Introductory Neuroscience & Neuro-Instrumentation
Indian Institute of Science, Bengaluru
Lecture No. 51
Physical Vapour Deposition Thermal Evaporation

Hello, welcome to this module. In the previous module what we have seen, we have seen lithography technique and in that I told you that let us see how the deposition of the film happens on the substrate before which we can perform the lithography. So, what does that mean? Let us say this is a substrate on this substrate let us say, this is a substrate, let say this is a silicon substrate. So, its looks like this. Now I want to deposit a film, so its starts looking like this after deposition a film I had to pattern it.

So, some of the area the film is protected and the remaining area is etched, that patterning is called lithography and if you use photons, photon lithography. Now the question is how can I deposits on this substrate this film? So, there are techniques which are called thin film deposition technique. A film which is thin, when you say thin is a few microns, in fact less than a micron most the cases and the deposition is done under the vacuum and that is why it is also called vacuum deposition technique.

Now we will see why this called vacuum deposition and what is the role of the vacuum in depositing these films in a while? For you to understand the concept if you see the slide, let me just show you the concept so the topic is thin film deposition technique techniques.

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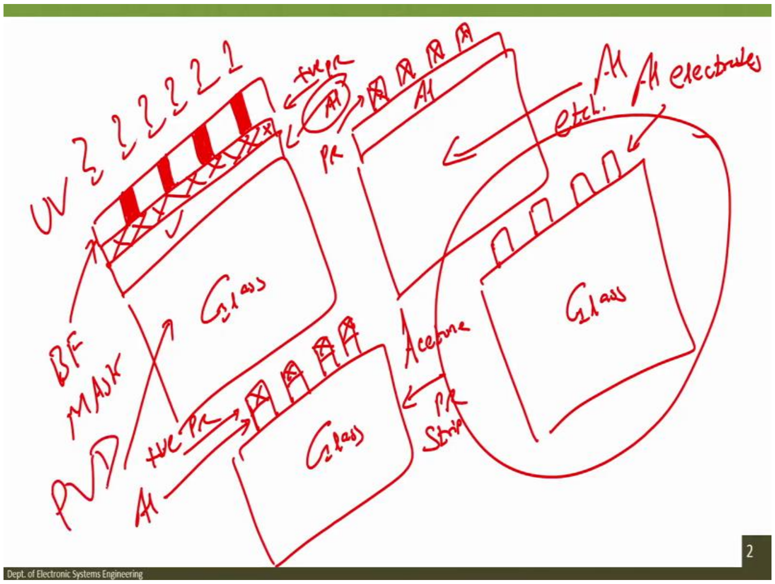

If you take a substrate and let us say that I have a glass substrate, glass, on which I want to pattern aluminum electrodes, aluminum electrodes. So, the top for, top view would look like, like this I can say like this, but the cross-sectional view will look like this. Now the point is how can I pattern aluminum electrodes on this glass; first is that I have to deposit aluminum on the glass wafer. And then I can perform lithography where you have your photoresist, let us say we draw photoresist like this, photoresist.

Then you have your mask, of course you have to perform soft bake, then mask. Mask will look like this, is a bad fill mask. And once you load the mask you have to align the mask with the wafer before the alignment step. Once you align the mask with the wafer what happens, you have, you can make a hard contact after that so this is our bright field mask. And then you can expose this wafer with UV light after that you do unload the mask and develop the wafer.

When you develop the wafer what it will look like, you have a glass, and you have a aluminum, and the photoresist would be in 4 different places because the unexposed region will be stronger since our photoresist is positive photoresist. So, this what you will get. After that, what is this one? This is our aluminum. So, after that this is my photoresist, this is aluminum. If I dip this or if I place this wafer in aluminum etchant is called aluminum etch. Then what will happen, we will have, where I did this for an aluminum etchant.

What I will have? Let me just draw it here I will say that this is the wafer that we are placing in Al etching. I will have my wafer which will look like this. What I have, I have, this is not correct. I will have my wafer like this, where your aluminum is at the bottom and a photoresist is at the top. What happens is the aluminum etchant will etch aluminum from the area which is not protected by photoresist you have aluminum, you have positive photoresist and you have the glass.

Now if I dip this wafer in acetone for being in a, see the process would be you have aluminum which is this layer, then you have photoresist you spin coat photoresist, soft bake, align, expose, develop, after developing you have to go for a hard bake, after that you etch aluminum from the area which is not protected by photoresist and then dip the wafer in acetone. Always remember after every chemical etching step you have to rinse the wafer with DI water and then dry the wafer with nitrogen gas. This is the process by default always remember any chemical when you use you have to do this step.

Now if I dip the wafer acetone what will happen? Acetone is the stripper, it is stripper for your photoresist, so acetone will get etch and you will have this particular wafer or you can say glass substrate with aluminum interdigitated electrode patterned using for photon lithography technique. The question here is how we got the aluminum layer deposited on the glass substrate?

