experienced in a single deposition. Parallel growth occurs on the substrate by surface diffusion of the adsorbed atoms while perpendicular growth is because of direct impingement of the incident material. However, the rate of parallel growth sometimes called lateral growth is much higher than the perpendicular growth and the grown nuclei are called islands. In the coalescence stage, small islands start coalescing with each other on the surface of the substrate to reduce the substrate surface area and replace it with a thin film coating. This resulted in the formation of a bigger island and the process is called agglomeration. Increasing the surface mobility of the adatoms enhances the agglomeration process and growth density of the nucleation site, i.e., by increasing the temperature at the surface of the substrate. In some reactions, the formation of new nuclei can also take the areas newly exposed because of coalescence. Bigger islands grow together, leaving channels and holes of the uncovered substrate between the islands and leading to discontinuous film formation on the surface of the substrate creating a porous defect on the surface. Further growth of the larger islands causes continuous agglomeration and filling of the created channels and holes.

In the production of many opto-electronic thin film deposition has a significant manufacturing step like in solid state, medical equipment and products, microscopy & microanalysis samples, including consumer electronics, semiconductor lasers, fibre lasers, optical filters, precision optics, compound semiconductors, medical implants and LED displays

1.4 Material Used for Deposition

Deposition Technique. PtSe₂ is a distinctive Transition Metal DiChalcogenide (TMDC) material with a wide range of unique optical features. Its fascinating physical features and potential practical uses, has attracted PtSe₂ lot of attention. It is a semiconductor with bandgaps of 1.2 eV and 0.21 eV in monolayer and bilayer forms. It becomes semi metallic when the thickness reaches. Moreover, it has greater carrier mobility than any other TMDC. It is a great material for next level-generation sensors, optoelectronics, and ultrafast photonic devices, according to the researchers.

And PtSe₂ is tempting due to its amendable electronic properties, depending on its thickness. For instance, monolayer PtSe₂ has an indirect bandgap of around 1.2 eV, which decreases as number of layers increases. Hence, it enables effective control of bandgap through a change of the layer thickness. Furthermore, defects can also be added for widening of its electrical properties. Even at biaxial strain, the semiconducting nature of 1T-PtSe₂ monolayers remains unaffected.

1.4.1 Crystal Structure of PtSe₂

There are two common structural phases for monolayer TMDCs, which are characterized by either octahedral trigonal prismatic (2H or D_{3h}) or (1T or D_{3d}). Unlike group-6 TMDCs, group-10 TMDCs tend to form d₂sp³ hybridization due to group-10 metal atoms holding rich d-electrons and less d orbitals are involved. As a result, group-10 TMDCs lead to the generation of the thermodynamically favored 1T-phase. PtSe₂ has a thermodynamically favored 1T-phase structure and the atoms stack in the AA arrangement. Many techniques have been employed to characterize the atomic structure of monolayer PtSe₂, such as high resolution scanning transmission electron microscope (HR-STEM), scanning tunneling microscope (STM), low energy electron diffraction (LEED), and density functional theory (DFT) calculation which clearly shows that each Pt atom is in a tilted octahedral site and surrounded by six Se atoms, which is consistent with the octahedral structure of 1T phase.

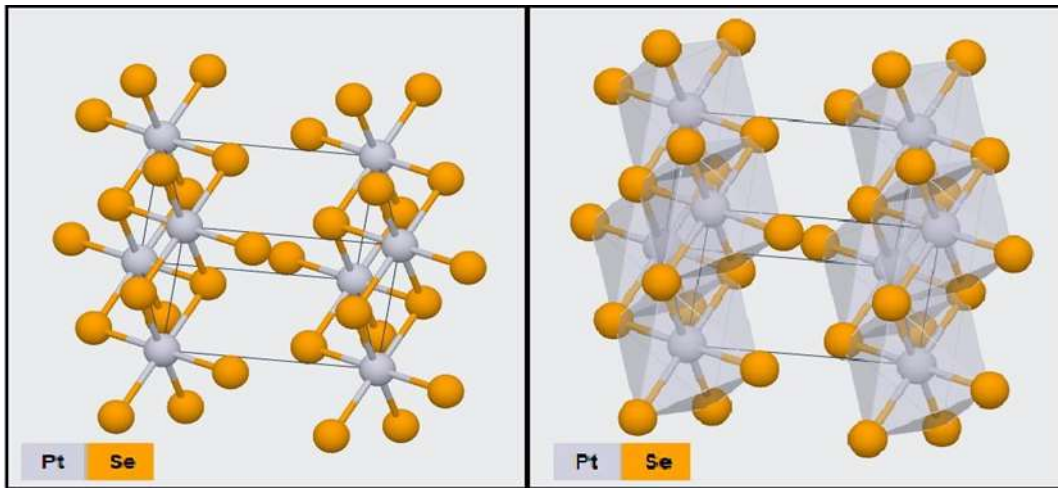


Figure 1.1 Crystal structure of PtSe₂

1.4.2 Properties of PtSe₂

There are certain properties that define PtSe₂ in different ways, such as band gap tunability and phase transition properties.

- BAND GAP TUNEABILITY:** The band structure of 2D TMDCs can be tuned by doping, defect engineering, strain, and external electric field. Other than this, their inherent thickness-dependent band gap of PtSe₂ can also be tuned by applying external parameters.
- PHASE TRANSITION:** Because of the highly strong covalent bond and weak inter-layer interaction, the structure of 2D materials majorly depends on varying external criteria (pressure, strain, irradiation, annealing). Phase transition can be induced by introducing ionic intercalation, high pressure, strain, thermal treatment, and external electrical and magnetic field. Since 1T-PtSe₂ is a very stable structure, it is hard to expect a continuous phase transition unless inducing additional electron beam irradiation and annealing treatment. 1T phase PtSe₂ can transform into non-layered 2D PtSe₂ ultrathin film.

1.5 Silicon Substrate

An n-type Silicon (100) is used as a substrate in this study. A Si substrate or silicon wafer is a thin slice of crystalline silicon. Silicon wafers have wonderful electrical and mechanical properties and for the same reason they are the most extensively used wafers in industries. It provides versatility to the surface properties. In experiments pertaining to imaging, nanotechnology and micro-fabrication applications polished silicon have shown to be an excellent substrate. Since the background signal is low of the highly polished surface, Si is an ideal specimen substrate for small particles in imaging applications. These wafers in addition also provide excellent conductivity for SEM, FIB, STM applications. In the project we have used silicon as the substrate for studying the magnetic properties of thin films of Ni-Mn-Al. Silicon is a semi-conductor with high stability and due to its potential scalability, coefficient of thermal expansion and cost it proves to be an excellent base substrate. Silicon is suitable for its use as a substrate because it has relatively low costs due to use of well established processing techniques. It has a high efficiency and a thermal stability upto 1100°C. Due to its hardness, large wafers can be handled safely without any damage.

1.6 DEPOSITION TECHNIQUES

A deposition technique is an important aspect in the development of novel thin film materials. The applications of thin films depends on their stability and the morphology. The morphology of the thin films highly depends on deposition techniques. Thin films can be deposited by the physical and chemical techniques. Deposition techniques must be considered carefully based on the application because not all deposition procedures provide the same microstructure, surface morphology, electrical, biocompatibility, optical, corrosion, and hardness properties. There are various other types of deposition techniques, but as the focus in this study is on thin film deposition for forming layers less than one micron, the two most common thin film deposition techniques will be discussed. Physical vapour deposition (PVD) and chemical vapour deposition (CVD) are two of the most significant types of deposition

process:

