

## A STUDY ON THIN FILMS DEPOSITION TECHNIQUE

Poonam

Research Scholar, Calorx Teachers' University  
Ahmedabad (Gujarat)

### ABSTRACT

Physical vapor deposition (PVD) is a process of transferring growth species (material) from a source or target and depositing them on a substrate to form a film. In this process no chemical reaction takes place. This process controllably transfers atoms from a source to a substrate where film formation and growth proceed atomistically.

**KEYWORDS:** Generation of vapor, Tungsten wire sources, Sublimation Furnace, Layer by layer deposition by this technique, Physical processes

### INTRODUCTION

These are several methods for the deposition. The different deposition techniques can be in the following ways.

1. Physical processes
2. Chemical processes
3. Physical-chemical process (Glow-Discharge process)

### PHYSICAL METHODS

**Thermal evaporation (Physical vapour Deposition):** Physical vapor deposition (PVD) is a process of transferring growth species (material) from a source or target and depositing them on a substrate to form a film. In this process no chemical reaction takes place. This process controllably transfers atoms from a source to a substrate where film formation and growth proceed atomistically.

In evaporation, atoms are removed from the source by thermal means. This process consists of several physical stages.

- (i) Generation of vapor (evaporation of material)
- (ii) Transfer of vapor (atoms) from the source to substrate.
- (iii) Deposition of vapor particles on the substrate.
- (iv) Condensation of vapor into a solid film on the surface of substrate.

The evaporation of materials may be achieved by a variety of physical methods. Different heating methods are given below.

**(a) Resistive – Heating Evaporation:** Resistively-heated evaporation sources are available in a wide variety of forms. These sources can be divided into following types.

- (i) Tungsten wire sources
- (ii) Refractory metal sheet sources
- (iii) Sublimation Furnace
- (iv) Crucible sources

Generally tungsten (w), molybdenum (Mo), tantalum (Ta) is used as resistive heated wires and metal foil because they have high melting point and low vapor pressure. Evaporation occurs at high power input.

This method suffered from the disadvantages of possible contamination.

**(b) Electron Beam Evaporation:** This technique is now the most widely used vacuum evaporation technique for preparing highly pure films. This technique is free from the disadvantage of possible contamination. In this technique an electron beam of sufficiently high intensity (4 to 20 K V) is ejected from a cathode accelerated and focused onto an evaporate material.

This method provides us very high temperature to evaporate the materials. The additional advantage of this method is the presentation of impurity by the evaporant source material. The beam heats only the evaporant whereas support holder is usually cooled.

**(c) Leaser Evaporation:** The enormous intensity of coherent laser beam may be used to heat and vaporize the material by keeping the laser source outside the vacuum system and focusing the beam onto the surface of the material to be evaporated.

**(d) Flash Evaporation:** Flash evaporation technique is used to deposit multi component alloy or compound which are incompatible in bulk form at high temperature.

In this process, material is taken in powdered form. This powder is continuously dropped onto the evaporator. The some advantage of this method is given below.

1. For multi component alloys film.
2. Layer by layer deposition by this technique.
3. No change e.g. dissociation.

**(e) Arc Evaporation:** By striking an arc between two electrodes of a conducting material, sufficiently high temperature can be generated to evaporate refractory materials such as Nb & Ta. Thus electrode tip can be melts to form a film. This method has following advantages.

**(f) The physics and chemistry of Evaporation:** The evaporation of a material requires that it should be heated to a sufficiently high temperature to procedure the require vapour pressure. It is found that the rate of evaporation & difference between the equilibrium pressure ( $P_e$ ) & hydrostatic pressure ( $P_h$ ) acting on the evaporant.

Thus the rate of evaporation ( $\phi_e$ ) is given by kinetic theory equation from a solid surface.

$$\phi_e = \alpha_e N_A (P_e - P_h) / \sqrt{2\pi M R^* T}$$

This expresses the relation between vapour pressure ( $P_e$ ) and temperature (T). Where

$\phi_e$  = Evaporation rate i.e. the evaporation flux in number of atoms (or molecules) per unit area per unit time.

$\alpha_e$  = coefficient of evaporation ( $0 \leq \alpha_e \leq 1$ )

$N_A$  = Avogadro's number

$P_e$  = Equilibrium vapour pressure.

$P_h$  = Hydrostatic pressure acting on evaporant.

$R_g$  = Gas constant.

$M$  = Molecular weight of vapour species.