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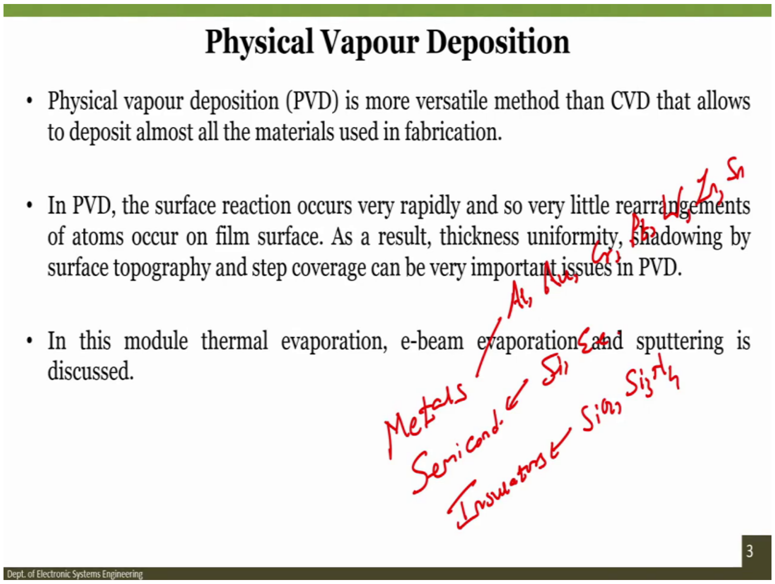

Physical Vapour Deposition

- Physical vapour deposition (PVD) is more versatile method than CVD that allows to deposit almost all the materials used in fabrication.
- In PVD, the surface reaction occurs very rapidly and so very little rearrangements of atoms occur on film surface. As a result, thickness uniformity, shadowing by surface topography and step coverage can be very important issues in PVD.
- In this module thermal evaporation, e-beam evaporation and sputtering is discussed.

And the answer is PVD which is called physical vapour deposition technique. So, physical vapour deposition technique is more versatile method than CVD. What do you think by what do you mean by CVD? CVD is Chemical Vapour Deposition technique. Now PVD is more versatile than CVD, because PVD allows to deposit all materials used in fabrication that means it can allow us to deposit metals, it can allow us to deposit semiconductors, it allows us to deposit insulators. Metals; aluminum, gold chrome, platinum, tungsten, zinc, tin, tin is not t, tin is Sn.

So, lot of metals we can, we can deposit semiconductor; silicon, germanium, we can deposit very easily; insulators, Sio2, Si3n4 silicon dioxide, silicon nitrite and, and so and so forth. So, the PVD, in PVD the surface reaction occurs very rapidly and so very little time for rearrangement of the atoms and that is why you will see that the orientation of the atoms is random in most of the cases when you take the PVD technique.

While there is an effect of this on the thickness uniformity of the metal or the material that be deposit on the substrate. What does that mean? If you see the slide that you can see that because of the higher or faster surface reaction or rapid surface reaction the thickness uniformity, shadowing by surface topography and step coverage can be very important issues in physical vapour deposition. So the uniformity, step coverage and the topography will get effected in PVD. However, there are ways to overcome these limitations and let us see in this module thermal evaporation e-beam evaporation and sputtering.

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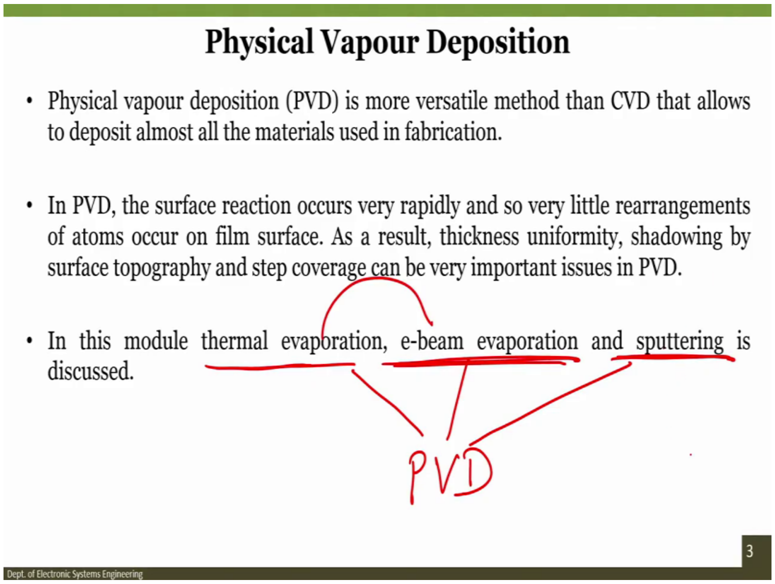

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So, when you always talk about physical vapour deposition it is generally in 3 steps or 3 different processes; 1 is called evaporation which is thermal evaporation, the another one is called the electron beam evaporation and third one is called sputtering method to deposit the material on to the substrate. The first two are electrical and the while sputtering is a mechanical way of dislodging the atoms and depositing onto a substrate, we will look into that in detail in few slides.

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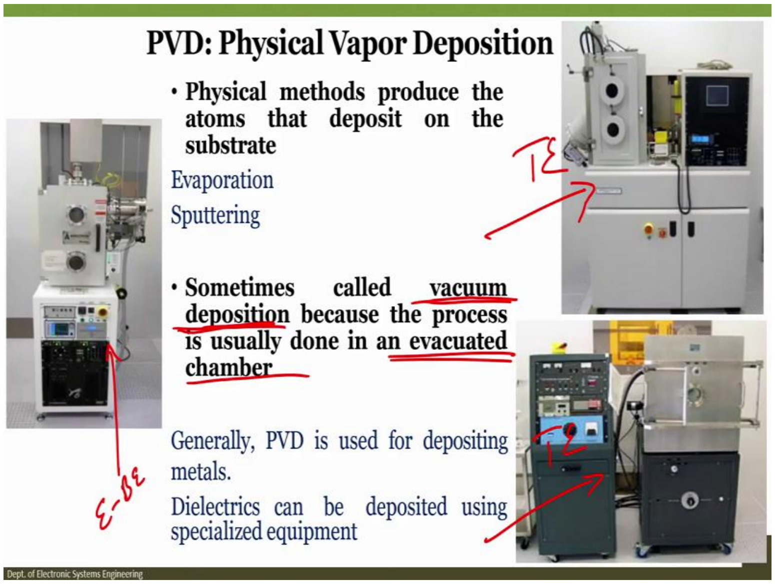

PVD: Physical Vapor Deposition

- **Physical methods produce the atoms that deposit on the substrate**

Evaporation

Sputtering

- **Sometimes called vacuum deposition because the process is usually done in an evacuated chamber**

Generally, PVD is used for depositing metals.

