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Thin film Deposition Methods: A Critical Review

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Abstract: The aim of this review paper is to present a critical analysis of existing methods of thin film deposition. Paper discusses some thin film techniques which are advanced and popular. The advantages and disadvantages of each method are mentioned. The two major areas of interest discussed are physical and chemical vapor deposition techniques. In general, thin film is a small thickness that produces by physical vapour deposition (PVD) and chemical vapour deposition (CVD). Despite the PVD technique has a few drawbacks, it remains an important method and more beneficial than CVD technique for depositing thin films materials. It is examined that some remarkable similarities and difference between the specific methods. The sub methods which are having common principle are classified. The number of researchers attempted to explain the how the specific method is important and applicable for the deposition of thin films. In conclusion the most important method of depositing thin films is CVD. For our research work the Spray Pyrolysis technique, which is versatile and found suitable to use.

Keywords: Thin films, CVD, PVD, advantages and Disadvantages of CVD and PVD.

I. INTRODUCTION

Thin film deposition is the process of creating and depositing thin film coatings onto a substrate material. These coatings can be made of many different materials. Thin film coatings also have many different characteristics which are leveraged to alter or improve some element of the substrate performance. 'Thin' is, of course, a relative layer, but in most cases, the techniques of thin film deposition are designed to produce layers which are only a few tens of nanometers thick. In today's world, thin films are commonly thought of in connection with semiconductors. However, thin films are important in a multiplicity of other areas where coatings only a few microns thick are needed. The thin film is important because it changes the surface interactions of the newly formed platform from the bulk substrate properties. Thin films have a range of properties depending on those types are-

1) Optical thin films
2) Electrical or electronic thin films
3) Magnetic thin films
4) Chemical thin films
5) Mechanical thin
6) Thermal thin films

The electron microscopy techniques can be used to 'see' thin films. Various refinements of these methods have been used such as scanning electron microscopy (SEM). Thus, the wavelength of light is not a hard and fast rule either when it comes to defining thin films [1].

II. THIN FILM DEPOSITION TECHNIQUES

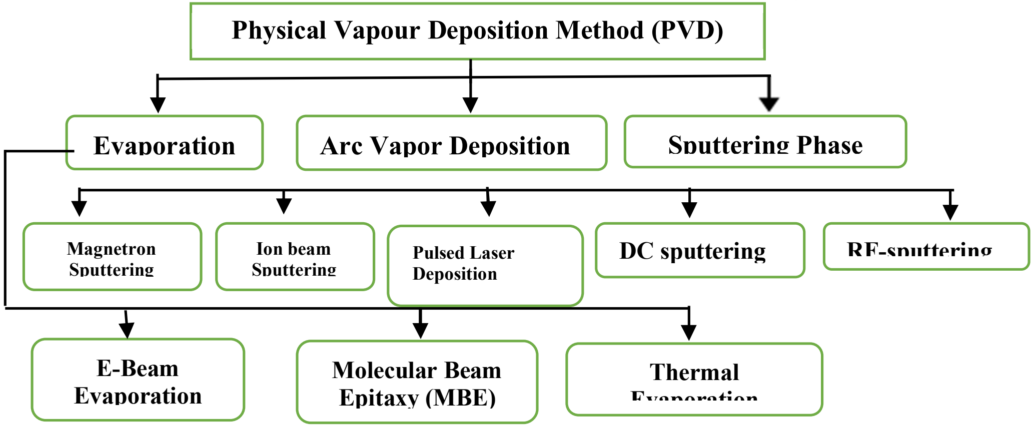


Fig.1.Classification of PVD techniques

Over the years, various materials have been synthesized in the form of thin films due to their prospective technological significance and scientific interest in their properties. They have very extensive range of applications and extend from nanostructures to coatings of several square meters on window glasses. Several techniques have been inspected in the search for the most reliable and inexpensive method of producing the thin films. Based on the nature of the deposition process, the techniques employed for thin film deposition can be classified into two groups, namely, physical and chemical deposition processes [2].

III. COMPARATIVE STUDY OF PVD AND CVD

The deposition process is classified into two types systems physical vapour deposition (PVD) and chemical vapour deposition (CVD), depending on the different principle causing film deposition. The table 1 shows the difference between them. The key difference between PVD and CVD is that the coating material in PVD is in solid form whereas in CVD it is in gaseous form.

TABLE.I.
Difference Between PVD AND CVD

Parameter	Physical Vapour Deposition (PVD)	Chemical Vapour Deposition (CVD)
Definition	Physical vapor deposition (PVD) is a series of vacuum deposition methods used to produce films and coatings. PVD is characterized by a process in which the material changes from a condensed phase to a gas phase and then back to the condensed phase of the film (physical process). The most common PVD processes are sputtering and evaporation.	Chemical vapor deposition (CVD) is a vacuum deposition method. In a typical CVD, the wafer (substrate) is exposed to one or more volatile precursors that react and/or decompose on the surface of the substrate to produce the desired deposit. Volatile by-products are also typically produced which are removed by the gas stream through the reaction chamber.
Application	PVD is used to make articles that require films for mechanical, optical, chemical or electronic functions. For example, semiconductor devices, thin-film solar panels, glass coatings, and etc.	Chemical vapor deposition (CVD) is used to produce high quality, high-performance solid materials. This process is commonly used in the semiconductor industry to produce thin films.
Temperature	Relatively low	The process temperature of CVD method is very high, usually exceeds the tempering temperature of the high speed steel. Thus in order to restore the hardness, the tools must be subjected to vacuum heat treatment after coating.
Cleaning Requirement	High	Low
Film thickness	about 2.5 µm	about 7.5 µm
Surface	The PVD coating well reflects the surface of the tool and has a good metallic lustre without grinding.	The surface of the CVD coating is slightly rougher than the surface of the substrate.
Crafting Process	Nearly all PVD technologies have poor coating performance both on the back and sides of the tool due to low air pressure. The PVD reactor must reduce the loading density to avoid shadow formation, and the loading and fixing are complicated.	CVD occurs in a low-vacuum gaseous environment and has good coating performance. Therefore, except for the support points, all the surfaces of the cutters sealed in the reactor, even deep holes and inner walls, can be completely coated.
Cost	More expensive	Less expensive
Safety	PVD is a kind of "green engineering", which produces little pollution during the process because it is "physical".	The reactive gas and reaction tail gas of CVD may have certain corrosiveness, flammability and toxicity, and there may be powdery and fragmented substances in the reaction tail gas.

IV. PHYSICAL VAPOUR DEPOSITION (PVD) METHOD

PVD stands for Physical Vapor Deposition. PVD Coating refers to a variety of thin film deposition techniques where a solid material is vaporized in a vacuum environment and deposited on substrates as a pure material or alloy composition coating. It evaporates or sputters a material, producing a gaseous plume or beam that deposits a film on the substrate. It describes a group of thin film deposition techniques that involve vaporizing a solid material in a vacuum, then depositing that material onto a substrate. Coatings created in this manner are highly durable, and resistant to scratching and corrosion. PVD is useful in the production of devices ranging from solar cells to eyeglasses to semiconductors [3].

The benefits of PVD are numerous, and include the creation of a hard coating that is resistant to corrosion and scratching. PVD also creates thin films that can tolerate high temperatures. Potential drawbacks of PVD include cost, as these methods may require a larger investment than other thin film deposition processes. The cost can also vary among PVD methods themselves. These are environmentally friendly or “plating” technique that greatly reduces the number of toxic substances that must be used, manage and disposed of as compared to other “wet” processes that involve fluid precursors and chemical reactions used to achieve the same result. Because it is capable of producing extremely pure, clean and durable coatings, Physical Vapor Deposition is the technology of choice for the surgical and medical implant industry [4].

The most common types of physical vapor deposition (PVD) are evaporation and magnetron sputtering.