The maximum evaporation is achieved when  $\alpha_e = 1$ ,  $P_h = 0$

$$\Phi_e (\text{max}) = 3.513 \times 10^{22} P_e / \sqrt{MT} \text{ molecules / Cm}^2\text{-sec}$$

When  $P_e$  is expressed in torr, then

$$v_e = 5.834 \times 10^{-2} \sqrt{M} / T P_e \text{ gm / Cm}^2\text{-sec}$$

Where  $v_e$  = mass evaporation rate.

The equilibrium vapour pressure of elements – It is given by

$$\ln P_e = -\Delta H_e / RgT + C$$

Where

C is the constant of integration.

$\Delta H_e$  = Molar heat of evaporation.

The rate of condensation/deposition of the vapour atoms depends on the vapour source-substrate geometry and the condensation coefficient. Evaporation of compounds involves the chemical reactions.

**(g) Ideal Conditions for Deposition:** The rate of deposition depends on the vapor source substrate geometry and condensation coefficient. Low pressure ( $10^{-10}$  to  $10^{-3}$  torr) is required for good deposition. Under low pressure condition i.e. in vacuum we have following advantages. ~~The material will boil at a lower temperature in vacuum, i.e. melting point will decrease.

1. There will be a reduction in oxides or other impurities in the deposited material.
2. Since  $\gamma \text{ mfp} \propto \lambda / p$  i.e. as the gas pressure decrease the mean free path increases. Therefore there is less no. of atoms increases. Due to this there will be less number of collisions and the atom and ions will have more energy (loss of energy is less). Finally the atom and ion will strike at the surface with more kinetic energy (or velocity) so that the adhesion will be better.

**(h) Design of a High Vacuum System:** vacuum evaporation requires high vacuum conditions. These conditions can be achieved by a suitable condition of vacuum pumps. We can classify the vacuum system as following.

Rough vacuum ~ 0.1 to 760 torr

Medium vacuum ~ 0.1 to  $10^{-4}$  torr

High vacuum ~  $10^{-4}$  to  $10^{-8}$  torr

Ultra High vacuum ~  $\alpha 10^{-8}$  torr

Thin film formation requires high vacuum chamber. Thus vacuum system comprises a backing pump also.

**Following are the suitable combinations to produce high vacuum.**

|   | Pump type            | Pumping capacity | Preferred baking pump        |
|---|----------------------|------------------|------------------------------|
| 1 | Diffusing pump       | unlimited        | Rotary oil<br>Hook and claws |
| 2 | Turbo molecular pump | unlimited        | Rotary oil<br>Hook and claws |
| 3 | Getter- ion pump     | limited          | Sorption                     |
| 4 | Getter pump          | limited          | Sorption                     |
| 5 | cryopump             | limited          | any                          |

A diffusion pump backed by a rotary pump continue to be  $10^{-6}$  to  $10^{-8}$  torr work horse in thin film technology largely because of its price, simplicity, and high speed. By using special diffusion pump oil (e.g. poly phenyl ether), a cryogenic baffle and Al - system Ultra -High vacuum (UHV) in the range  $10^{-8}$  to  $10^{-10}$  torr are easily obtained.

**(i) Baffles, Cold traps & Sorption traps:** An important element of any vacuum system is the baffle or cold trap. It is important to be clear about the uses of these two pieces of equipment. A trap (cold trap) is actually an entrapment pump fluid vapors and returns them to the pump. It is therefore generally associated with diffusion pumps, which are the type of pump most able to produce oil vapor and oil vapor decomposition products. An even more effective trap is provided by liquid nitrogen cooling of such a baffle. The liquid  $N_2$  has temperature about  $-196^{\circ}C$ .

Finally, sorption traps are required to be interposed between rotary oil backing pumps and any main vacuum pump.

Metal used for vacuum system must have very low vapor pressure. Most commonly used structure material is stainless steel followed by aluminium.

### MOLECULAR BEAM EPITAXY (MBE)

MBE is the one of the finely controlled method for growing single crystal epitaxial films in a high vacuum ( $10^{-11}$  torr). The most important aspect of MBE is the the slow deposition rate (less than 1000 n m per hour) which allows the film to grow epitaxially.

The film are formed on single crystal substrates by slowly evaporating the element or molecular constituents of the film from separate Knudsen effusion source cells onto substrates held at a temperature appropriate for chemical reaction, epitaxy, and re-evaporation of excess reactants. Fast shutters are interposed between the source and the substrates.

In the example of gallium and arsenic, single crystal gallium arsenide is formed. The term "beam" means that evaporated atoms do not interact with each other or vacuum chamber gases until they reach the wafer, due to the long mean free paths of the atoms fig.