Dielectrics can be deposited using specialized equipment

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So, let us start with your physical vapour deposition and as I told you the physical vapour deposition mainly it is divided into two where evaporation when we further divided in thermal and e-beam. So, physical vapour methods produced the atoms that deposit on the substrate. Evaporation and sputtering is are two different techniques.

Sometimes this thermal evaporation or the physical vapour deposition is also called vacuum deposition technique because the process is usually done in the evacuated chamber that means chamber without air that means chamber with vacuum. So, generally PVD is used for depositing metals however, the dielectric material can also be deposited. Now let me show you what are the photographs in this particular slide.

The photograph on the bottom right is a thermal evaporation system, it is a manual thermal evaporation system that means we have to open the chamber, we have to load the material and then we have to create the vacuum by using the electronic server here. And then you use the heating to evaporate the materials, we will go in stepwise, do not worry about it what are their further steps, the point is there is the requirement of personnel next to the system to operate the system, manual way of thermal evaporating.

The second image that I will show you which is on that right, the top right which is this one is your semi-automated thermal evaporation system. This one is the bottom is manual thermal evaporation, the one on the top is semi-automated thermal evaporation and finally in this case this is electron beam evaporation.

We have two thermal evaporation let say Te, Te let say one is the e beam evaporation Ebe. This is not a short form, so in the exam do not write Te and Ebe this is just like I am showing it to you so that it is easy for you to understand, rather I am writing the electron beam operation, I am just writing Ebe. So, let us understand further thermal evaporation technique in the next slide.

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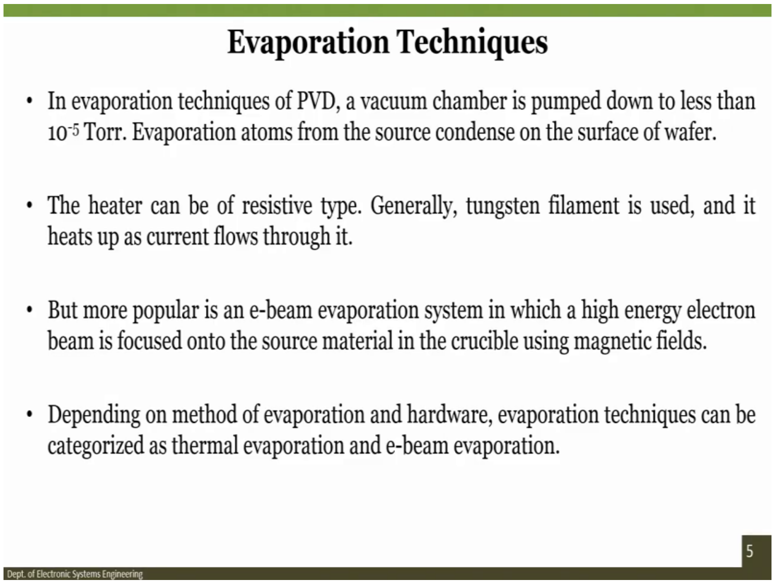

Evaporation Techniques

- In evaporation techniques of PVD, a vacuum chamber is pumped down to less than 10^{-5} Torr. Evaporation atoms from the source condense on the surface of wafer.
- The heater can be of resistive type. Generally, tungsten filament is used, and it heats up as current flows through it.
- But more popular is an e-beam evaporation system in which a high energy electron beam is focused onto the source material in the crucible using magnetic fields.
- Depending on method of evaporation and hardware, evaporation techniques can be categorized as thermal evaporation and e-beam evaporation.

So, evaporation techniques particularly when we talk about PVD vacuum chamber is pumped down to less than 10 raise to the power minus 5 Torr and evaporation of atoms from the surface or source condense on the surface of the wafer. Now what does that mean? So, let me just show it to you in one more slide which is right over here I just delete these things.

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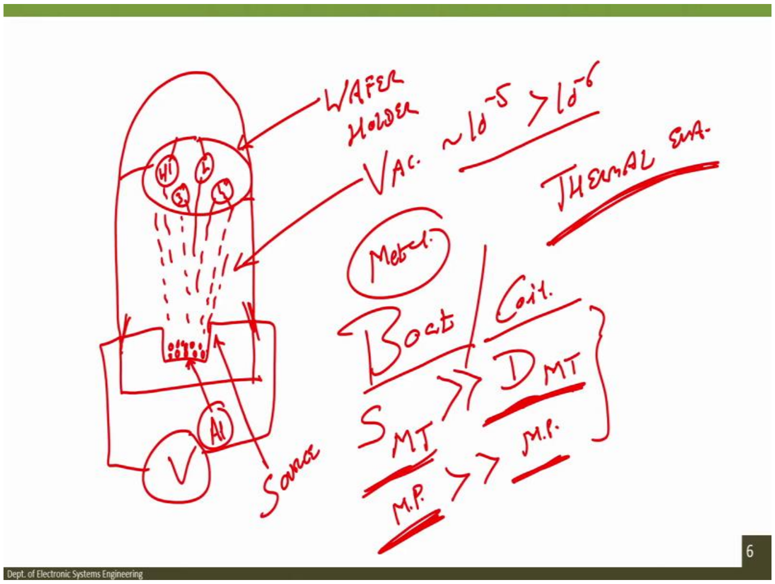

So, what does that mean that you have a chamber and then on the top there is a wafer holder, in the bottom there is source holder. What is this called source holder this is called wafer holder on which you can place wafer. This is wafer is holded by some pins or there are slots and the source holder it is also called boat; it can be boat it can be coil. Now I will explain it, do not worry about it.