A. Evaporation

Evaporation methods are considered as the common deposition of materials in the form of thin-layer films. The general mechanism of these methods is obtained by changing the phase of the material from solid phase to vapor phase and converting again to solid phase on the specific substrate [5-6]. It takes place under vacuum or controlled atmospheric condition. There are two types of thermal evaporation: pulsed laser deposition and electron beam deposition. Both processes use energy to evaporate a metal material (such as Titanium, Zirconium, Chromium, Aluminium, or Copper) into a vacuum. The vacuum then allows vapor articles to travel to the relatively cooler production parts where it will once again condense and crystallize into a thin, hardened, metal state. This PVD type is most commonly used in computer industry microfabrication or for products such as film packaging [7].

There are multiple types of evaporative deposition:

1) E-Beam Evaporation (Electron Beam)

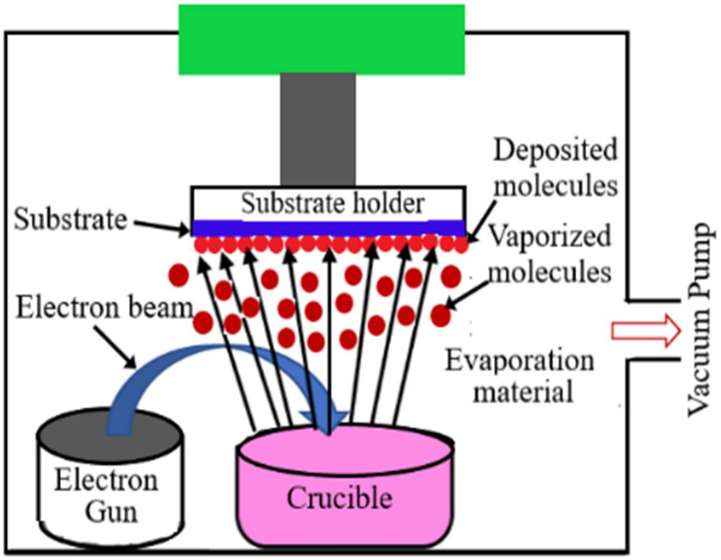


Fig.2.Schematic diagram of electron beam evaporation

In this process, a highly-charged electron beam evaporates the target material. The evaporated material is then deposited onto the substrate, and the atoms formed create the thin film. This process is often used for optical thin films such as solar panels, glasses and architectural glass. In this method the intensive beam of electrons is generated from a filament and steered through both electric and magnetic fields to hit the target and vaporize it under vacuum environment as shown in figure 2. Thin films prepared by electron beam evaporation are of good quality and purity [8].

2) *Molecular Beam Epitaxy (MBE):* It is one of the several methods of depositing single crystals and invented in the late 1960s.In the process of epitaxy, a thin layer of material is grown on a substrate. With respect to crystal growth, it applies to the process of growing thin crystalline layers on a crystal substrate. The deposition of single crystal films by the condensation of one or more beams of atoms and molecules from Knudsen sources under ultra-high voltage (UHV) condition is called molecular beam epitaxy [9].

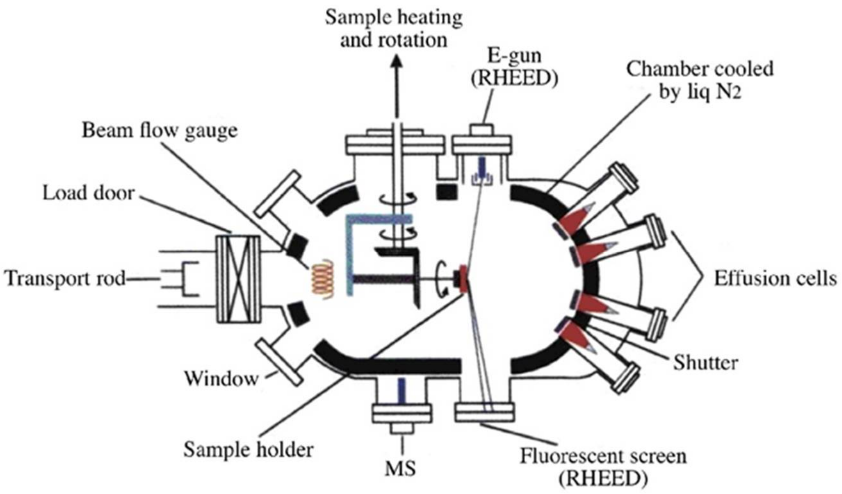


Fig.3. shows the experimental set up of molecular beam evaporation

Epitaxial growth takes place due to the interaction of molecular or atomic beams on a surface of a heated crystalline substrate. Molecular beam epitaxy takes place in high vacuum or ultra-high vacuum (10−11torr). The Knudsen effusion source consists of a metallic chamber, containing the evaporant with a small orifice. The orifice dimension is smaller than the mean fee path of the vapor in chamber [10]. Flow of the molecules from source is by effusion. The effusion molecular beam has a large mean free path compared to source substrate distance. The flux of beam is precisely determined by the partial pressures of the vapor species within the chamber, their molecular weight, and source temperature and orifice dimension. The beam is directed on the substrate by orifice slits and shutters. By controlled these shutters, one can grow superlattices with precisely controlled uniformity, lattice match, composition, dopant concentrations, thickness, and interfaces down to the level of atomic layers.

A typical MBE chamber is shown in Figure 3. The substrate is chemically washed and then put into a loading chamber where it is further cleaned using argon ion bombardment followed by annealing. This removes the top layers of the substrate, which is usually an undesired oxide which grew in air and contains impurities. The annealing heals any damage caused by the bombardment. The substrate then enters the growth chamber via the sample exchange load lock. It is secured on a molybdenum holder either mechanically or with melted indium or gallium which hold the substrate by surface tension. Each effusion cell is a source of one element in the film as shown in figure3.

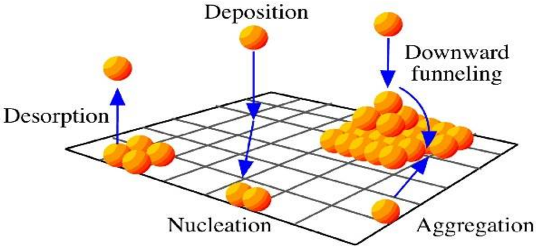


Fig.4. Schematic illustration of processes on growing surface during MBE. Adsorption of atoms on the surface, surface diffusion of atoms, formation of crystalline lattice, desorption of particles from the surface.

Advantages: complex layers or superlattices, low temperature requirement epitaxy (Si: 400 - 800°C, GaAs: 500 - 600°C)
Disadvantages: limited product throughput, complex operation, expensive equipment. Also in Molecular beam epitaxy the substrate rotation is required for uniformity in thickness and conformity.

3) Thermal evaporation

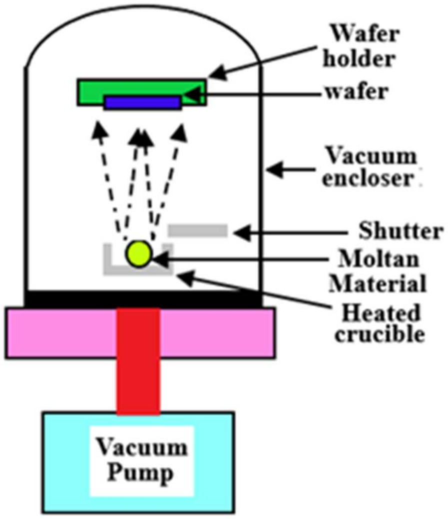


Fig.5. Schematic diagram of thermal evaporation system

In this simpler form of PVD, a resistive heat source heats the target material until vapor pressure is produced. The evaporated material then coats the substrate, forming the thin film. It is used to deposit metals such as silver and aluminium for OLEDS, solar cells and thin film transistors.

B. ARC Vapor Deposition

In Low-temperature arc vapor deposition (LTAVD) process uses a low-voltage arc to evaporate metal source material into vaporized metal particles. These evaporated metal atoms combine with reactive gas molecules in a plasma state that once again condense on relatively cooler production parts in close proximity. Production parts spin on a multi-axis rack carousel to produce an evenly distributed lifetime coating in colours that include hues of black, bronze, gold, graphite, nickel, blue, purple, and “rainbow” combinations of more than one colour.