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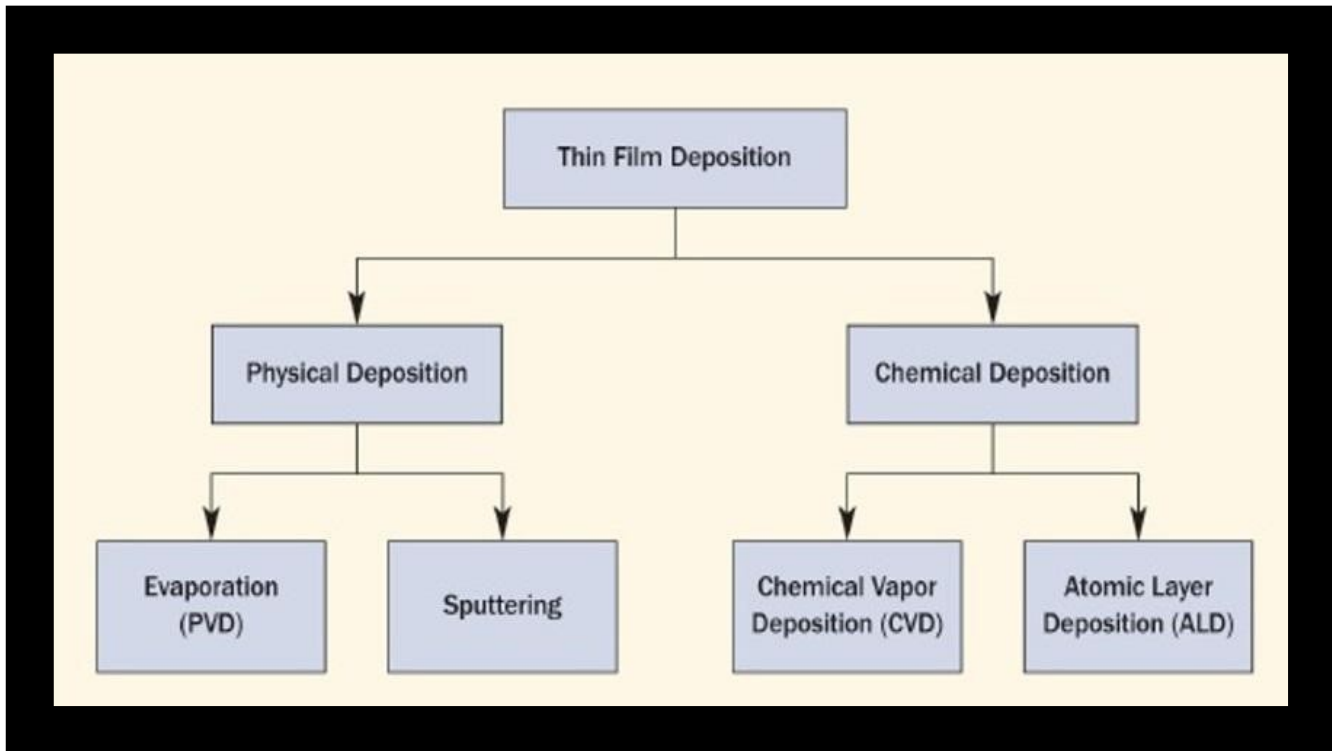


Figure 1.2 Deposition Technique

1.6.1 CHEMICAL VAPOUR DEPOSITION

Chemical Vapor Deposition (CVD) is a deposition process/technique where a solid material is deposited from vapor by some chemical reaction occurring on or in vicinity of normally heated substrate surface. It is an example of vapor-solid reaction. It is a thermodynamically a very complex process which involves chemical reactions under specific parameters such as pressure, temperature, reaction rates, and the transmission of momentum, mass, and energy.

CVD is quite useful in producing coatings for a broad variety of applications plus medical devices, automotive components and silicon wafers. Advantage of CVD methods involve potentiality to use these processes on a broad variety of substrates, also the ability to coat detailed or highly complex topographies. The Thin films formed through CVD also maintain their bonds precisely in a high-stress environments.

Few disadvantages involve size limitations, which depend on the size of the vacuum chamber, as well as that almost all methods typically need high value temperature, drive the chemical reactions. It can be tough to mask the surface of the coating.

Some materials prepared by CVD are Polysilicon, Silicon dioxide, Silicon nitride, Metals and Graphene. There are different types of CVD methods:

- Atmospheric Pressure CVD
- Ultravacuum CVD
- Atomic Layer Deposition CVD
- Plasma enhanced CVD

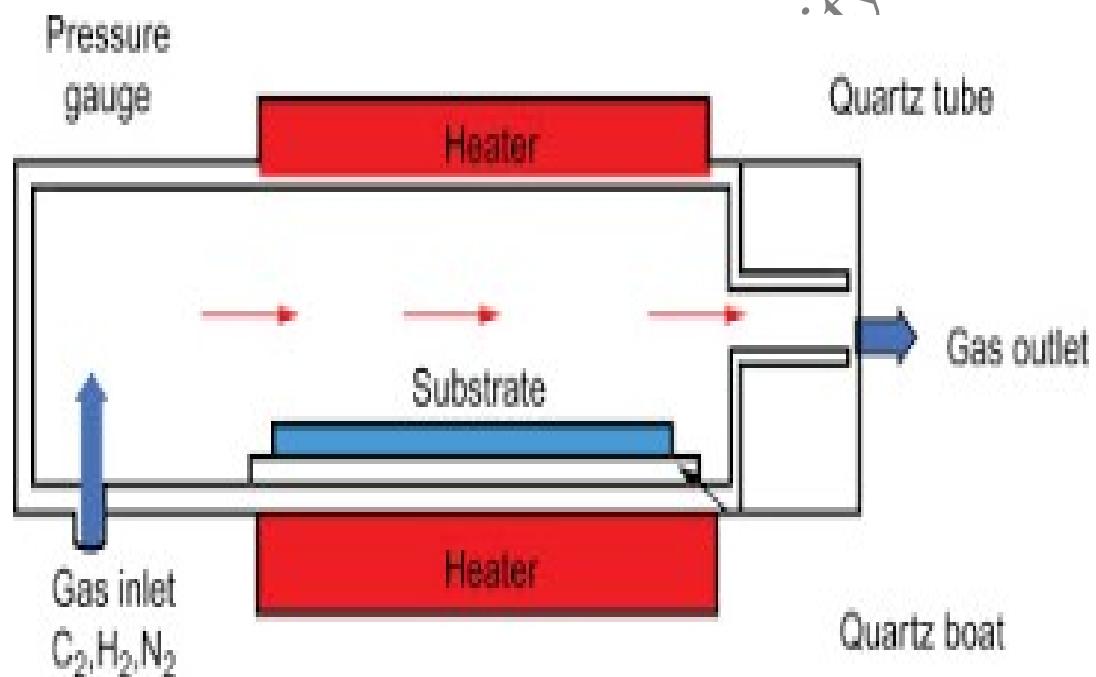


Figure 1.3 Chemical Vapor Deposition

1.6.2 Physical Vapor Deposition (PVD)

Physical vapor deposition includes a variety of vacuum deposition methods that can be used to produce thin films and coatings. Physical vapor deposition is a process in which the material goes from a condensed phase to a vapor phase and then back to a thin film condensed phase. Coatings produced in this type are long lasting and offer resistant to scratching and corrosion. PVD could be used to put in a broad variety of coating materials like, ceramics, metals, alloys and other inorganic compounds. feasible substrates include glass, metals

and plastics. PVD is very useful in the manufacturing of devices spanning from solar cells to eyeglasses to semiconductors.

The process is somewhat kind of chemical vapour deposition (CVD) omitting the raw materials/precursors (the material that going to be deposited starts out in solid form) where in CVD, the precursors are introduced to the chamber of reaction in form of gaseous state. PVD process showcase a versatile coating technology that is appropriate for almost unlimited combination of coating the substances and the substrate materials