### ADVANTAGES

Complex layers or super lattices, low temperature requirement epitaxy (Si:  $400-800^{\circ}C$ , :  $500-600^{\circ}C$ )

### DISADVANTAGES

Limited product through put, complex operation, expensive equipment

### VACUUM PUMPS

A large number of clever designs for vacuum pumps have been implemented over the years, dating back to the first leather- and -grease sealed, hand-operated pumps of the 1600's. These first pumps were modified ships' of the day, and they operated by a simple valve-and-piston mechanism.

The valve-and-piston principle is still the most widely used way of extracting air in the viscous-flow regime, though today our implementation air in the viscous-flow regime, though today our implementation is considerable more efficient! Modern mechanical pumps feature multiple stage, specialized low-vapor-pressure oil sealants, and electric motors. Good, modern mechanical pumps can often attain base pressures of a few millitorr of a few tens of millitorr, though below about 100 mtorr the oil used in them will often leak back into the chamber being pumped on. This is called

back streaming and is usually undesirable. Back streaming can be eliminated by placing a trap or high-vacuum pump between the mechanical pump and the chamber.

Mechanical pumps are seldom operated below 100 mtorr, and for this reason they are often referred to as roughing pumps. To achieve even a moderate vacuum of  $10^{-2}$  torr or better, a different pump design must be employed. The most common and reliable high-vacuum pumps in use today are turbo molecular pumps, or turbo pumps for short.

Cross section of a single-stage, rotary-vane mechanical roughing pump. Gas is pulled in the inlet (arrow pointing down), circulated counter clockwise and compressed, then below out through a ball valve on the outlet. The theoretical ultimate base pressure is the pressure at the outlet (approximately atmospheric) divided by the compression ratio.

Turbo pumps are capable of sustaining vary high compression ratios, the ratio of the gas pressure at the output to that at the input. Typical compression ratios are on the order of  $10^7$  for air, for an outlet pressure of 100mtrorr. This low outlet pressure is maintained by a mechanical pump, which acts as both a roughing pump for the system and a backing pump for the turbo. One advantage of using a turbo pump in conjunction with a mechanical pump is that the turbo pump's compression ratio depends strongly on the molecular weight of the gas being pumped. Specifically, the log of the compression ratio is proportional to the square root of the molecular weight of the gas. Because the oils used in mechanical pumps typically have very high molecular weights, the compression ratio across the turbo pump for these oils is considerable higher than  $10^7$ , and the turbo pump effectively blocks any back streaming from the roughing pump.

Speeds for turbo pumps are usually independent of the type of gas being pumped. Turbo pumps are specified by their speed, and the small turbo pump used in this lab has a speed of 80 1/s.

## **DIFFUSION PUMP**

Diffusion pumps are designed to operate in the molecular flow regime and can function over pressures ranging from well below 10- 10torr to about 1torr. Because they cannot discharge directly into the atmosphere, a mechanical forepump is required to maintain an outlet pressure of about 0.1 torr. A pumping speed of 11.7 AL/sec would be theoretically expected for air at room temperature. Actual pumping speeds are typically only 0.4 of this value. Diffusion pumps have been constructed with pumping speeds ranging from a few liters per second to over 20000 L/sec. pumping is achieved through the action of a fluid medium (typically silicone oil) that is boiled and vaporized in a multistage jet assembly. As the oil vapour stream emerges from the nozzles, it collides with and imparts momentum to residual gas molecules, which happen to bound into the pump throat. These molecules are thus driven toward the bottom of the pump and compressed at the exit side where they are exhausted. A region of reduced gas pressure in the vicinity of the jet is produced, and more molecules from the high-vacuum side move into this zone, where the process is repeated. Several jets working in series serve to enhance the pumping action. A serious problem associated with diffusion pumps is the back streaming of oil into the chamber. Such condensed oil can contaminate both substrate and deposit surfaces, leading to poor adhesion and degraded film properties. Oil vapour dissociated on contact with hot filament or by electrical discharges also leaves carbonaceous or siliceous deposits that can cause electrical leakage or even high-voltage breakdown.

For these reason, diffusion pumps are not used in surface analytical equipment such as Auger electron and secondary ion mass spectrometers or in ultrahigh-vacuum deposition system. Nevertheless, diffusion-pumped system are widely used in non- electronic (e.g., decorative, optical, tool) coating applications. To minimize back streaming, attempts are made to condense the oil before it enters the high-vacuum chamber. Cold caps on top of the uppermost jet together with refrigerated traps and optically dense baffles are used for this purpose, but at the expenses of somewhat reduced conductance and pumping speed.