LTAVD is the most environmentally friendly method, and it results in a very thin (0.25 to 4.0 microns) hard metallic coating that is available in a wide variety of colours. PVD finish has a transparent quality that allows underlying chrome or polish to shine through. Matte and brushed metal finishes also receive PVD to lock in these desired physical appearances for a lifetime of corrosion, chemical, and scratch resistance.

C. Sputtering

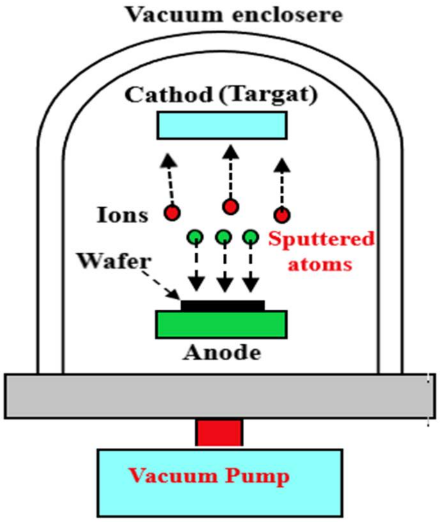


Fig. 6. Schematic of sputtering process

Sputtering involves the bombardment of a target material with high energy particles that are to be deposited on a substrate like a silicon wafer or solar panel. The substrates to be coated are placed in a vacuum chamber containing an inert gas, usually Argon and a negative electric charge is placed on the target material to be deposited causing the plasma in the chamber to glow. Atoms are “Sputtered off” the target by collisions with the Argon gas atoms, carrying these particles across the vacuum chamber and are deposited as a thin film. Several different methods of vapor deposition methods coating systems are widely used, like magnetron sputtering, DC sputtering, RF sputtering, Ion beam sputtering, Pulse Laser Deposition etc. [11].
Ion beam sputtering and magnetron sputtering. In the former, an ion beam directs a high electric field toward the surface of the material to be vaporized. This causes the metal vapor gases to ionize after which momentum transfer directs those ions toward the target production part. In magnetron sputtering, positively charged ions are accelerated by an electrical field and then superimposed onto the target parts. This process is commonly used in the medical industry for manufacturing lab products and optical films.
The disadvantage of sputtering is -
Sputtering rates are low compared to those that can be attained in thermal evaporation.

- Sputtering targets are often expensive and material use may be poor.
- Most of the energy incident on the target becomes heat, which must be removed.
- In reactive sputter deposition, the gas composition must be carefully controlled to prevent poisoning the sputtering target.

1) *Magnetron Sputtering:* Sputter deposition, commonly called sputtering, removes atoms/molecules from a solid target's surface, projecting them into the gas phase from which they condense on another surface. In contrast to the various evaporation techniques available, sputtering does not require melting of the base material, rarely "spits" lumps at the substrate, and the source can be mounted in any orientation. There is comparatively very little radiated heat transferred to substrates, the coverage is not strictly line-of-sight, and elemental mixtures, alloys, and compounds can be sputtered without changes in stoichiometry. It is plasma-based coating technique. In this process, magnetically confined plasma is created near the surface of the target material. Ions from that plasma collide with the target material, and the atoms ejected from those collisions are what are “sputtered”, or deposited onto the substrate to create the thin film. It is often used for metallic or insulating coatings for optical and electrical purposes. Because the plasma is both electrically neutral and highly conductive, there is little voltage drop across it. The voltage drop occurs across thin "dark space" regions. The target's negative potential attracts positive ions from the plasma's edge, which in turn hit the target with sufficient kinetic energies to eject neutral target atoms/molecules by energy transfer. While traveling from target to substrate, each ejected atom hits numerous gas atoms/molecules that deflect them and cause energy loss. By optimizing the target-substrate distance, the atoms approach the substrate's surface from partially randomized directions, producing a uniform film thickness across a textured substrate's surface. The advantage of Magnetron sputtering is-

- Excellent precision of film thickness and density of the film. Also it achieve denser coatings than evaporation
- Perfect for metallic or insulating coatings with specific optical or electrical properties
- Can be configured with multiple magnetron sources

The disadvantages of magnetron Sputtering are an erosion track in the target, this leads to poor efficiency of sputtering yield versus target volume compared to non-magnetron sputtering also non-uniform removal of particles from target result in non-uniform films on substrate.

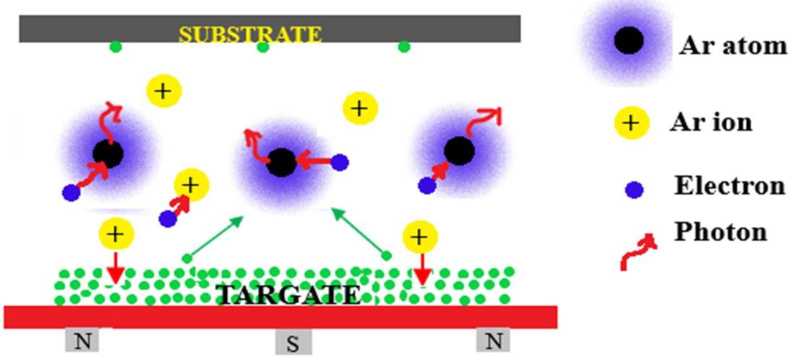


Fig.7. Spatial distribution of particles inside the high vacuum sputtering chamber.

The basic process of sputtering involves a process gas (in our case argon, Ar) which is introduced inside the high vacuum chamber. An electric field is applied between the target and the substrate which leads to the creation of plasma with the positive Ar+ ions accelerated towards the cathode (target) located on the bottom of the chamber. Particles are detached from the target due to the momentum transfer of Ar+ ions when colliding with the target material, contained within a plasma plume similar to a flash light or a cone resulting in deposition of film on the substrate situated just above the cathode. Free electrons generated during the process are confined close to the target material using a magnetic field created by a magnet ring located just behind the cathode (target). It is then less probable that these ions recombine with free electrons in their way to the target, leading to an increased sputtering rate [12].

2) *Ion Beam Sputtering:* This process uses an ion source to sputter the target material—either metallic or dielectric—onto the substrate, forming the thin film. Thin films created through ion beam sputtering are of a high quality, and have very precise thickness due to the monoenergetic ion beam used. This process is often used to create coatings for precision optics and semiconductors, where the utmost precision is required. Ion beam directs a high electric field toward the surface of the material to be vaporized. This causes the metal vapor gases to ionize after which momentum transfer directs those ions toward the target production part.
3) *Pulsed Laser Deposition (PLD):* Pulsed-laser deposition (PLD) is another physical deposition technique to deposit the thin film coating system [13]. During the thin-film deposition process, the laser beam is used to ablate the material for depositing the thin films inside a vacuum chamber as shown in Figure 8. Pulsed laser deposition is a type of evaporation process that uses laser pulses to evaporate the target material. This produces a plume of plasma that then deposits onto the substrate, forming the thin film. While optimizing this process can require more time and effort than other methods, due to the many variables at play, its benefits include high deposition rates and a streamlined process, as well as no filaments for easier maintenance. In the case of metal oxide thin films, oxygen is used to deposit the oxides of metals [14]. The thin-film quality from the PLD depends on the various parameters such as wavelength of the laser, energy, ambient gas pressure, pulsed duration, and the distance of the target to the substrate [15]. The ablation process during the deposition may control and monitor by using laser-induced fluorescence [16], laser ablation molecular isotopic spectroscopy [17], and optical emission spectroscopy [18].