So, the source is placed inside the boat, what is the source? These are aluminum pellets. If you want to have semiconductors, semiconductors pellets, if you want have insulator, insulator pellets. So the point is here we are talking about aluminum so aluminum pellets. Now, this boat is made up of metal, the chamber is evacuated that means there is a creation of a vacuum. Approximately 10 to the power minus 5, generally it is greater than 10 raise to minus 6 higher the vacuum, better your quality of the film.

Now the system is called thermal evaporation system, thermal evaporation system, why? Because you have to apply a voltage across this, it is funny no? Our chamber I can delete the chamber so powerful. So, anyway so the point is these are of course wafer 1, I say 1, 2, 3, 4 I had to apply a voltage across my boat which is a metal. What will happen voltage is there, there is a metal resistance, there is a resistance so there will be flow of current.

Now metal has extremely low resistance and that is why the current flowing through the boat would be extremely high. There will be i^2r heating which is also called joules heating. And because of that the metal which is aluminum inside the boat will start melting. So, we will say boat metal as Bm we can also say instead of boat, source metal because this is a source, is a source metal, source can boat, source can be coil. So, source metal and then we will say the source metal temperature and we will have the material that we want to deposit.

So, let us say deposition metal temperature in this case aluminum is there so we said deposition metal which is DMT and source metal is SM and temperature is T. Always remember that SMT should be greater than greater than DMT that means the melting point of SMT would be very high compared to the melting point of the deposition metal. That means the, of course the source metal should be high than the deposition metal then only deposition metal will get evaporated but also important thing is a melting point of source metal is also very high than melting point of the deposition material.

So, if I apply voltage there will be flow of current, because of flow of current the source metal will heat. When it heats the material inside which is aluminum will also get heat and heated up and it will melt, when melt, it melts, it will start evaporating and condensing on the substrate. So, we are thermally evaporating a material which is holded onto the source and that is why this technique is called thermal evaporation. Now the question is why my melting point or why the melting point of the source material should be extremely high than the deposition material?

The answer is that if your source material is equal to melting point what will happen that the source will also start evaporating. We do not want source material to evaporated, we want aluminum to get deposited, we do not want our source material to also get deposit along aluminum otherwise it become alloy is a two different materials two different metals that will be evaporating. So, we require the source material to be at extremely high temperatures compare to the deposition material.

Now, the another equation that we should we ask is what should be the vacuum in DC evaporation system, and the answer is the vacuum should be extremely high. That means minus 6 or minus 7 Torr, in fact anything which is greater than 10 raise to minus 5 we can use in the system. So, the point is what is our requirement of having a vacuum? Why we cannot deposit the material in the ambient situation in the air? So, for that there is a reason and let us see the reason in few slides to help you out.

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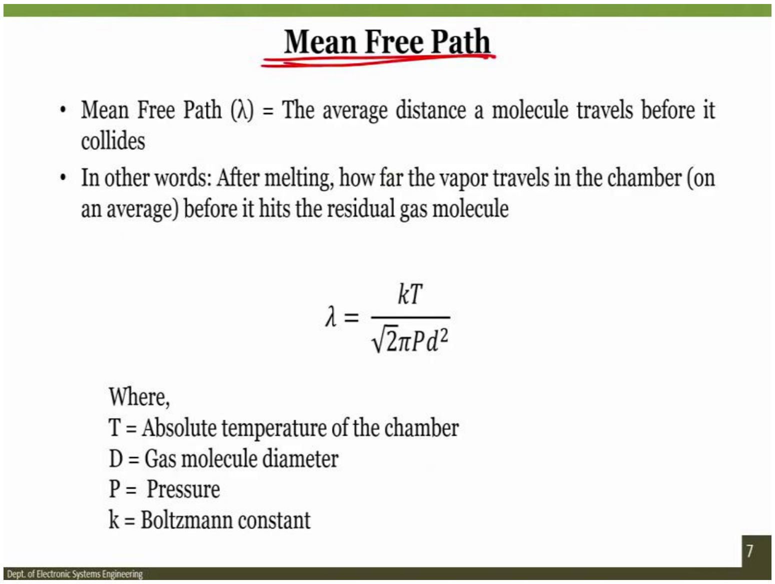
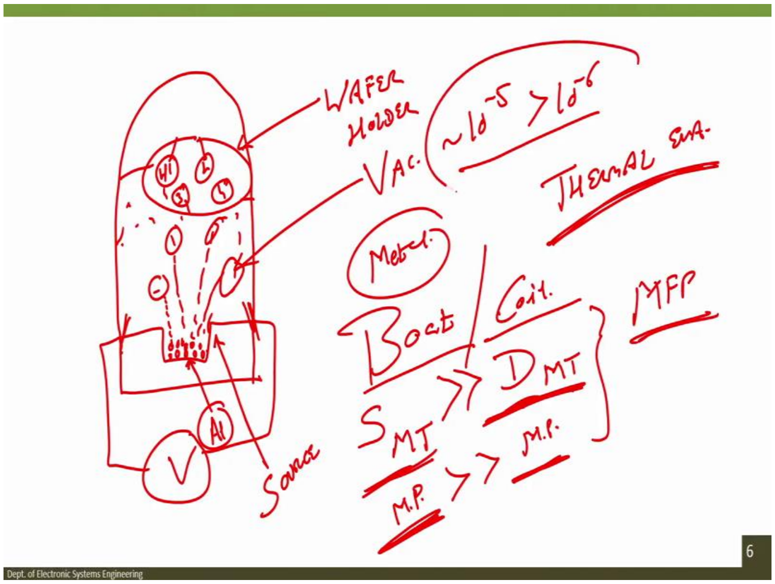