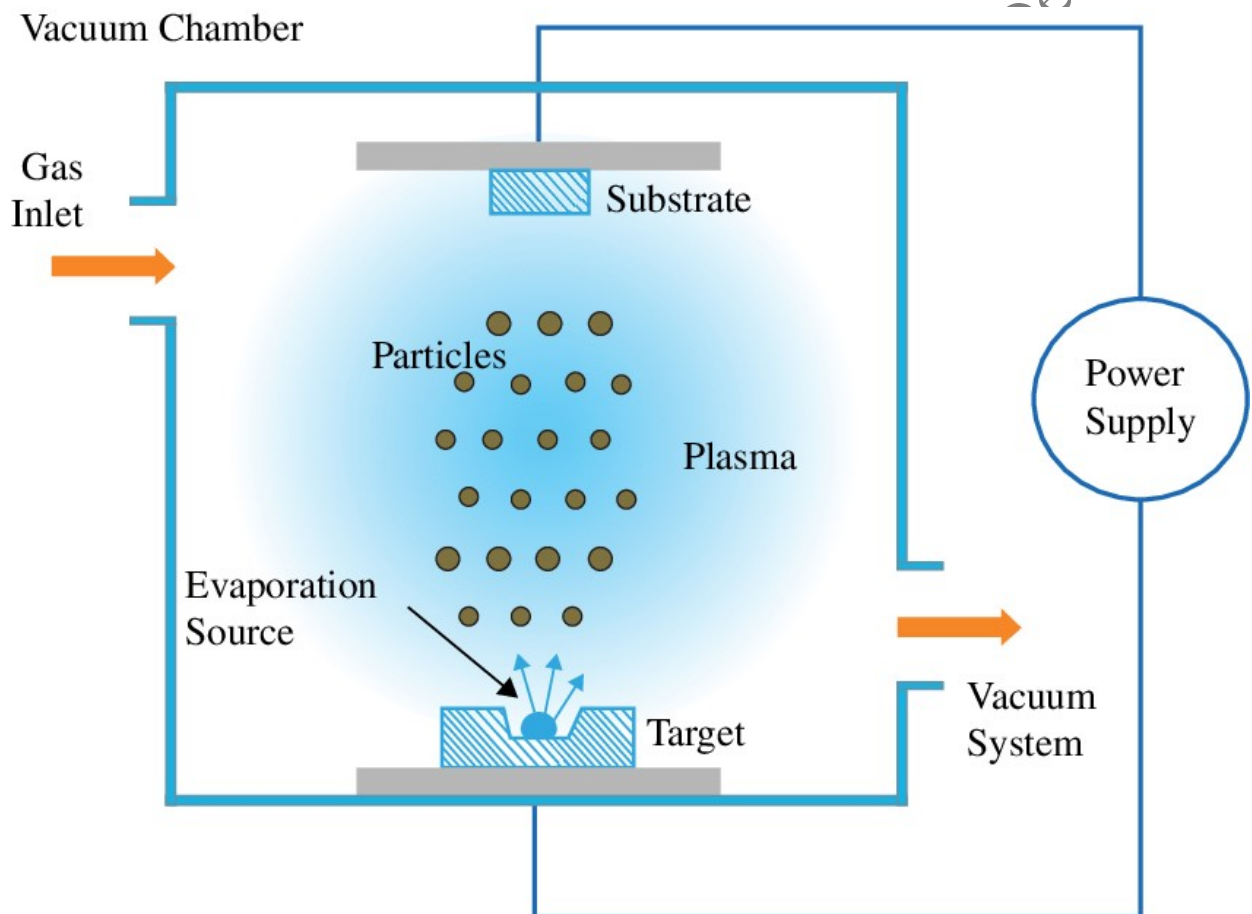


Figure 1.4 Schematic Diagram of PVD

Every PVD process can be attributed to three basic steps.

- First is, creation of vapor-phase species. This requires evaporation, sputtering, or ion bombardment of materials that are to be deposited from a solid state to a vapour phase.
- And there's the transfer from the source to the substrate. Molecular flow conditions and thermal scattering processes will move the ejected atoms or molecules from the target. There will also be considerable number of collisions in the vapour phase during travel to the substrate if the partial pressure of the metal vapour or gas species in the vapour state is high enough for some of these species to be ionised.

Film grows on the surface.

- By a number of techniques, the transferred atom or molecule will begin nucleate around substrate and grow.

The major forms of PVD are:

Electron beam evaporator: Electron Beam Evaporation is a form of Physical Vapor Deposition in which the target material is bombarded with highly charged electron beam from a charged tungsten filament to evaporate and convert it to a gaseous state for deposition on the material to be coated. It takes place in vacuum chamber, where the atoms or molecules in a vapor phase then precipitate and form a thin film coating on the substrate. Electron Beam Evaporation is used for optical thin film ranging from laser optics, solar panels, eye glasses and architectural glass to give them the desired conductive, reflective and transmissive qualities.

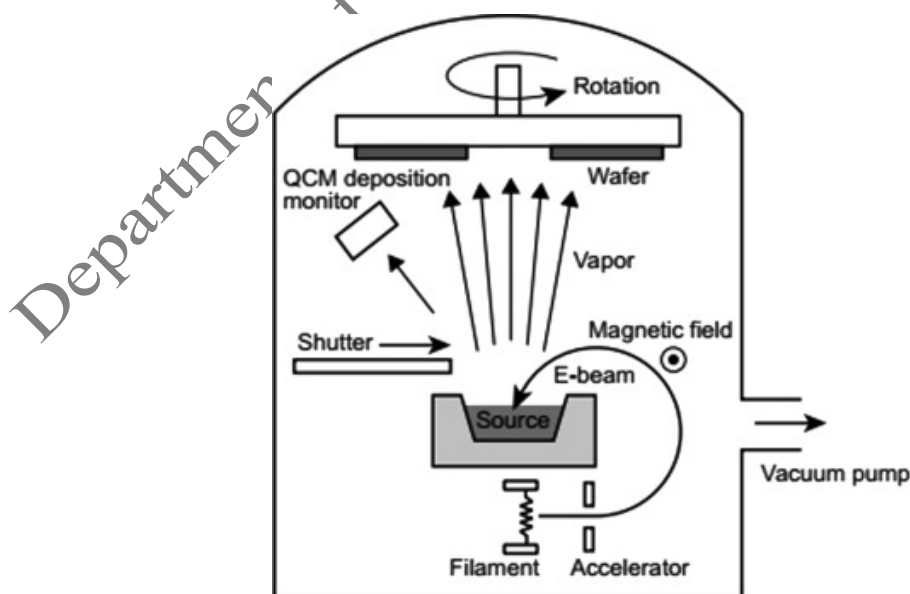


Figure 1.5 Schematic of Electron Beam Evaporator

Thermal evaporator: In this technique the material is melted and the vapour pressure is raised to a desirable range using an electric resistance heater in this approach. This is done at high vacuum to allow the vapour to reach the substrate without interacting with or scattering against other gas-phase atoms in the chamber, as well as to reduce the incorporation of impurities from the vacuum chamber's residual gas. Deposition rates for evaporated materials are generally very high, which minimizes incorporation of impurities into the film and can result in a very fine grain microstructure.

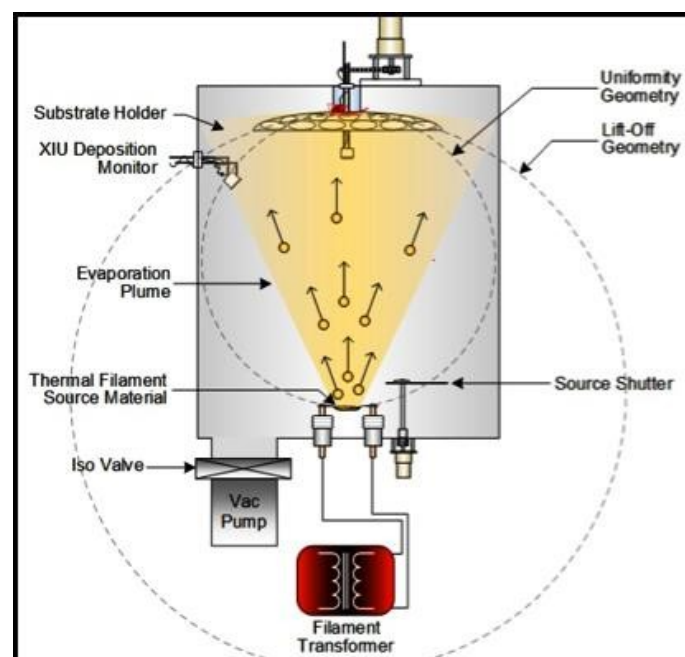


Figure 1.6 Schematic of Thermal Evaporator

Molecular beam epitaxy (MBE): One of the most frequent procedures for placing single crystals. This demands a high or ultra-high vacuum. The slow deposition rate (usually less than 1000 nm per hour), which permits the films to develop epitaxially, is the most critical aspect of MBE. Slow streams of an element can be directed at the substrate in this method, allowing material to deposit one atomic layer at a time. The major advantage of MBE comes from its controllable deposition rate at the atomic or molecular scale. This can be achieved due to the use of effusion cells that enable the generation of “molecular beam,” where the generated vapors have low interparticle collisions before they reach the substrate surface. This means the evaporated atoms escape from the orifice of the cells through effusion and have a long mean free path. They neither interact with each other nor with the gases in the vacuum chamber.

MBE is an advanced form of thermal evaporation technique with highly controllable deposition thickness (and thus with the drawback of slow deposition rate) due to the use of effusion cells and high vacuum.

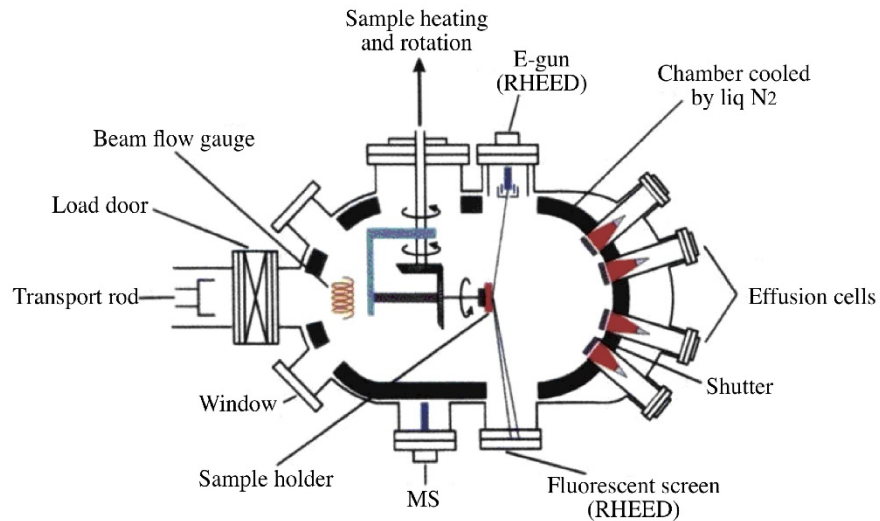


Figure 1.7 Schematic of MBE

Sputtering: It is a process where atoms are released from a solid target material due to bombardment of the target by energetic particles such as ions and atoms. The atoms released from the target can then be deposited as thin film on substrates and is commonly referred as PVD by sputtering. Plasma (usually a noble gas like argon) is used to knock material from a "target" one atom at a time. Because the procedure does not include evaporation, the target must be kept at a low temperature, making this one of the most adaptable deposition processes. It's best for compounds or combinations where various components evaporate at different rates. The coverage of sputtering steps is more or less conformal. It's also used a lot in optical media. It's a quick technique that also allows for fine thickness control. Sputtering only takes place when the kinetic energy of the bombarding particles is extremely high, much higher than normal thermal energies in the "Fourth state of nature" plasma environment. This can allow a much more pure and precise thin film deposition on the atomic level than can be achieved by melting a source material with conventional thermal energies.