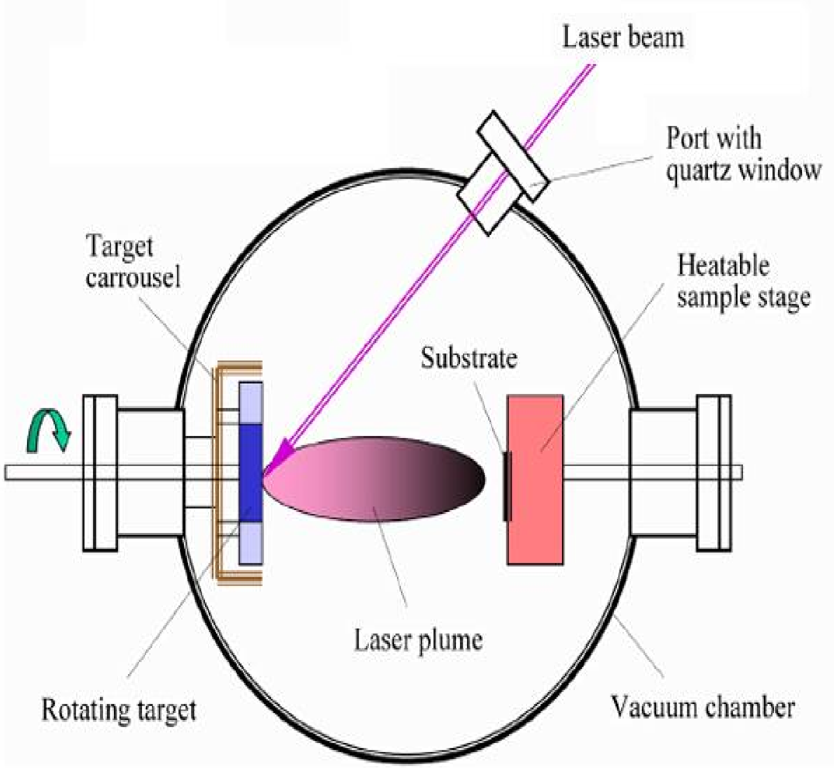


Fig. 8: A schematic diagram of the pulsed laser deposition setup.

4) *DC Sputtering:* This is a very straight forward technique of the deposition, in which target is held at high negative voltage and substrate maybe at positive, ground or floating potential. Substrates may be simultaneously heated or cooled depending upon the requirement. Once the required based pressure is attained in the vacuum system, usually argon gas is introduced at a pressure < 0.1 Torr. or visible glow is offered and current flows between anode and cathode indicating deposition on set. When sufficiently high voltage is applied between anode and cathode with gas in it, a glow discharge is set up with different regions as Cathode glow, Crook's dark space, negative glow, Faraday dark space, positive column, anode dark space an anode glow. These regions are result of plasma, i.e. mixture of electrons, irons, neutrals and photons released in various collisions. The density of various particles and the length over which they are spread and distributed depends upon the gas pressure. Energetic electron impacts cause gas ionization. Ratio of ions/neutrals can be typically 10^{-4}. Thus at a few milli torr (mTorr) pressure, sufficiently large number of ions are generated that can be used to sputter the target.

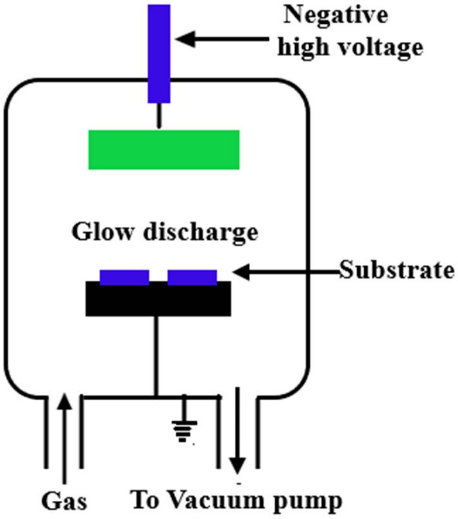


Fig.9. Schematic of DC sputtering unit

5) *RF Sputtering*

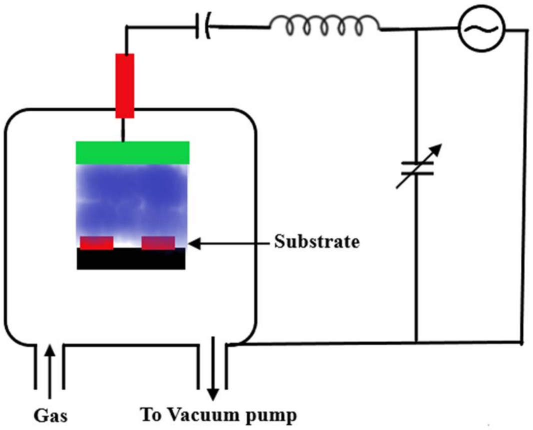


Fig.10.R F Sputtering set up with required network

If the target is to be sputtered is insulating, it is difficult to use DC sputtering. This is because it would mean the use of exceptionally high voltage (>10^6 V) to sustain discharge between the electrodes. In DC sputtering, 100 to 3000 Volts is usual voltage. However, if some high frequency lower voltage is applied, the cathode and anode alternatively keep on changing the polarity an oscillating electrons cause sufficient ionization. In principle, 5 to 30 MHz frequency can be used and electronics can be insulating. However, 13.56 MHz is commonly used frequency for deposition, as it is reserved worldwide for this purpose and others are available for communication. Target itself biases to negative potential becoming cathode when the arrangement is depicted.

Table II
Difference Between Evaporation And Sputtering

Parameter	**Evaporation**	**Sputtering**
Rate	1000 atomic layer/sec (Thickness control is difficult)	1 atomic layer/sec (thickness control possible)
Choice of Material	Limited (to those with low melting point)	Almost unlimited
Purity	Better	Possibility of incorporating impurity
Alloy composition	Little or no control	Can be tightly controlled
Changes in source Material	Easy	Expensive
Decomposition	High	Low
Adhesion	Often poor	Very good

V. CHEMICAL VAPOR DEPOSITION (CVD)

A. Chemical Vapor Deposition (CVD)

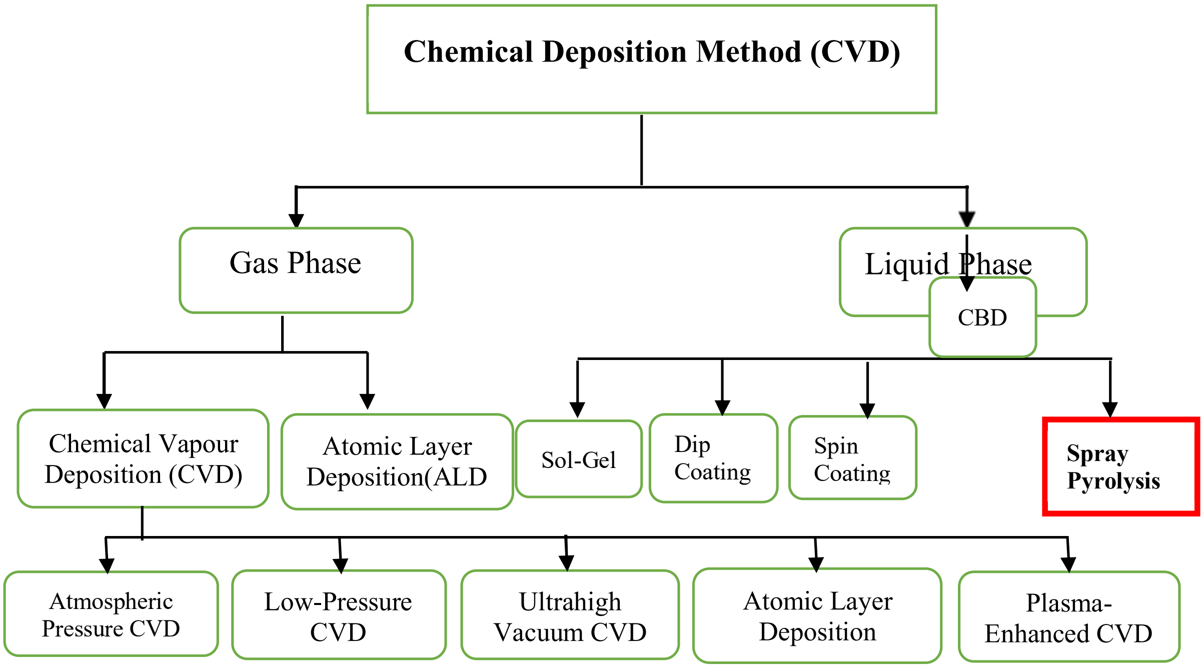


Fig.11. Classification of chemical thin film deposition methods [27]

The chemical methods comprise gas phase and liquid phase deposition methods. The gas phase methods are chemical vapor deposition (CVD) [19-20] and atomic layer epitaxy (ALE) [21], while spray pyrolysis [22], sol-gel [23], spin- [24] and dip-coating [25] are liquid phase methods.