Mean Free Path

- Mean Free Path (λ) = The average distance a molecule travels before it collides
- In other words: After melting, how far the vapor travels in the chamber (on an average) before it hits the residual gas molecule

$$\lambda = \frac{kT}{\sqrt{2}\pi P d^2}$$

Where,
T = Absolute temperature of the chamber
D = Gas molecule diameter
P = Pressure
k = Boltzmann constant



The technique or the process is called mean free path or the parameter is called mean free path and what does mean free path means, mean free path means the average distance a molecule travels before it collides. That means if I have this chamber how much my, this melted material would travel to my wafer before hitting an atom. Because there would be some atoms, is there is a vacuum but still some atoms would be left

So, if I start evaporating at some point of time as you see this there is a possibility of hitting with this, hitting with this, hitting with this, hitting with this. How much length it can travel before it hits the atom is called mean free path and this mean free path, mean free path can be increased by having a higher vacuum inside the chamber. So, that is what it says, it is written here that the average distance a molecule travels before it collides with another atom and in other words after melting, how far the vapor travels in the chamber before it hits the residues gas molecule. That means a gas which are here, I said the atom but that is not a correct way to express it, there will be gas molecules inside and those gas molecules we evacuate it by using the vacuum.

However, the remaining gas molecules would be there inside the gas chamber, when the material deposits, it will hit those gas molecules, how long it can travel before hitting gas molecules is our mean free path and you can see from the equation here, it is given by lambda equals to if you see the slide, $\lambda = kT / \sqrt{2}\pi P d^2$, where T is the absolute temperature of the chamber, D is gas molecule diameter, P is pressure and k is your Boltzmann constant.

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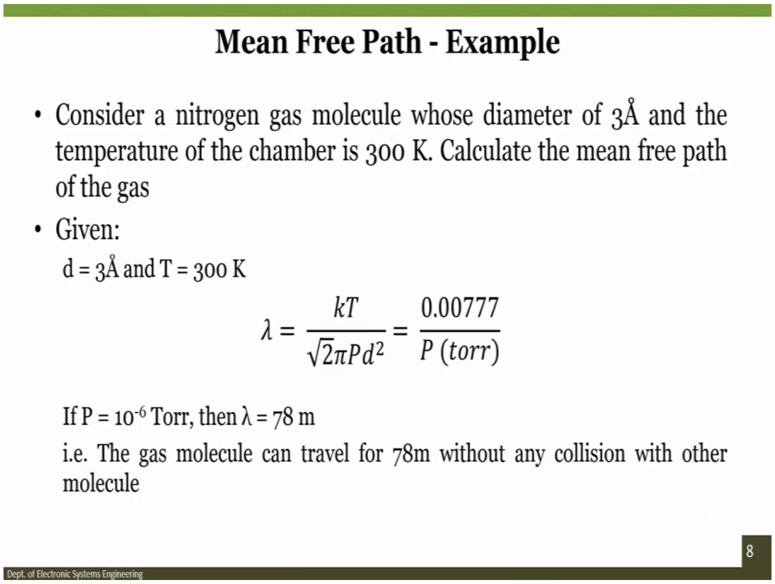

Mean Free Path - Example

- Consider a nitrogen gas molecule whose diameter of 3Å and the temperature of the chamber is 300 K. Calculate the mean free path of the gas
- Given:

d = 3Å and T = 300 K

$$\lambda = \frac{kT}{\sqrt{2}\pi P d^2} = \frac{0.00777}{P\,(torr)}$$

If P = 10^{-6} Torr, then λ = 78 m

i.e. The gas molecule can travel for 78m without any collision with other molecule

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Now let us see an example of a mean free path, so if you consider a nitrogen gas whose diameter of 3 angstrom and the temperature of the chamber is about 300 Kelvin, then if you want to calculate the mean free path, what should be the answer? The d is given by 3 angstrom and T is 300 Kelvin. Now we already had equation lambda equals to kT $\sqrt{2}\ \pi P d^2$.

So, if we put these values in the equation, we will have P = 10 to the minus 6 Torr and lambda equals to 78 meter. That means the gas molecule can travel for 78 meter without any collision with the other molecule. If the pressure is minus 7, the 78 meter will become larger, if the pressure is minus 5, it will become smaller.

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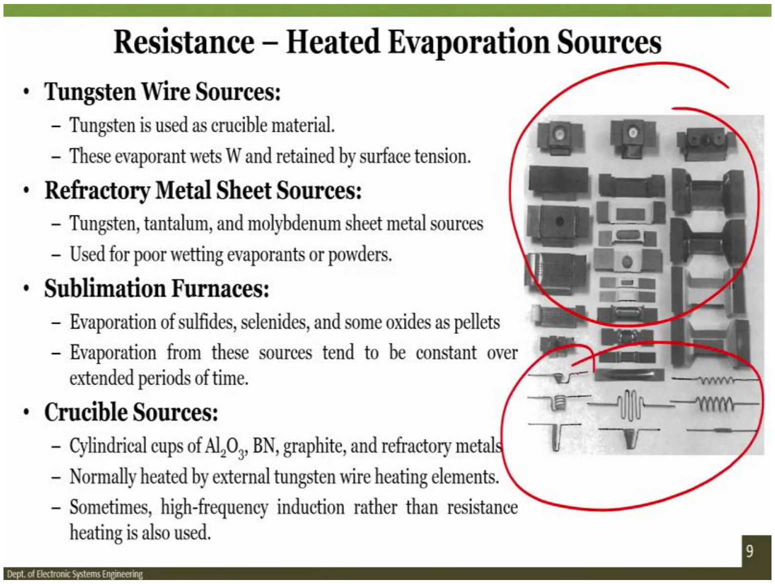

Resistance – Heated Evaporation Sources

- **Tungsten Wire Sources:**
  - Tungsten is used as crucible material.
  - These evaporant wets W and retained by surface tension.
- **Refractory Metal Sheet Sources:**
  - Tungsten, tantalum, and molybdenum sheet metal sources
  - Used for poor wetting evaporants or powders.
- **Sublimation Furnaces:**
  - Evaporation of sulfides, selenides, and some oxides as pellets
  - Evaporation from these sources tend to be constant over extended periods of time.
- **Crucible Sources:**
  - Cylindrical cups of Al_2O_3, BN, graphite, and refractory metals
  - Normally heated by external tungsten wire heating elements.
  - Sometimes, high-frequency induction rather than resistance heating is also used.