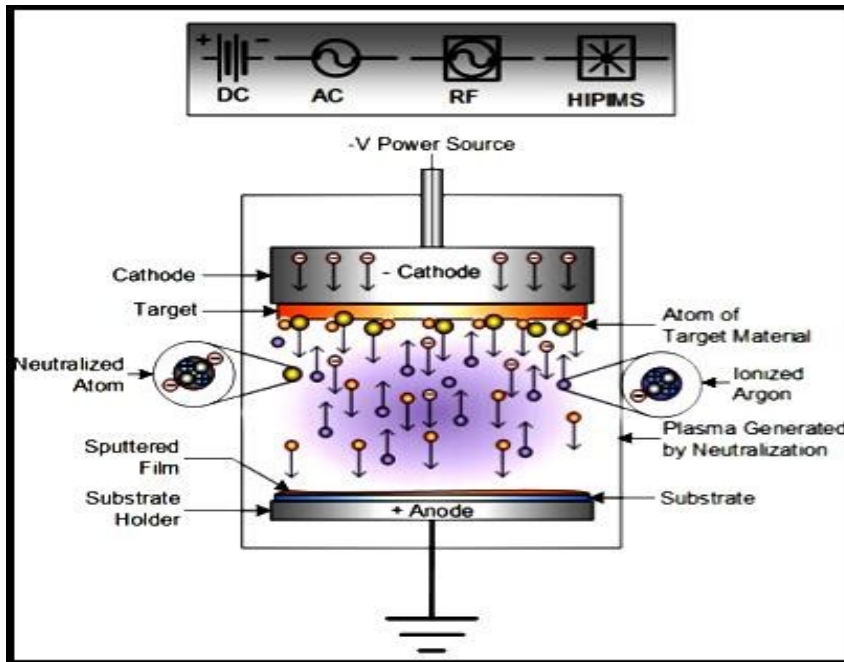


Figure 1.8 Schematic Diagram of the Sputtering Process

Pulsed laser deposition (PLD): This is a thin film deposition process in which a high-power pulsed laser beam is focused inside a vacuum chamber to strike a material target and deposit the material. When the laser pulse is absorbed by the target, energy is converted to electronic excitation and then into thermal, chemical and mechanical energy. This results in rapid evaporation or ablation of the target which produces a luminous plasma plume. The plume contains many energetic species including atoms, molecules, electrons, ions, particulates and molten globules. The plume which contains the ablated target material will rapidly expand into the surrounding vacuum and then condenses into a thin-layer film on the substrate. This procedure is carried either under ultra-high vacuum or in the presence of a background gas.

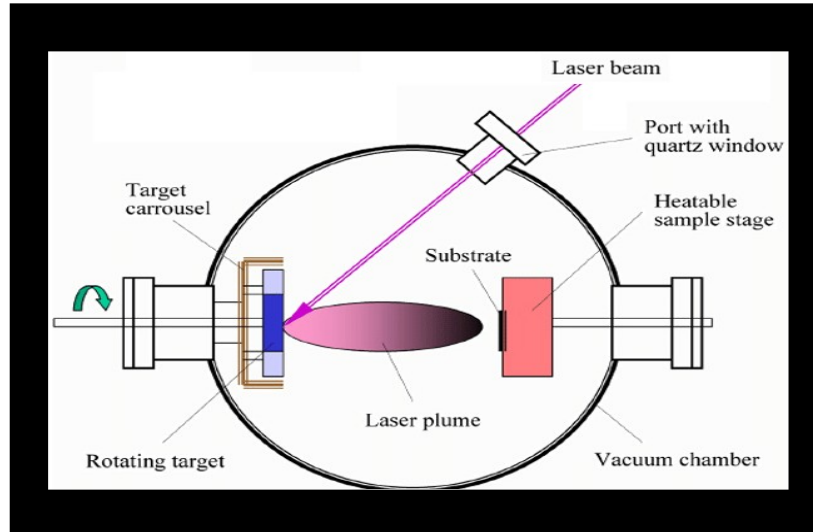


Figure 1.9 Schematic Diagram of PLD

PVD coatings deposited for numerous reasons. Some of the main ones are:

- Highly Improved hardness and wear resistance
- Reduced friction
- Improved oxidation resistance

The usage of this type of coatings are aimed at improvisation of efficiency through improved performance along with longer life of component. They might also permit coated components to drive in environments that the uncoated component will not other way are able to carry out.

1.7 ADVANTAGES OF THE PHYSICAL VAPOUR DEPOSITION PROCESS

- The Materials could be deposited with improvised properties in comparison with the substrate material

- Almost every sort of inorganic material could be used

The process is highly environmental friendly as compared to processes like electroplating.

1.8 DISADVANTAGES OF THE PHYSICAL VAPOUR DEPOSITION PROCESSES

- It has a line of sight technique which means that it is very difficult to coat under the cuts and same kind of surface attributes
- High capital cost
- Few processes drive at very high vacuums and temperatures requiring skilled operators
- Processes in need of large amounts of heat need appropriate cooling systems
- The rate of coating deposition is generally very slow.

CHAPTER 2 PULSED LASER DEPOSITION

The Pulsed laser deposition comes under physical vapor deposition method. In this type of method, the laser beam of high power pulsed focuses on the target that is in the vacuum chamber and the target material is further vaporized by a laser beam in the pattern of a Plasma Plume and deposited over the substrate like a thin film. This process could be performed in the environment of high vacuum or in the environment with background gases like oxygen. Oxygen is generally used for the process oxide deposition to wholly oxygenate the deposited thin film at the time of pulsed laser deposition (PLD) process.

While the equipment required to perform the deposition in this way is roughly alike other deposition methods (like sputtering), the physical interaction in the middle of the laser beam

and the target material and the creation of the thin film is way too much complex. When the laser pulse is absorbed by the target, its energy is first converted to electron excitation and then thermally, chemically and mechanically, resulting in vaporization, ablation, plasma formation and exfoliation of the target material. The particles separated from the Target surface are spread out in a vacuum environment as a plume. These highly energetic particles involve atoms, molecules, ions, electrons, and molten globules that would be deposited on the substrate.

The proper mechanism of the PLD process is complex, involving the process of target material ablation by laser irradiation, the formation of plasma plume along with energetic ions, electrons, atoms, molecules, and the charge-free particles and this process of the crystalline growth on the substrate.

2.1 PLD EQUIPMENTS

There are many parts which make up a complete PLD system each has its own specific purpose. First, an appropriate laser for the desired application must be selected. The second aspect of the system is the vacuum system, which includes a processing chamber, vacuum pumps and pressure sensors. This subsystem can provide an appropriate vacuum environment for the deposition. Finally, the substrate holder and an appropriate substrate heater constitute the third subsystem. Let's discuss this in brief:

- **Laser:** Pulsed laser can easily achieve the energies needed for a material to convert it into plasma. thin film growth can be done at various wavelengths but when considering a laser for PLD most thin film growth is done between 200nm and 400nm laser wavelengths. The base of this purpose for using these wavelengths is because the majority of the materials used for PLD have major absorption in these ranges.

- Deposition Chamber:** This vacuum subsystem has a multi-port vacuum chamber, two vacuum pumps and pressure sensors. The main chamber has six ports for the target and substrate stages, power supply, laser beam delivery, connecting sensors, and evacuating of the chamber. High vacuum to be achieved using a turbo-molecular pump, with which pressure below 10^{-9} Torr can be attained with room temperature. Operation of the turbo-molecular pump also requires a mechanical pump, with which pressure in the chamber can be reduced to 10^{-3} Torr.
- Substrate Holder and heater:** The substrate system contains a substrate holder and a substrate heater. The film substrate is held by a fastener that is made from two plates stickly attached with four screws to the plate connected to the shaft. To monitor substrate temperature, a thermocouple is inserted into the fastener. Two halogen lamps at the back of the plates work for the heat source to adjust the temperature of the substrate at the time of the deposition process. The temperature can be maintained by the heater and the temperature of the substrate up to $1000\text{ }^{\circ}\text{C}$. This entire system fixed on a linear shaft making the distance between the target stage and substrate holder adjustable.