The CVD method uses reactive, volatile compounds that decompose on a heated substrate. The starting materials are often organo- or hydrido-compounds that pyrolyse at relatively low temperatures into a non-volatile (film) component and a pumpable vapor/gas. Both methods sub-divide into a variety of techniques with auxiliary mechanisms to achieve some goal. In this process substrate is placed into a vacuum chamber, two chemical precursors are heated, which causes them to vaporize. When they meet on the substrate surface, a chemical reaction occurs to form a high-performance thin film coating. CVD is useful in creating coatings for a wide variety of applications including medical devices, automotive components and silicon wafers [26]. Benefits of CVD methods include the ability to use these processes on a wide variety of substrates, as well as the ability to coat intricate or complex topographies. Thin films created through CVD also typically maintain their bonds well in high-stress environments.

The liquid phase methods for the preparation of thin films have been studied extensively owing to their simplicity and inexpensiveness and such processes facilitate the designing of materials on a molecular level.

The CVD is the process to chemically reacting a volatile compound of a material to be deposit, with other gases to produce a non-volatile solid that deposits automatically on the surface of a substrate.

There are many possible reactions and the chemistry is the most important component of this method. Gas phase and surface reactions are intrinsically mixed in this method. The wide range of possible reactions can be grouped into pyrolysis, reduction, oxidation and compound formation

Examples of the above chemical reactions are:

1) Pyrolysis or thermal decomposition

$SiH_4(g) \rightarrow Si(S) + 2H_2(g)$ at 650^0C

$Ni\ (CO)_4 \rightarrow Ni(s) + 4CO(g)$ at 180^0C

2) Reduction

$SiCl_4(g) + 2H_2\ (g) \rightarrow Si(s) + 4HCl(g)$ at 1200^0C

$WF6 + 3H2(g) \rightarrow W(s) + 6HF\ (g)$ at 300^0C

3) Oxidation

$SiH_4(g) + O_2(g) \rightarrow SiO_2(s) + H_2(g)$ at 450^0C

$4PH_3(g) + 5O_2(g) \rightarrow 2P_2O_5(s) + 6H_2(g)$ at 450^0C

4) Compound formation

$SiCl_4(g) + CH_4(g) \rightarrow SiC\ (s) + 4HCl\ (g)$ at 1400^0C

$TiCl4\ (g) + CH4(g) \rightarrow TiC(s) + HCl\ (g)$ at 1000^0 C

The versatility of CVD process is further demonstrated by the fact that several reactants or precursors can be used to deposit a given film. For example, silica films can be obtained by either of these possible reactions.

- $SiH_4\ (g) + O_2\ (g) \rightarrow SiO_2(s) + 2H_2$
- $SiH4(g) + N_2O(g) \rightarrow SiO_2(s) + 2H_2 + 2N_2$
- $SiH_2Cl_2(g) + 2N_2O(g) \rightarrow SiO_2(s) + 2HCl(g) + 2N_2(g)$
- $SiCl6(g) + 2N2O(g) \rightarrow SiO2(s) + 3Cl2(g) + 2N_2(g)$
- $Si\ (OC_2H_5)_4\ (g) \rightarrow SiO_2(s) + 4\ C_2H_4(g) + 2H_2\ O\ (g)$

A variety of CVD reactors can be constructed depending on the type of precursors used in the chemical reaction and the deposition conditions. When metalorganic compounds are used as precursors, the method is referred as MOCVD (Metalorganic Chemical Vapor Deposition), and when plasma is used to enhance the reaction is called PECVD (plasma enhanced CVD).

B. Atomic Layer Deposition (ALD)

Atomic layer deposition, ALD, is a thin film technology that enables new and highly competitive products. ALD is also a powerful resource for advanced nanotechnology research. Typical applications of ALD contain a requirement to manufacture very precise nanometre–thick, pinhole–free and totally conformal thin films on any shape and geometry. For today’s businesses, Beneq ALD offers the necessary tools to accelerate growth, by means of new and innovative applications, production equipment you can count on and affordable cost of ownership.

ALD is based on surface controlled thin film deposition. During coating, two or more chemical vapours or gaseous precursors react sequentially on the substrate surface, producing a solid thin film. Most ALD coating systems utilize a flow–through traveling wave setup, where an inert carrier gas flows through the system and precursors are injected as very short pulses into this carrier flow. The carrier gas flow takes the precursor pulses as sequential “waves” through the reaction chamber, followed by a pumping line, filtering systems and, eventually, a vacuum pump.

C. Disadvantage of ALD

1) The ALD process is a high precision process and this often leads to high precursor gas usage and energy.

2) Approximately 60% precursor dosage is wasted in the ALD process which implies that most of the energy has been wasted.

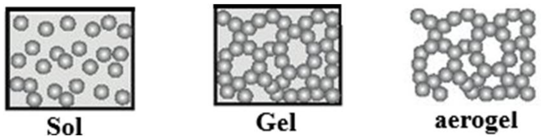
3) Another drawback in ALD for commercial use is the cost-effectiveness which is principally due to its deposition rate; however, this challenge has been partially overcome using spatial atmospheric ALD [28].

D. Gas Phase CVD

1) *Atmospheric Pressure CVD:* In this process, which takes place at normal, or atmospheric, pressure and a lower temperature than other methods, the substrate is exposed to at least one volatile precursor. The precursor(s) react on the surface of the substrate to deposit the thin film. It can be used to deposit doped and undoped oxides, and the deposition is fairly quick. Thin films produced by this method are low-density and have moderate coverage.
2) *Low-Pressure CVD:* In low-pressure CVD, heat is used to break down a precursor gas inside the chamber where the reaction will take place. This causes the reactive gas to react with the substrate when it is injected into the chamber, and this reaction creates the thin film coating. Low-pressure CVD is commonly used for the deposition of materials including polysilicon and silicon nitride, and can be useful for batch processes. Coatings created with this process are more uniform and feature fewer defects, but the process requires a higher temperature which can limit the materials available to use.
3) *Ultrahigh Vacuum CVD:* In ultrahigh vacuum CVD, the substrate is exposed to precursor gases in an ultrahigh vacuum near about (<10^ {-6} Pa). These precursors then react and deposit onto the substrate, forming the thin film.
4) *Atomic Layer Deposition:* While atomic layer deposition falls under the CVD umbrella, it differs in that precursor materials are kept separate during the reaction. In this process, the reaction occurs due to sequential pulsing of precursor vapours, one atomic layer is formed during each pulse. Pulses are repeated until the thin film reaches its desired thickness. Benefits of atomic layer deposition include high quality defect-free coating, as well as improved thickness uniformity.
5) *Plasma-Enhanced CVD:* It is a lower-temperature alternative to standard CVD, and is often used in the production of electronic devices. One common application for PE-CVD coatings is to protect these devices from corrosion. In plasma-enhanced CVD, a pair of reactive gases are excited to create a plasma. This causes a chemical reaction that results in the thin film being deposited onto the substrate

E. Liquid Phase CVD

1) Sol-Gel



The name suggests that there are two types of materials 'Sol' and 'Gel'. The sol-Gel is synthesized by M. Ebelman in 1845. Sol-gel formation is low temperature process so there is minimum energy consumption and less pollution. It is therefore not surprising that in the nuclear fuel synthesis it is a desire process. Although sol-gel process generates highly pure, well controlled ceramics, it competes with the process like CVD. The choice, of course, depends the upon the product off interest, its size, instrumentation available ease of processing etc. In some cases, sol-gel can be an economical route, provided precursors are not very expensive. Some of the benefits like getting unique materials such as aerogels, zeolites, ordered porous solids by organic-inorganic hybridization are unique to sol-gel process. It is it is also possible to synthesize nanoparticles, nanorods, nanotubes etc. using sol-gel technique. Sols are solid particles in a liquid. They are thus a subclass of colloids. Gels are nothing but a continuous network of particles with pores filled with liquid (or polymers containing liquid).