So, what I told you that the source material we can say it is a boat which is shown here or it can be coils, this is a basket, so it can be different materials in which we can load the source material or deposition material. So, we have tungsten wire sources; tungsten is a used as a crucible material and these evaporant wets tungsten and retained by surface tension.

The refractory metal sheet sources are tungsten, tantalum and molybdenum. And for sublimation furnaces we use sulfides, selenides and some oxides as pellets, while for crucible materials we use generally Al2O3 or BN, graphite and refractory metals. Normally heated by external tungsten wire of heating elements and sometimes high frequency induction heater can be used for heating these particular source materials.

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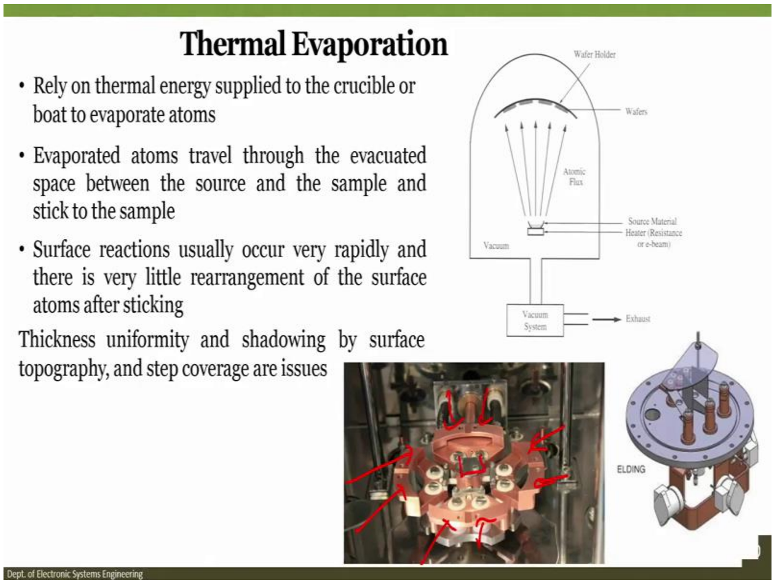

Thermal Evaporation

- Rely on thermal energy supplied to the crucible or boat to evaporate atoms
- Evaporated atoms travel through the evacuated space between the source and the sample and stick to the sample
- Surface reactions usually occur very rapidly and there is very little rearrangement of the surface atoms after sticking

Thickness uniformity and shadowing by surface topography, and step coverage are issues

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So, let us understand the first topic of the PVD and that is our thermal evaporation system. So, the thermal evaporation system as you can see there is a chamber and then there are wafers which are loaded on the wafer holder, there is a source in which you have loaded the material and then when you heat this material, it will, so this is an actual schematic of a, within a chamber, there are 4 source holders, 1, 2, 3 and 4.

And if you see carefully, you can load the boat between these two, between these two, these two and these two. So, the point is this is 4 source system and you apply a voltage through this particular bar, this is 1, voltage 2, voltage 3 and voltage 4 and the boat that is connected between these two electrode will get heated up when you apply a high voltage because of the low resistance of the boat.

So, surface reactions usually occur very rapidly and there is very little rearrangement as we have seen earlier. Thickness uniformity and shadowing by surface topography and step coverage are some of the issues. Let me play 3 videos which are about the thermal evaporation system, here is the video 1.

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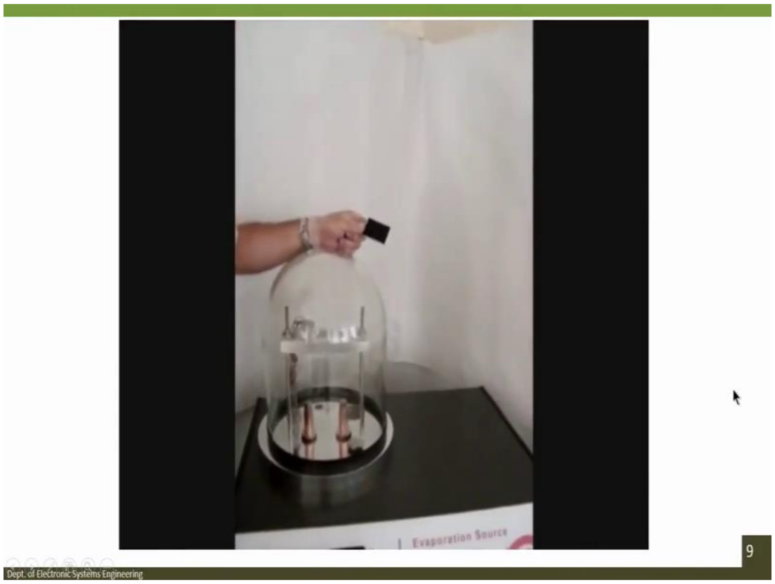
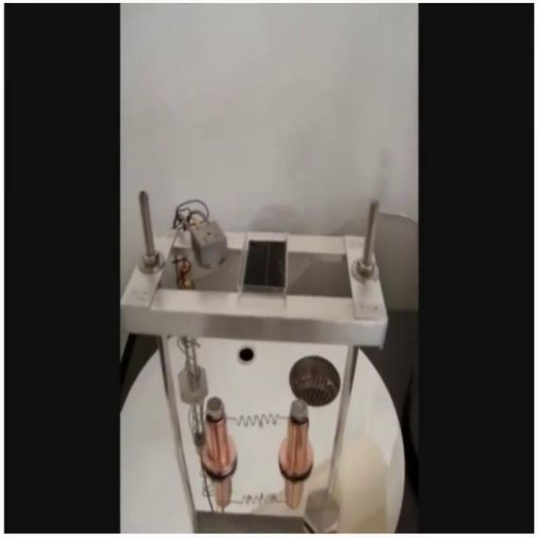