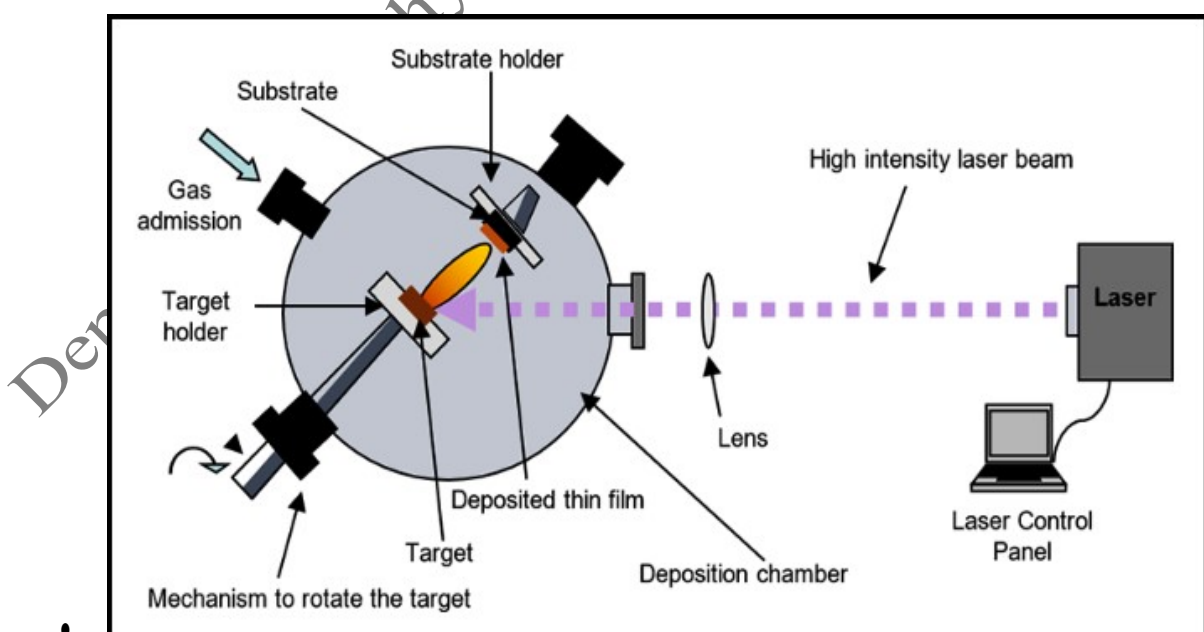


Figure 2.1 Pulse laser deposition schematic diagram

2.2 PRINCIPLE OF PULSED LASER DEPOSITION

The Pulsed Laser Deposition Technique includes of four main parts:

- Laser absorption at the target material surface, its ablation, and plasma formation.
- Plasma dynamic.
- Deposition of the material separated from the target on the substrate.
- Nucleation and growth of the thin film on the substrate surface.

2.2.1 Laser Beam-Target Interaction

To minimize the amount of energy lost due to carrier during absorption, short laser pulses with a wavelength strongly absorbed by the material are recommended. To avoid overlapping between the laser beam and the plume, the beam was irradiated onto the target at an incident angle of 45 degree. The absorbed energy causes the electrons in the material to oscillate. The fluence (time integral of laser intensity over the pulse duration) of the laser determines how much energy per unit area is absorbed by the target material. During film deposition, the average beam fluence is similar to heat required to the target material above its melting temperature and initiates evaporation. The collision cascade among the atoms in the target material, oscillation of the electrons, electron excitation, and electron-lattice (ions) energy transfer are the underlying principles behind evaporation of the target material upon laser beam absorption. When the energy delivered to a single atom by the laser beam exceeds its binding energy, the atom is ejected from the material's surface. The ejection of the atom is followed by a snapping sound and bright-coloured plasma (plume) of the emitted particles.

2.2.2 The Dynamics of Ablation of Materials and Plume Formation

Laser ablation has been analyzed using different models, namely mechanical, photochemical, thermal, photo physical and the defect model. Ablation was treated as the major mechanism in these models. The ablation process begins with the stimulation of a single or multiple phonons in the target material. The instantaneous conversion of excitation energy into heat changes the optical characteristics of the material as the temperature rises. Thermal ablation occurs when

the temperature of the target material's surface is raised without melting it. Depending on the target material and laser wavelength, the ablation threshold varies between 0.1 and 1 J/cm². When the ablation threshold of the target material is reached, ionization causes plasma (plume) formation instantaneously. The plume, which has a stoichiometry similar to that of the target material, is collected on a substrate placed close to the target.

2.2.3 The Evaporation of the Ablated Materials onto the Substrate

To a large extent, the thin film's quality is determined by how the ablated species evaporates onto the substrate. Different laser characteristics, such as laser energy, pulse repetition rate, and number of pulses, could have an impact on this. The energy of the ejected species arriving on the substrate can be influenced by these laser properties. When the average intensity of the laser beam exceeds the material's ionization threshold, a high plume density can form. It should be noted that at very high plume densities, damage to the substrate may occur. Three phases are involved in the interaction processes between the incident plume flow and the substrate. These include,

- (i) The sputtering of atoms by energetic incident plume from the surface of the substrate.
- (ii) The creation of a collision region between the incident plume and the sputtered atoms.

The collision region serves as a particle condensation source that initiates the final step.

- (iii) Film growth.

2.2.4 The Nucleation and Growth of a Thin Film on the Surface of the Substrate

On the substrate surface, there is a transition from the plasma (plume) phase to the crystalline (solid) phase during the nucleation and growth stage of the film. The laser intensity, pulse repetition rate, density and degree of ionization of the ablated material, substrate temperature, and physicochemical features of the substrate all play a role in crystalline thin film nucleation and growth. The substrate temperature (T_s) and the supersaturation (S) that occurs between the

plasma and solid phases of the material during crystallization are the two key thermodynamic factors involved in the growth mechanism. These two parameters are related by equation:

$$S = k_B T \ln(R/R_e)$$

where k_B is the Boltzmann constant, R is the rate of deposition and R_e is the equilibrium deposition value at temperature T . The equation illustrates that supersaturation is proportional to the temperature of the substrate. Large nuclei describe a low amount of supersaturation, resulting in the formation of distributed patches (islands) of films on the substrate's surface. The interstep distance between the islands grows as the supersaturation decreases, and the growing surface becomes smooth. The island density rises as the number of clusters impinging on the substrate surface rises, and the nucleus of the islands reduces to the atomic level as supersaturation rises. The islands arise as a result of the coalescence process as the supersaturation value rises (at high substrate temperature) (which is liquid-like for some cases). To initiate nucleation, a high rate of supersaturation is necessary. At a later stage, however, a low supersaturation rate is required to permit the formation of a single crystal film. Generally, there are three modes of thin film growth, namely island or the Volmer–Weber mode, layer-by-layer or the Frank–van der Merwe mode and layer plus island or the Stranski–Krastanov mode. Island or the Volmer–Weber mode: When the cohesion between the atoms of the target material is larger than the adhesion between the target atoms and the substrate, an island growth occurs, also known as the Volmer–Weber mode. As a result, the adatoms (atoms deposited on the substrate's surface) are more closely bonded to each other than to the substrate, resulting in cluster formation. This mode of growth is characterized by three-dimensional (3D) islands.

Layer-by-layer or the Frank–van der Merwe mode: When the adhesion between the adatoms and the substrate is greater than the cohesion between the adatoms, layer-by-layer growth occurs. The adatoms create smooth monolayers on the substrate's surface in this manner of growth, resulting in 2D growth. Layer plus island or the Stranski–Krastanov mode: layer plus

the island growth mode occurs when islands are formed after the formation of one or two monolayers on the surface of the substrate.

2.3 ADVANTAGES AND DISADVANTAGES OF PLD

ADVANTAGES

- The stoichiometric transferability of materials from Target to the substrate, the precise chemical composition of a complex material such as YBCO, can be reproduced in the deposited film.
- Deposition rate is relatively high (usually 100 angstroms per minute). Also, the thickness of the deposited thin film can be controlled simultaneously with the deposition process only by turning the laser on and off.
- The fact that a laser is used as an external energy source results in a very clean process without heat filaments.

DISADVANTAGES

- PLD is used less industrially as of now and most applications are limited to the research environment only. Three main reasons for this being:
- The plasma plume created is in the forward direction as a consequence of the nonuniform thickness of the particles accumulated on the substrate and the composition may change and extend along it of the created thin film and away from the centre of accumulation. The resulting film area is small.
- The plasma plume thus created has melt globules with an average diameter of 10 μm . These particles along with substrate deteriorate the quality of thin film.
- The processes that occur in plasma produced by the laser is not completely understood. Which re

sults in deposition of new material generally incorporates a period of experimental enhancement of coating parameters.