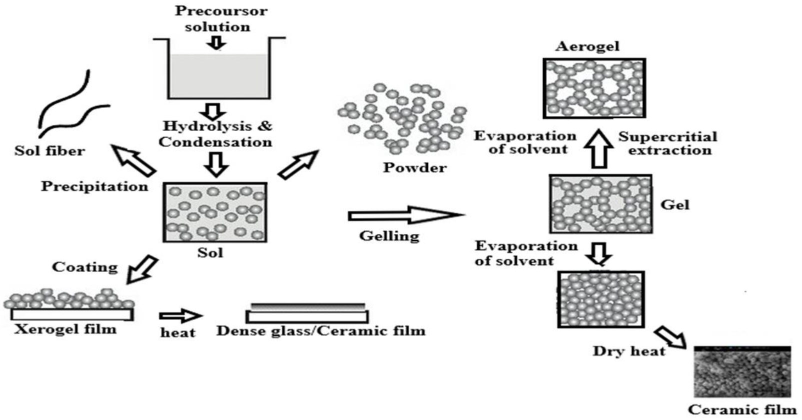


Fig.12. Different sol-gel process steps [29]

A sol-gel process involves formation of ‘sols’ in a liquid and then connecting the sol particles (or subunits capable of forming a porous network) to form a network. By drying the liquid, it is possible to obtain powders, thin films or even monolithic solid. Sol-gel method is particularly useful to synthesize ceramics or metal oxides although sulfides, borides and nitrides are also possible.

Synthesis of sol-gel in general involves hydrolysis of precursors, condensation followed by polycondensation to form particles, gelation a drying process by various routes as shown in figure 12. Precursors are to be chosen so that they have a tendency to form gels. Both alkoxides or metal salts can be used. Alkoxides have a general formula $M(ROH)_n$, where M is a cation, R is an alcoholic group and Italian n is the number of (ROH) groups with each cation. For example (ROH) can be methanol (CH_3OH), ethanol (C_2H_5OH), propanol (C_3H_7OH) etc. bonded to a cation like Al, Si etc. Salts are denoted as MX, in which M is a cation an X is an anion; Like in $CdCl_2$, Cd is a cation and Cl^- is an anion. Although it is not mandatory that only oxides be formed by a sol-gel process, often oxides ceramics are best synthesized by a sol-gel route. For example, in silica, SiO_4 group with Si at the center and four oxygen atoms at the apexes of tetrahedron are very ideal for forming sols with interconnectivity through the corners of tetrahedrons, creating some cavities or pores. By polycondensation process (i.e., many hydrolyzed units coming together by removal of some atoms from small molecules like OH) sols are nucleated and ultimately solgel is formed. The sol-gel technique is broadly used for the synthesis of oxide materials [30]. Sol-gel process is one of the famous wet-chemical methods. It works under lower-temperature processing and gives better homogeneity for multicomponent materials. The word “‘sol”’ means the formation of a colloidal suspension and ‘gel’ means the conversion of ‘sol’ to viscous gels or solid materials. Two routes are used to prepare transition metal oxides (TMOs) as follows:

a) Preparing of inorganic precursors via inorganic salts in aqueous solution.
b) preparing of metal alkoxide precursors via metal alkoxides in nonaqueous solvents.

In this section, we are concerned on the famous route “the metal alkoxide precursor solution by an alcoholic solution.

Alkoxide precursors in organic solvents

The sol-gel technique is based on the polycondensation of metal alkoxides M (OR)z in which R represents an alkyl group ($R = CH_3$, C_2H_5, …) and z is the oxidation state of the metal atom M^{z+} [31-32]. It can be synthesized via the reaction of metal salt (chloride, acetate, nitrate, etc.) with alcohol as follows:

$$(CH_3COO)_z M + zROH \rightarrow M(OR)_z + zCH_3COOH \tag{1}$$

After this process, two important steps should be involved:

- *Hydrolysis:* this step is aimed to form reactive M-OH groups-

$$M{-}OR + H_2O \rightarrow M - OH + ROH \tag{2}$$

- *Condensation:* condensation is the second step after hydrolysis leading to the departure of a water molecule. The process of condensation can be either olation process or oxolation process.
- *Olation:* a hydroxyl bridge ("ol" bridge) is formed between two metal centres as shown in Figure.
- *Oxolation:* oxolation is a reaction in which an oxo bridge (— O —) is created between two metal centres. When the metal is co-ordinately unsaturated, oxolation with rapid kinetics leads to edge or face shared polyhedral.

2) Dip Coating

Dip coating is a simple and effective technique which is commonly used in manufacturing across a wide range of different industries. In research and development, it has become an important coating method for the fabrication of thin films using a purpose-built dip coater. When the process is optimised, dip coating can be used to produce highly uniform films. Importantly, key factors such as film thickness can be easily controlled. Dip coating is a relatively straight-forward technique. However, in order to achieve maximum control when coating a substrate, it is important to be aware of what can affect your results. To make high-quality films, parameters such as withdrawal speed must be optimised. Atmospheric factors including temperature, airflow, and cleanliness also play a big part in film quality and must be closely monitored during the dip coating process. As with other methods, defects can occur, but by understanding the underlying causes it is relatively easy to find the root of the problem and take appropriate action. Dip coating involves the deposition of a liquid film via the precise and controlled withdrawal of a substrate from a solution using a dip coater. The dip coating process involves a minimum of four unique steps (or stages) followed an optional fifth curing step:

- Immersion
- Dwelling
- Withdrawal
- Drying
- Curing (Optional)

All these stages are essential in the dip coating process. However, the two critical points for determining the properties of the deposited film are the withdrawal and drying stages. Dip coating is one of the most effective processes for the production of thin films. The solution substrate is immersed in the solution for effective formation of the material. Once the material is deposited then the substrate can be removed by evaporation which will result the thickness of the layer are unique. The major forces that are used for the dip coating process are force of inertia, viscous drag, gravitational force, and surface tension . The merits of dip coating are low cost and layer thickness can be easily adjusted. The drawbacks of dip coating are process is slow and it has the ability to block the screen, which will create major impact in the final product [33].

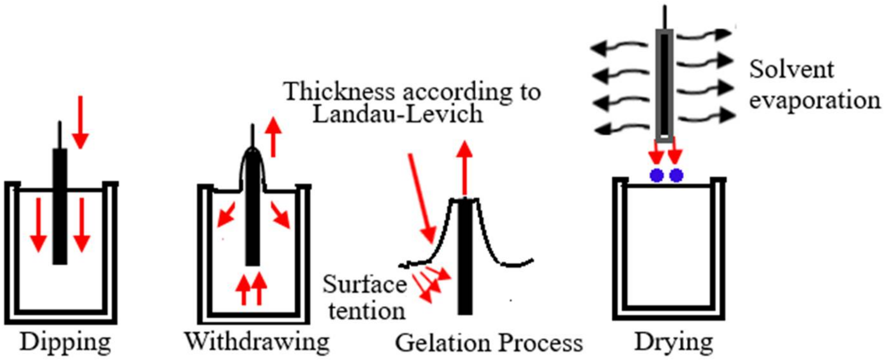


Fig. 13. A schematic view of the dip-coating method

3) *Spin Coating:* Spin coating is a procedure used to apply uniform thin films to flat substrates. A typical process involves depositing a small puddle of a fluid resin onto the centre of a substrate and then spinning the substrate at high speed (typically around 3000 rpm) [34]. (Mitzi et al. 2004). Spin coating is a common technique for applying thin films to substrates. When a solution of a material and a solvent is spun at high speeds, the centripetal force and the surface tension of the liquid together create an even covering. After any remaining solvent has evaporated, spin coating results in a thin film ranging from a few nanometres to a few microns in thickness. Spin coating is used in a wide variety of industries and technology sectors. Its primary advantage of spin coating over other methods is its ability to quickly and easily produce very uniform films. Spin coating generally involves the application of a thin film (a few nm to a few um) evenly across the surface of a substrate by coating a solution of the desired material in a solvent (an "ink") while it is rotating. Put simply, a liquid solution is deposited onto a spinning substrate in order to produce a thin film of solid material, such as a polymer. Spin coating generally involves the application of a thin film (a few nm to a few um) evenly across the surface of a substrate by coating (casting) a solution of the desired material in a solvent (an "ink") while it is rotating. Put simply, a liquid solution is deposited onto a spinning substrate in order to produce a thin film of solid material, such as a polymer. The rotation of the substrate at high speed (usually >10 rotations per second = 600 rpm) means that the centripetal force combined with the surface tension of the solution pulls the liquid coating into an even covering. During this time the solvent then evaporates to leave the desired material on the substrate in an even covering.