## CHAMBERS AND SEALS

Two things that limit the level of vacuum in any experiment are leaks and outgassing. (Both are mass flows and are expressed in torr liters per second.) Leaks are just poor seals that allow air to enter the chamber from the outside atmosphere. Outgassing refers to sources of gas “stored up” inside the vacuum chamber and released slowly into the vacuum. Typical sources of outgassing are trapped pockets of air in blind screw holes, rough surfaces.

### EXPLODED VIEWS OF A GASKET SEAL

Blind screw holes are often dealt with by using screws with a hole drilled through the centre, so that the screw hole communicates to the rest of the chamber and gets pumped out along with the rest of the apparatus.

Outgassing by contaminants can be eliminated by keeping the system clean. Always wear gloves when handling anything that goes inside a vacuum system, and never use ordinary lubricants on these parts.

The preferred modern method for lubricating threads is to silver plate them. Silver does not stick to stainless steel well, and a silver-plated screw will turn in a threaded, steel hole almost as easily as one that is lubricated. Look for silver-plated screws inside the vacuum chamber, as well as vented ones!

Rough surfaces outgas simply because air and especially water in the air, sticks to them, coming off at a low but regular rate when the system is under vacuum. Clean stainless steel typically outgases at a rate of 10<sup>-7</sup> torr liters per second per square centimetre of surface area. Dirty stainless steel outgases more.

The ultimate pressure of a system with leaks or outgassing is determined by the mass-flow equation  $Q=PS$ .

## PRESSURE MEASUREMENT

Just as different pumping schemes must be used in the viscous and molecular flow regimes, different methods of measuring the pressure must be used in different ranges as well. In this lab, we will use a thermocouple gauge for measuring pressure between 2 torr and 10 mtorr, and an ion gauge in molecular-flow regime. A thermocouple gauge consists of a filament and thermocouple in contact with each other. There is a range of pressures, approximately 10 mtorr to 2 torr, where the thermal conductivity of a gas depends on the pressure. If we dissipate a known amount of heat in the filament, then its temperature, as measured by the thermocouple, will depend on the rate of heat lost to the surrounding gas. Cross section of a copper gasket seal. The knife edges on either side of the flange bite into the copper gasket and form a bakeable, high-vacuum seal.

Gauge itself, the filament and thermocouple, are enclosed in a plug attached to the chamber. The controller supplies power to the filament and performs the necessary conversion of thermocouple temperature to pressure, displaying the result on its front panel. This type of gauge ceases to be useful below a few milli-torr, where the thermal conductivity of the gas is negligible. Very low pressures can be measured using an ion gauge. An ion gauge consists of a filament (cathode), a positively charged grid (anode), and a negatively charged collection wire. Electrons boil off the filament by thermionic emission and are accelerated towards the grid. On the way to the grid, they collide with atoms in the surrounding gas, producing ions. These positively-charged ions then go to the collection wire, and the resulting current in the collection-wire circuit is proportional to the gas pressure. The proportionality constant is different for different gases, because different gauges have different ionization potential.

## REFERENCES

- Husain M., Singh Beer Pal, Kumar Sushil, Sharma T.P., and Sebastian P.J., (2003), "Solar Energy Materials & Solar Cells" 76. pp. 399-415.
- Kumar Amit, Kumar Manoj and sing Beer pal, (2010), "Applied surface science", 256. pp. 7200-7203.
- Kumar Amit, Kumar Manoj and sing Beer pal, (2010), "IJAEST", 1(2). pp. 118-122.
- Klingshirm C., (1975), "phys. Status solidi", 71. pp. 547.
- Lee J.H., Park B.O., (2012), Thin Solid Films, 426. pp. 94.
- Lin S.S., and Huang J.L., (2004), Surf. Coat. Technol. 185. pp. 222.
- Look D.C., Reynolds D.C., Sizelove J.R., Jones R.L., Litton C.W., Cantwell G., and Harsch W.C., (1998), Solid State Commum. 105. pp. 39.
- Sundaram K.B., and Bhagavat .G.K., (1981), Thin solid films, 78. pp. 35.
- Tauc J (Ed.), (1974), Amorphous & Liquid Semiconductors, Plenum Press, New York. pp. 159.
- Zendehtar Akbar and Moghadan-Anghli Leila, (2010), World applied science journal, 10(12). pp. 1473-1479.