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CHAPTER 3

SYNTHESIS AND OPTIMIZATION OF THIN FILM

Here in this chapter we are going to discuss about the parameters used, experimental procedure and annealing of sample.

3.1 Experimental Procedure

In this experiment PtSe_2 material thin film was produced by pulse laser deposition technique that comes under physical vapor deposition using the substrate silicon, we have considered certain parameters and proceeded to perform experiment accordingly.

So, here certain parameters are mentioned like the laser used was krypton fluoride (KrF) excimer laser (lambda Physik compex Pro 201) was used to produce laser pulses of 10 ns at wavelength of 248 nm and energy of 0.2 joule/pulse, area of spot size was 0.04 cm^2 and the fluence of laser ablation was 1.0250 J/m^2 . The energy provided to laser was 350 megajoule, here to inform that because of fringing frequency deposition rate would vary and energy will tell the kinetic energy of ablated material, also it will tell the about the confined plume and area covered by it.

Now moving towards chamber, When laser fell the base pressure/vacuum taken was 5×10^{-6} torr and then with mass flow controller/organ cylinder we connected the chamber through pipe and this pipe is in a middle valve, we open it slowly and gradually after which pumping was done, thereafter using turbo rotatory pump we make vacuum inside the chamber, this turbo rotatory pump backed by rotatory pump where backing of vacuum was done by rotatory and main vacuum making was completed by turbo pump. This was done because turbo is a high-speed pump, if it was used directly the molecules of air would collide and bend its blade. So in initial phase we make the 10^{-3} torr vacuum from rotatory and then we switch to turbo and make 10^{-6} torr order of vacuum.

Now, that the energy is fixed, vacuum is generated setup is almost done, the laser beam focused at an angle of 45° onto the target vaporize an area of about 0.01mm^2 creating plasma plume that was emitted from the target. The evaporated target particle subsequently condenses on a substrate positioned opposite to the target. The target holder rotates such that subsequent laser pulses hit the target at different positions, preventing crater formation resulting from severe local heating and melting of the target material. The laser was made to fall on target that was loaded in chamber and in front of that approximately diagonally the substrate because we have to fix the substrate at a position that when ablation would happen from laser of target material that should go directly to substrate to the maximum value, plume coverage has to be towards substrate just so deposition take place more on substrate rather than chamber, here substrate target distance is 4cm

Moving forward, as the deposition started we had initially put the shutter in front of substrate so that first few shots would not directly fall onto substrate, in the first go pre ablation takes place to some shots then we fix the shots from motor that controls laser (that has few things that provide facility to set frequency, energy and no of shots, rotator control to know how much need is there to rotate the target to deposit the shots all over rather than one place or at one angle). Let's suppose now, pre ablation 200 shots were given once its done shutter will be removed and now as per the need 1000 shots were given or more will continue to ablate the material and deposition will remain happening.

By now the thick film will be deposited that would represent bulk like properties whose properties will match the properties of the target material.

Now, we started deposition so in first run the temperature that we fixed initially for substrate was 100°C , did its XRD, put the pressure 5 millitorr in chamber, gas we used was argon and was put in small amount/volume and slowly but PtSe₂ did not grow as per phase but we did not get the film as per requirement, then we started to increase temperature, again we failed to get the film precisely after checking its XRD, so many impurities were there, we further slowly increased the temperature till 500°C so at this temperature, impurities were less and PtSe₂ was

deposited properly and with good orientation, from starting we did not put the temperature at 500°C because we did not want selenium to get evaporated from it and there pumping also takes place continuously but it maintains itself at certain range so because of all this particular flow will remain maintained in chamber that will tell about its pressure that is 5 millitorr and it's like from one side you are evacuating the chamber and from another side you are inserting gas with controlled and certain flow so this maintains the particular pressure in the chamber.

Now we took out the substrate that is silicon (1,0,0) n type and to get two dimensional film we reduced no. of shots and got them deposited. And post annealing at 500°C for 2 mins we got tuned crystalline and physical properties. So overall a smooth thin film was successfully obtained

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3.2 DEPOSITION PARAMETERS

Parameters	Values
Target	Platinum diselenide (PtSe ₂)
Substrate	Si (110) (N-type)
Substrate Thickness	0.7 mm
Substrate-Target Distance	4 cm
Temperature	Varying (100°C - 550°C)
Base Pressure	5×10 ⁻³ Torr
Background Gas	Argon
Laser	Krypton fluoride (Krf)
Wavelength	248 nm
Frequency	5Hz
Pulse Energy	0.2 Joule per Pulse
Laser fluence	1.025 J/cm ²
Laser Shots	Varying (10,000 – 50 shots)

Table 3.1 Deposition Parameters used for PtSe₂ Thin Film

3.2 ANNEALING OF SAMPLE

Annealing plays a vital role to fabricate the thinfilm and to modify its structural properties, intrinsic stress liberation and to control thoroughness of surface of sample. Basically, annealing alter the microstructure and phases of the material. In this study, the sample was annealed in vacuum for 2 min at 500 degrees Celsius as the control of temperature couldn't be in direct control with the substrate, the measured temperature is a lower limit to ensure the growth of thin film was not influenced. Afterwards, the heater was switched off, and the sample under investigation was allowed to cool.

3.3 POSTEFFECTS OF ANNEALING

The effect of postannealing on thin film

- Crystalline property of the films were found to increase after annealing.
- The optical band gap of thin film increases due to the reduction of crystal defect
- Annealing effect improves the film quality with reduced roughness.

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CHAPTER 4

CHARACTERISATION TECHNIQUE

Samples were collected from the PLD system and stored in a dry air cabinet and characteristic were later carried out. These main techniques of structural, morphological, composition and optical measurements are discussed down. This chapter mainly tells about the principles and design of the X-Ray Diffraction (XRD) and X-Ray Reflectivity (XRR).

4.1 X-RAY DIFFRACTION AND ITS PRINCIPLE :

XRD Principle. When a crystal with an interplanar spacing d (crystal lattice constant) is irradiated by X-ray beam with a comparable wavelength λ , the X-ray diffraction, or the constructive interference between elastically scattered X-ray beams can be observed at specific angles 2θ when the Bragg's Law is satisfied. $n\lambda = 2d \sin \theta$.

X-ray diffraction is a powerful non-destructive characterization technique for characterizing crystalline materials. It gives information on crystal structure, observing phase composition, determine crystallite size, detect defects in samples, characterisation of polymers and many more. XRD peaks are produced by constructive interference of a monochromatic beam of X-ray scattered at specific angles from each set of lattice planes in a sample. The peak intensities determined by the atomic positions within the lattice planes. Consequently, the XRD pattern is the fingerprint of periodic atomic arrangements in a given material.

Powder X-Ray Diffraction (P-

XRD) is one such characterization tool that offers the advantage of simultaneous characterization of both the precursor and end products with a detailed qualitative presentation of their micro-structural behaviours.

XRD usually works in the principle of Bragg's Law

4.1.1 BRAGG'S LAW

Bragg's law states that when an X-ray is incident onto a crystal surface, its angle of incidence, θ , will reflect with the same angle of scattering, θ . And, when the path difference, d is equal to a whole number, n , of wavelength, constructive interference will occur. It is the best way to determine the structure of a material by passing it through X-rays. X-rays change their path when they strike an atom of the material.

Bragg's law gives the relation between wavelength and angle of diffraction.

The relation is $2d\sin\theta = n\lambda$, where n is the no of lines

d is spacing between lines

λ is wavelength of the X-ray

θ is the diffraction angle.

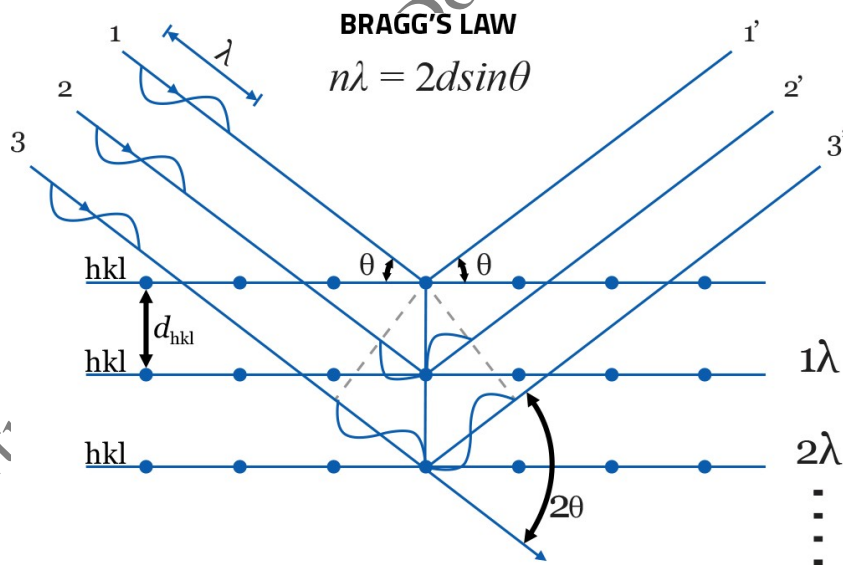


Figure 4.1 Schematic of XRD

4.1.2 CONSTRUCTIVE AND DESTRUCTIVE INTERFERENCE.

When the two rays are in precise phase, they combine and form an amplified ray, this is known as Constructive Interference. While, if two rays are out of phase, this results in the cancelling of the rays and is known as destructive Interference.