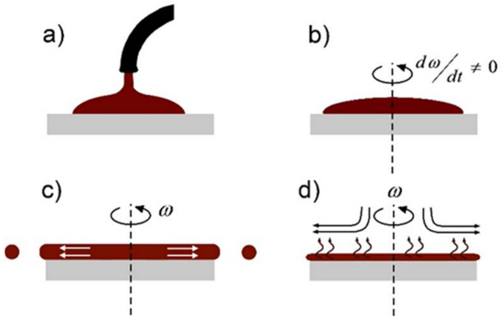


Fig.14. Example of spin coating a small molecule in solution using a static dispense. (a) First the substrate is coated in the ink containing the molecules dissolved in a solvent. (b) Then the substrate is rotated at high speed and the majority of the ink is flung off the side. (c) Airflow then dries the majority of the solvent, leaving a plasticised film. (d) before the film fully dries to just leave the molecules on the surface.

This process can be broadly divided into 4 main steps:

a) Deposition
b) Spin up.
c) Spin off
d) Evaporation

In the initial step, the solution is cast onto the substrate, typically using a pipette. Whether the substrate is already spinning (dynamic spin coating) or is spun after deposition (static spin coating), the centrifugal motion will spread the solution across the substrate. The substrate then reaches the desired rotation speed, either immediately or following a lower-speed spreading step. At this stage, most of the solution is expelled from the substrate. Initially, the fluid may be spinning at a different rate than the substrate, but eventually the rotation speeds will match up when drag balances rotational accelerations leading to the fluid becoming level. The fluid now begins to thin, as it is dominated by viscous forces. As the fluid is flung off, often the film will change colour due to interference effects. When the colour stops changing, this will indicate that the film is mostly dry. Edge effects are sometimes seen because the fluid must form droplets at the edge to be thrown off. Finally, fluid outflow stops and thinning is dominated by evaporation of the solvent. The rate of solvent evaporation will depend the solvent volatility, vapour pressure, and ambient conditions. Non-uniformities in evaporation rate, such at the edge of a substrate, will cause corresponding non-uniformities in the film [35-38].

4) Chemical Bath Deposition (CBD)

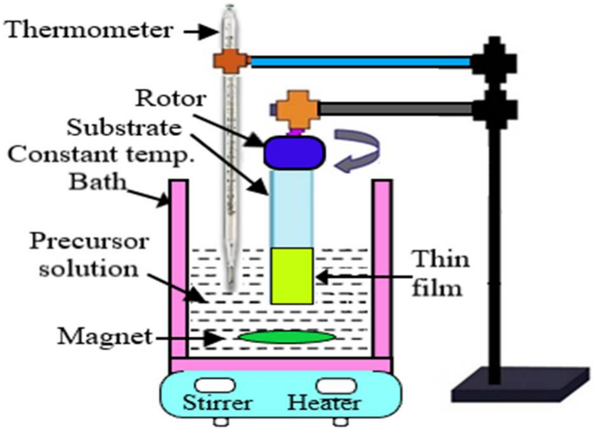


Fig.15. Schematic diagram of CBD

Chemical bath deposition (CBD) has been used as synthesis method for over 140 years.

Films can be grown on either metallic or non-metallic substrates by dipping them in appropriate solutions of metal salts without the application of any electric field. Deposition may occur by homogeneous chemical reactions usually reduction of metal ions in solution by a reducing agent. If this occurs on a catalytic surface, it is called an electro less deposition. The chemical methods are economical and easier than that of the physical methods. But there is no ideal method to prepare thin films, which will satisfy all possible requirements. Among the chemical methods, the chemical bath deposition (CBD) method is the most popular today because large number of conducting and semiconducting thin films can be prepared by this technique. It is also popular due to its simplicity and low cost. In this technique, the thin films can be deposited on different substrates like glass, ceramic, metallic etc. Many studies have been conducted over about three decades on chemical bath deposition (CBD) method for the preparation of thin films. Thereafter, due to good productivity of this technique on a large scale and simplicity of the apparatus, it offered most attractive way for the formation of thin films of metal oxides, metallic spinal type oxides, binary chalcogenides, ternary chalcogenides, superconducting oxides etc. It is simple and low-cost technique and has capability to produce large area of high-quality adherent films of uniform thickness [39-46].

The basic working principle behind the CBD process is similar to those for all precipitation reactions and it is based on relative solubility of the product. At a given temperature when the ionic product (IP) of reactants exceeds the solubility product (KSP), precipitation occurs. Whereas if the ionic product is less than the solubility product, then the solid phase produced will dissolve back to the solution resulting in no net precipitation [47].

A central concept necessary to understanding the mechanisms of CBD is that of the solubility product (Ksp). The solubility product gives the solubility of a sparingly soluble ionic salt (this includes salts normally termed "insoluble"). Sparingly soluble salt, $A(S)$, when placed in water, a saturated solution containing A^+ and B^- ions in contact with undissolved solid AB is obtained and equilibrium is established between the solid phase and ions in the solution as:

$$AB(S) \rightleftarrows A^+ + B^-$$

Applying the law of mass action,

$$K = \frac{[A^+][B^-]}{[AB]}$$

where K is stability constant, $[A^+]$, $[B^-]$and $[AB]$ are concentrations of A^+, B^- and AB in the solution, respectively.
The concentration of pure solid is a constant number, i. e. $C_{AB}\ (S) = constant = K^*$

$$K = \frac{[A^+][B^-]}{K^*}$$

$$KK^* = [A^+]\,[B^-]$$

Since K and K* are constants, the product of KK^* is also a constant, say K_{SP}, [48]. Therefore, above equation becomes

$$Ksp = [A^+]\,[B^-]$$

The constant, K_{SP}, is called solubility product (SP) and $[A^+]$ $[B^-]$ is called the ionic product (IP). When the solution is saturated, the ionic product is equal to the solubility product. But when the ionic product exceeds the solubility product ($IP/SP = S > 1$), the solution is supersaturated (where S is degree of supersaturation), precipitation occurs and ions combine on the substrate and in the solution to form nuclei.

5) *Spray Pyrolysis Technique:* The spray pyrolysis technique has lately attracted increasing attention due to some appealing features such as their low production cost, flexibility in the substrate choice, possibility of large area deployment.