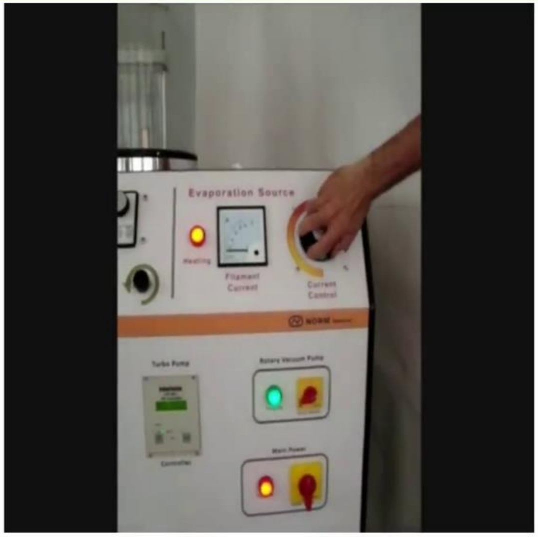



Evaporation Sourc
Heating


Evaporation Sour


Evaporat
NO
YES
Water
Vent
Valve
Heating
EVP 3030-80
Pressure
Controller
Turbo Pump
Controller


Turbo Pump
ALCATEL
ACT 200 T
Controller

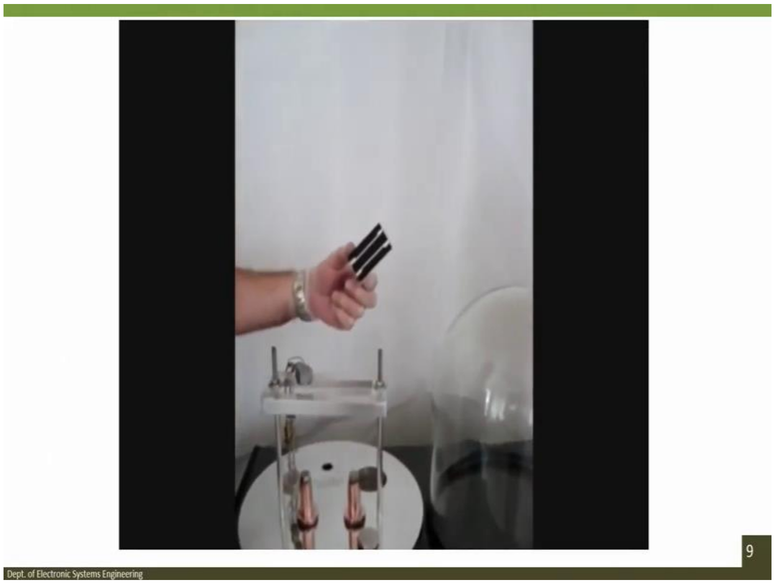

Evaporation Source
Heating
Filament Current
Current Control
Turbo Pump
Controller
Rotary Vacuum Pump
Main Power


Evaporation Source
NO
YES
Water
Vent Valve
Heating
Filament Current
EVP 3030-80
Pressure
Turbo Pump


Rate Å/s
Thickness kÅ
3.2
1.891
Time
INFICON
SQM-160
NO
YES
Water
Vent
Valve
EVP 3030-80
Pressure


Evaporation Sour
Heating
Filament Current
EVP 3030-80
Pressure
Controller
Turbo Pump
Controller


Evaporation Source
Heating
Filament Current
Current Control
Vent Valve



Let us see how this occurs in the actual system, I will play this video as well.

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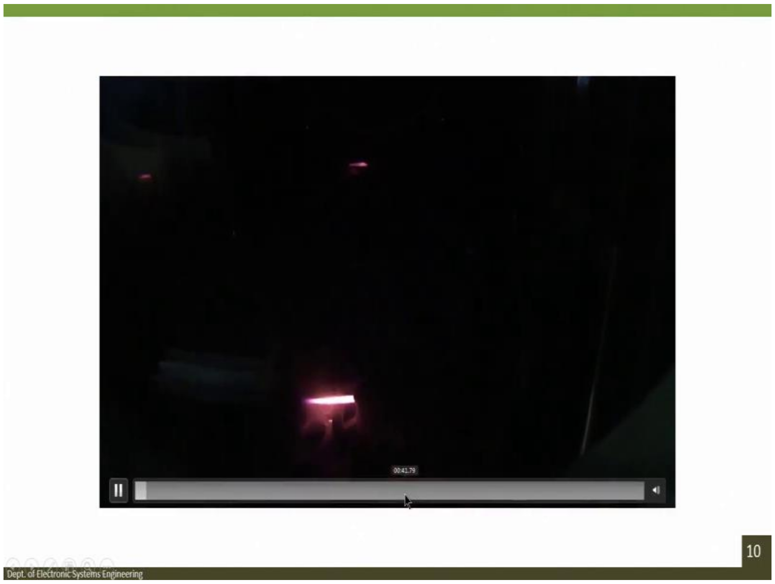