X-Ray detector notes the value of the rays at each 2θ angle and we obtain the diffraction direction of the lattice. This helps in the generation of the graphs and with the help of peaks we can determine the structure and properties of the sample material.

4.2 XRAY REFLECTIVITY

X-ray reflectivity (XRR) is a technique which is now widely used tool for the characterization of thin-film and multilayered structures. It is used to determine thickness, density and roughness for single and multilayer stacks on semiconductor wafers, XRR analysis can be performed on both crystalline and amorphous materials. At grazing angle of incidences, when X-rays applied to material's flat surface, total reflection occur at or below a critical angle, θ_c . This angle is exceedingly small and is referred to as the critical angle. The angle varies depending upon the electronic density of the material. The higher incident X-ray angle relative to critical angle, deeper the X-rays transmit into the material. With a material whose surface is ideally flat, the reflectivity suddenly decreases at angles above the critical angle in proportion to θ^{-4} .

If the material surface is rough, it causes a more drastic decrease in reflectivity. If such a material, serving as a substrate, is evenly overlaid with another material having a different electronic density, then reflected X-rays from the interface between the substrate and the thin film as well as from the free surface of the thin film will either constructively or destructively interfere with each other—resulting in an interference induced oscillation pattern. To first approximation, the intensity scattered by a sample is proportional to the square of modulus of the Fourier transform of the electron density. Thus, the electron density profile can be deduced from the measured intensity pattern, and subsequently the vertical properties (layer

thicknesses) as well as the lateral properties (roughness and correlation properties of interfaces or lateral layer structure) characterizing multilayers can be determined. Specifically, film thickness can be determined from the periodicity of the oscillation and information on the surface and interface from the angular dependency of the oscillation pattern's amplitude.

CHAPTER 5 RESULT AND DISCUSSION

The depositions of PtSe₂ thin films on silicon substrate was completed by the varying temperatures between 100°-

550°C inside the chamber and different laser shots were made to fall upon. The techniques of XRD and XRR was used to characterize the thin film of PtSe₂/Si through which we analysed the structure of crystal and purity of phase under varying temperature and laser shots and by XRR we can estimate the thickness of the sample. It was observed by XRD pattern that the sample synthesized till 300°C was not showing any diffraction peaks. Where with the increase in temperature up to 500°C diffraction peaks were obtained. few samples were taken at 500°C with different numbers of laser shots given to them. When the temperature was increased to 550°C, diffraction peaks were obtained but selenium starts to get evaporated. Therefore, for the rest of the samples the temperature was kept at 500°C. The no. of laser shots was varied. Table 1 shows the PtSe₂ samples obtained at different temperature and laser shots.

Table of Sample at varying Temperature and Laser shots

Sample	Pressure	Temperature	Distance	Laser Shots
1 0	5 mT	200° C	4 cm	5000
2 0	10 mT	100° C	4 cm	10,000
3 0	10 mT	300° C	4 cm	10,000
4 0	10 mT	400° C	4 cm	10,000
5 0	10 mT	400° C	4 cm	10,000
6 0	10 mT	500° C	4 cm	10,000
7 0	10 mT	550° C	4 cm	10,000
8 0	10 mT	500° C	4 cm	100
9 0	10 mT	500° C	4 cm	980
10 0	10 mT	500° C	4 cm	500
11 0	10 mT	500° C	4 cm	1000
12 0	20 mT	500° C	4 cm	1000
13 0	50 mT	500° C	4 cm	1000
14 0	5.0 mT	500° C	4 cm	300
15 0-15 20	7.2 mT	500° C	4 cm	600
16 0-16 20	7.2 mT	500° C	4 cm	500
17 0-17 20	7.2 mT	500° C	4 cm	150
18 0	7.2 mT	500° C	4 cm	50
19 0	7.2 mT	500° C	4 cm	20
20 0	7.2 mT	500° C	4 cm	10

The graph below shows the XRD pattern of the prepared samples of thin deposition at 500°C with different laser shots is shown in the graph.

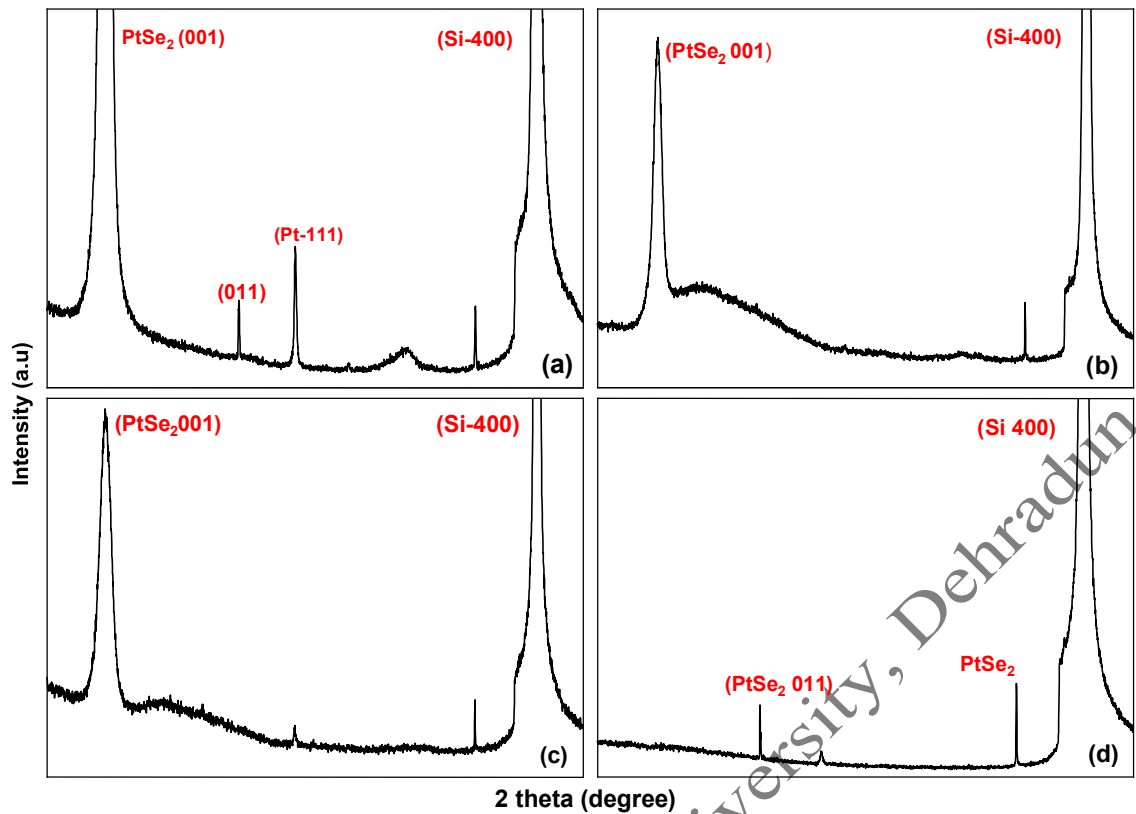


Figure 5.1 Graph shows the XRD peak of Samples (a) 9 θ , (b) 16-10, (c) 16-20, (d) 18 θ

The XRD graph peaks for Sample 9 θ , Sample 16 θ and Sample 18 θ shows the proper diffraction peak. The Sample 16-10 and 16-20 were the same sample but are kept in different position while deposition in order to check whether there will be any change in the XRD pattern if the position is changed. No particular changes were observed. Thus, it came into view that with the decrease in number of laser shots at 500°C more thin and uniform film was obtained.

The XRD pattern of the prepared sample in the figure shows the strong characteristic peaks. The main (001) characteristic peak of PtSe₂ is detected approximately at 17.6° degrees 2 θ and the other peaks were at 2 θ = 33.29°, 40.11°, 54.10° belonging to the (011), (111) and (102) planes of PtSe₂. The characteristic peak of Silicon substrate was detected at 2 θ = 69.35° in the plane (400). With the decrease in the number of laser shots the impurity peaks disappear and a uniform thin film was obtained over a substrate. The physical nature of 2D materials, PtSe₂, is determined by the layered

crystalstructure.