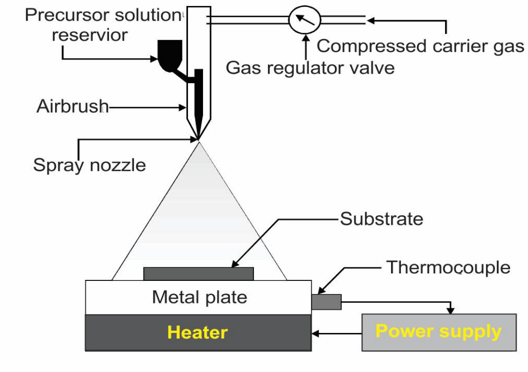


Fig.16. Schematic diagram of spray pyrolysis system

Numerous materials have been prepared in the form of thin film because of their potential technical value and scientific curiosity in their properties. A number of techniques have been examined in the search for the most reliable and cheapest method of producing thin films. Spray pyrolysis (SP) technique was initially suggested by Chamberlin and Skarman [49] in 1966 to prepare CdS thin films on glass substrates. Spray pyrolysis involves spraying of an aqueous solution containing soluble salts of the constituent atoms of the desired compounds to the heated substrates. The liquid droplets vaporize before reaching the substrate or react on it after splashing. Doped and mixed films can be prepared very easily, simply by adding to the spray solution a soluble salt of the desired dopants or impurity.

a) *Aerosol Transport*

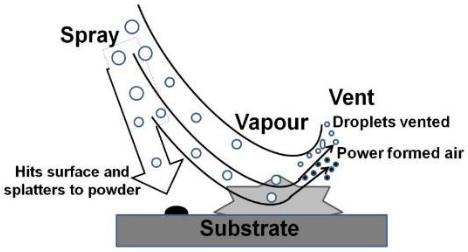


Fig.17.Schematics of aerosol transports

In the SP method, the metal salt precursor solution is atomized through a spray nozzle. Using pressurize air nozzle forms small droplets from the solution known as aerosols. These aerosols made fall on to the preheated substrates. The thermal decomposition takes place of the aerosols according to substrate temperature. The desired formation of film with specific properties can be done at optimum substrate temperature. It was observed that the film growth take place from the vapors droplets which are passing very close to the preheated substrate chemical vapor deposition as shown in Fig. 16. The solution droplets which fall on the substrate form a powdery deposit. This is suggested that instead of forcing solution droplets closer to the substrate but avoiding direct contact would improve the efficiency of film growth.

b) Advantages of spray Pyrolysis Technique compared to other methods

Spray pyrolysis technique has a number of advantages as depicted in the following points-

- It offers an extremely easy way to dope films with virtually any element in any proportion by merely adding it in some form to the spray solution.
- Unlike closed vapor deposition method, SP does not require high quality targets and/or substrates, and it does not require vacuum at any stage, which is a great advantage if the technique is to be scaled up for industrial applications.
- The deposition rate and the thickness of the film can be easily controlled over a wide range by changing the spray parameters, thus eliminating the major drawbacks of chemical methods such as sol-gel method which produces films of limited thickness.
- Operating at moderate temperatures (100 – 500ºC), SP method can produce films on less robust material.
- Unlike high – power methods such as radio frequency magnetron sputtering (RFMS), it does not cause local over – heating that can be detrimental (harmful) for materials to be deposited.
- By changing composition of the spray solution during the spray process, it can be used to make layered film and films having composition gradients throughout the thickness.
- It is believed that reliable fundamental kinetic data are more likely to be obtained on particularly well characterized film surface, provided the film are quite compact, uniform and that no side effects from the substrate occur. SP offers such an opportunity.
- Low cost comparing with other methods which require complex devices and instruments with high cost.

6) Successive-ionic Layer Adsorption and Reaction (SILAR): The SILAR method is another promising technique which was first employed by Nicolau in 1984 to deposit CdS and ZnS thin films on different substrate at room temperature. This process involves the alternate immersion of substrate into separate solution of anion and cation of the material to be deposited. Further, rinsing with high purity distilled water is carried out after every successive immersion [50]. The several advantages of this method are as

a) it is a simple and versatile method.
b) This method provides direct growth and simultaneous deposition of doped and multi-layered compounds.
c) It is reproducible and relatively short duration process.
d) It avoids wastage of material after reaction as there is no formation of precipitate.
e) It can deposit film on less robust materials [51-53].

VI. CONCLUSION

Over the last 200 years, there is increase in the processes of depositing thin films materials have been considerably developing. Here we discussed some of them. In order to optimize the desired film thickness and characteristics, good understanding of the various deposition methods and processes is necessary. In this paper, several important aspects and techniques about thin film deposition process have been presented such as a brief overview of PVD and CVD processes. The brief overview of gas phase and liquid phase processes like Spin and Dip coating, CBD, SILLAR, Sol-Gel that are significant technique of CVD. The advantages and disadvantages of these techniques are discussed and fount Spray Pyrolysis is feasible for us consequently, in our study, the process of spray pyrolysis (SP) was proposed which is based on the principle of CVD. Unlike closed vapor deposition method, SP does not require high quality targets and/or substrates, and it does not require vacuum at any stage, which is a great advantage if the technique is to be scaled up for industrial applications. It is low cost comparing with other and it doesn’t require complex devices and instruments which are high cost.

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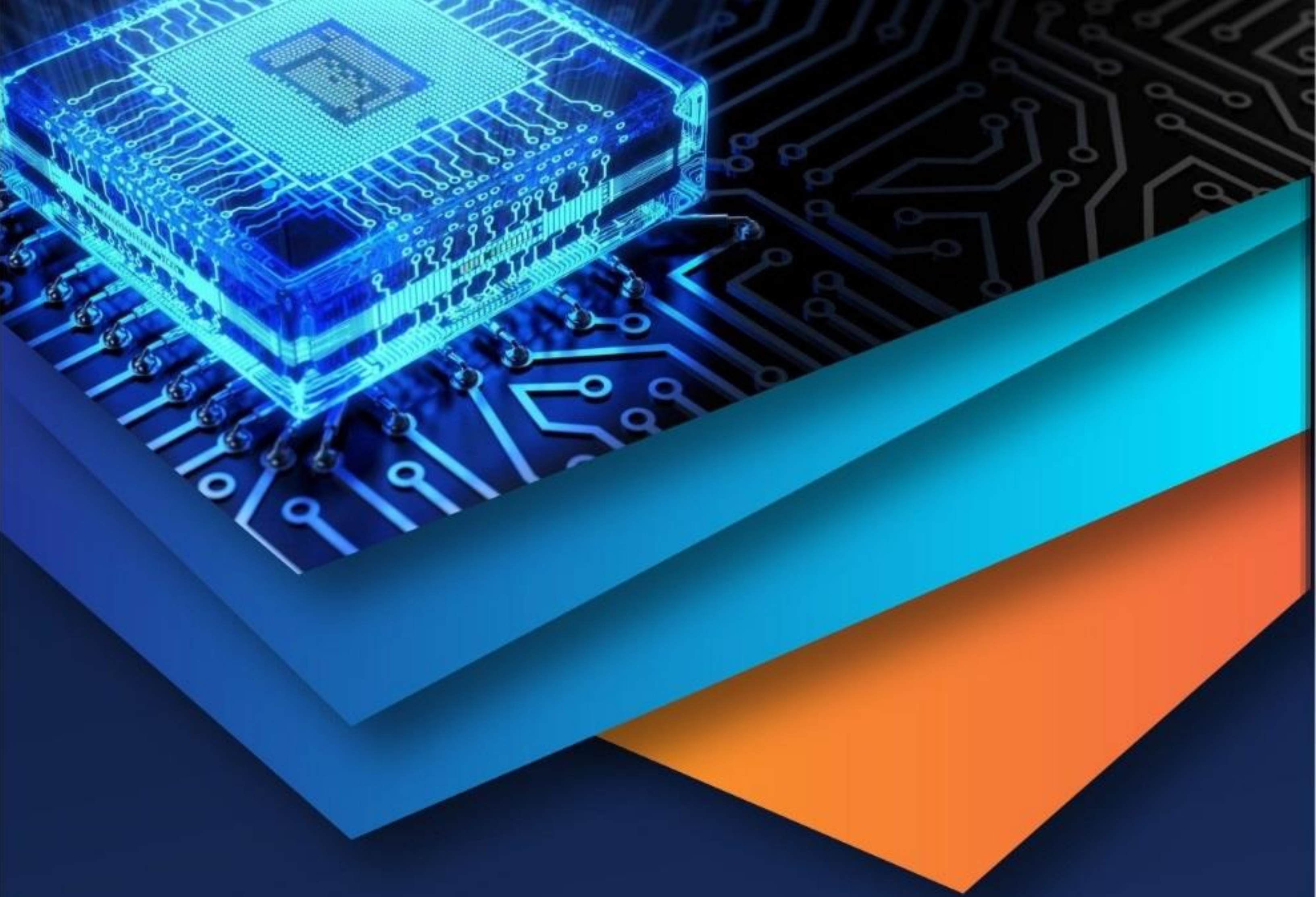

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