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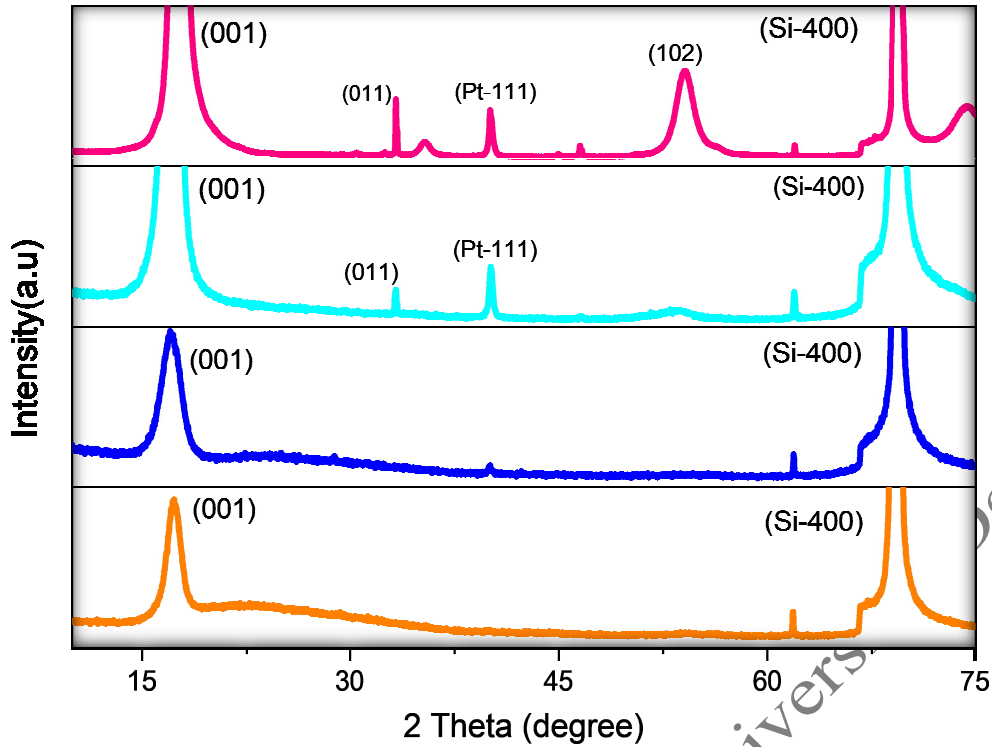


Figure 5.2 shows the graph of samples with different pulse shots

This PtSe₂ is determined by the layered crystal structure. The XRD pattern identifies the PtSe₂ film crystal lattice with unit cell parameters determined to be $a = 3.728\text{\AA}$ and $c = 5.06\text{\AA}$. The main (001) characteristic peak is detected approximately at 17.35° degrees 2θ . In this section an enlarged diffractogram sector (in the range $20\text{--}80^\circ$) is presented, showing the other peaks with diminished intensity as a consequence of the layered structure and dominant (001) orientation—implying a high crystalline quality and c-axis growth of the film plane.

X-RAY REFLECTIVITY

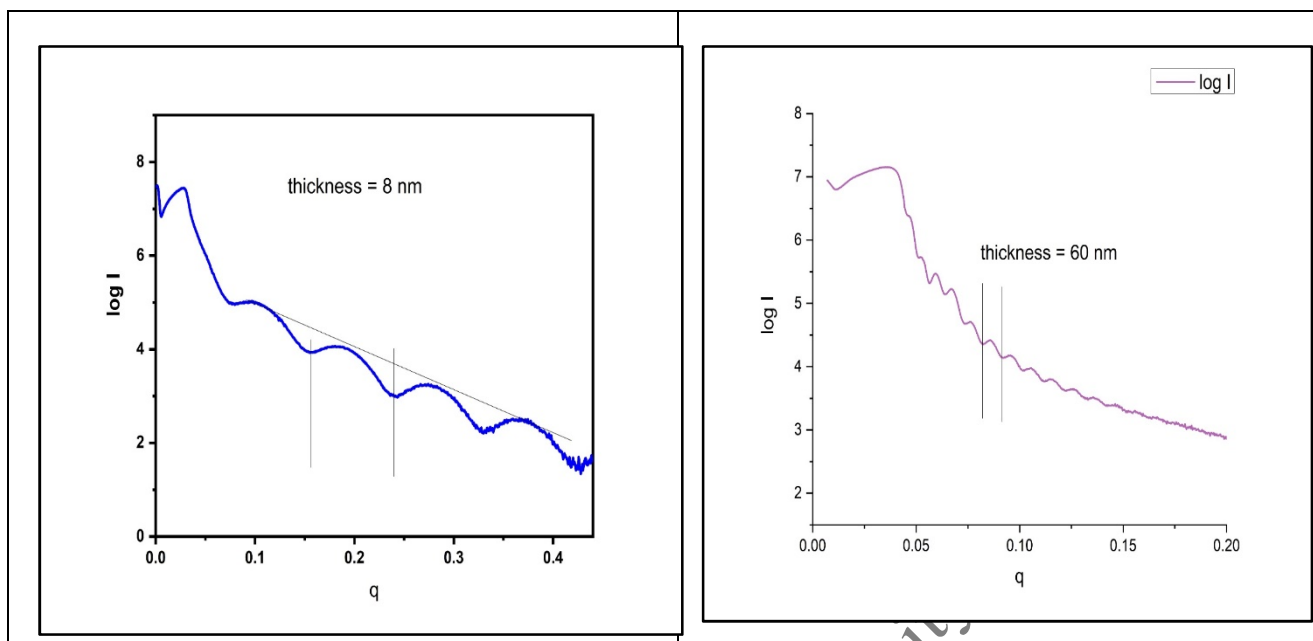


Figure 5.3 shows the XRR graph of sample (a) 16-2 θ (b) 9 θ

The graph between reflectivity and grazing angle of incidence is plotted. The Figure above represents the graph of two samples 16-2 θ and 9 θ . The sample 16-2 θ has a thickness of around 8nm and the graph 9 θ has a thickness of around 60 nm. The Y axis of XRR curve is shown in the logarithmic scale of normalized intensity of (I/I_0) . Interference occurs between the X rays reflected from the surface of the film and the interface between the film and the substrate. The reflectivity profile shows the oscillations caused by this Xray interference. The oscillation depend on Film thickness as thicker the film shorter the period of oscillations.

CONCLUSION

The aim of this thesis was the deposition of thin film PtSe₂ on a silicon substrate that was brought out by varying temperatures and laser shots and eventually by doing its annealing. The crystallinity, shape, and size of the sample were carried out.

We carried out the uniform thin film by Characterization through XRD and XRR. So overall, high-quality, well-crystalline, thin film of PtSe₂, through a pulse laser deposition technique under varying temperature and number of laser shots was successfully synthesized. It was seen through XRD that at 500°C when the laser shots were decreased a fine and precise thin film was deposited with no impurities. The sample was heated only till 500°C as the selenium present in PtSe₂ gets evaporated if heated more; therefore the temperature was increased slowly and in various runs. Thus, our study provides a new technique and method in expanding the family of transition-metal dichalcogenides (TMDs). The structural characterization of the obtained PtSe₂ confirms the composition and crystalline quality. The obtained results allow further ways for improvement of the preparation procedure to facilitate the nanostructures synthesis approach towards the 2D PtSe₂ applications. We have explained the wholesome review based, experimental and theoretical research evolution on 2D layered PtSe₂, covering the improvement, challenges, and prospects in future 2D material.

FUTURE SCOPE

Layered PtSe₂ possesses a multitude of advantages such as long-term air stability, low-temperature synthesis methods along with unprecedented photonic, physical and chemical properties, making it one of the most promising 2D materials for adoption by industries. Lately, this two dimensional (2D) platinum diselenide (PtSe₂) has attracted the attention of the research community due to its novel physical, chemical and electronic properties. Among 2D group-10 noble TMDCs materials, platinum diselenide (PtSe₂) came out as assured materials in photodetectors and for developing high speed electronics. Recently, due to their outstanding properties including widely tunable band gap, high carrier mobility, and phase control ability, PtSe₂ has become highly engrossing in the 2D materials research, much effort has focused on exploiting it for applications in a range of fields, particularly in ultrafast and nonlinear photonics. 2D PtSe₂ has evolved greatly in many areas such as photocatalytic, pressure gas sensors, electronic, and optoelectronic devices. One of the important applications of 2D PtSe₂ materials is the field effect transistors (FETs). As a saturable absorber, PtSe₂ has been used in the cavities of a range of lasers including fibre, solid-state and waveguide lasers. 2D PtSe₂ has been theoretically predicted to be a promising candidate to fabricate high performance electronic and optoelectronic devices. There are still outstanding challenges and much research work will need to be done in the future before making full use of this layered material. Many PtSe₂ synthesis methods can be carried out at low temperature but with relatively low efficiency and crystallinity. It can further be used to obtain high quality thin film with large size single crystalline films.

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*laserdepositedNiOfilmsviaannealingandlithiumdopingforimprovingperovskites
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