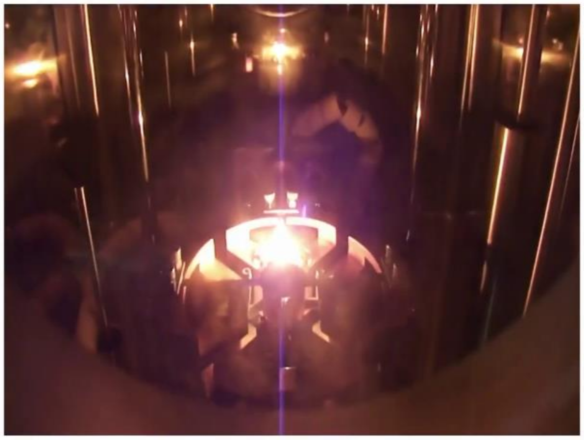

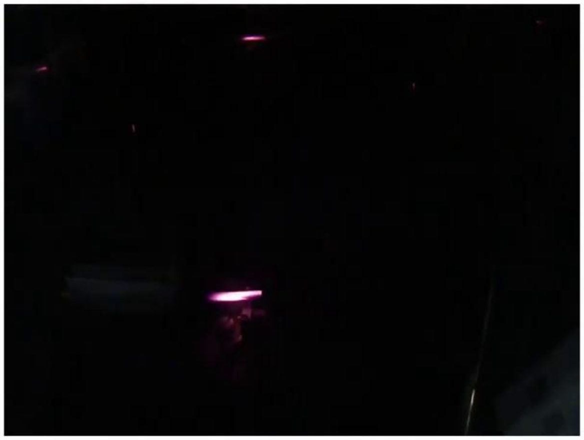

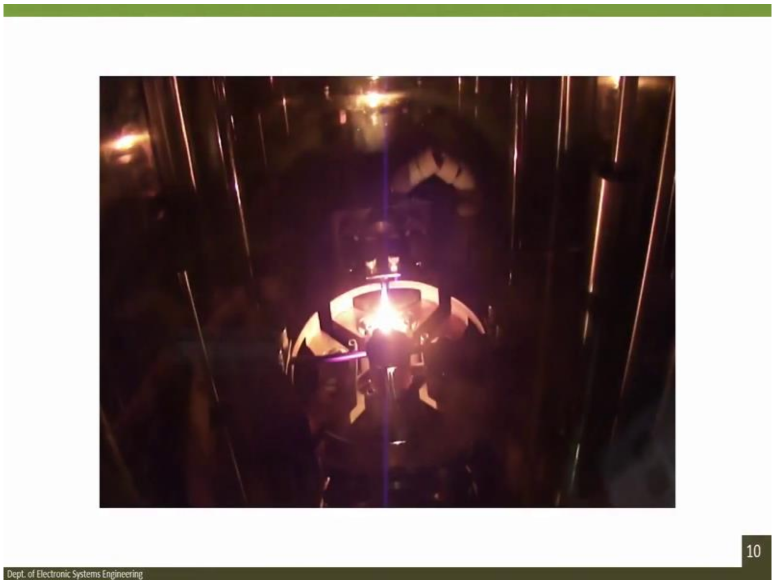

So, what you see here is the, these are videos for thermal evaporation system.

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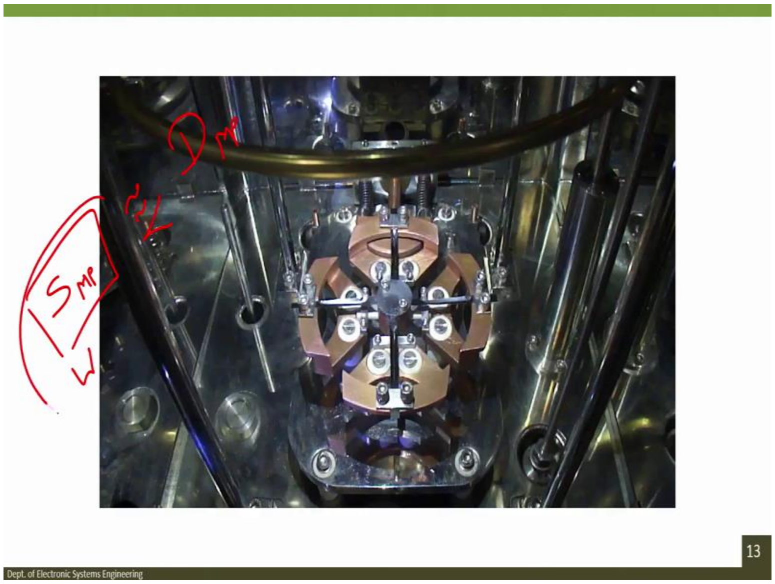

Now if you see the system again, if you see the slide and, let us finish this module here then I will come with the next one, there is an alloy and what of kind of temperature can be used but for this module, you understand that this is a another angle of the vacuum system. These are the chamber walls, and these are the 4 sources, 1, 2, 3 and 4, in which you can load the material. Then there is a, within a, this is a camera installed within the chamber, this is where you can see it.

And you have instead of, in case of thermal evaporation system, it is by heat but there is a difficulty with thermal evaporation and what can be the difficulty, the difficulty is that if your source's melting point, melting point of the source material or source substrate or source metal which can be tungsten, if it is equal to or less than your deposition material melting point, what will happen? That the source will melt early before your deposition material. This should not happen.

Because if you see the holder, this particular holder, this should not get melt, we want the source material to get melt which is the, this material, which is loaded inside the source and not the source itself. So, when you have the deposition material temperature extremely high compare to source material, in this case thermal evaporation cannot be used and for that we a system called e-beam evaporation that we will talk in a next module. But let us finish this one by talking about one more slide which is a thermal evaporation of alloy.

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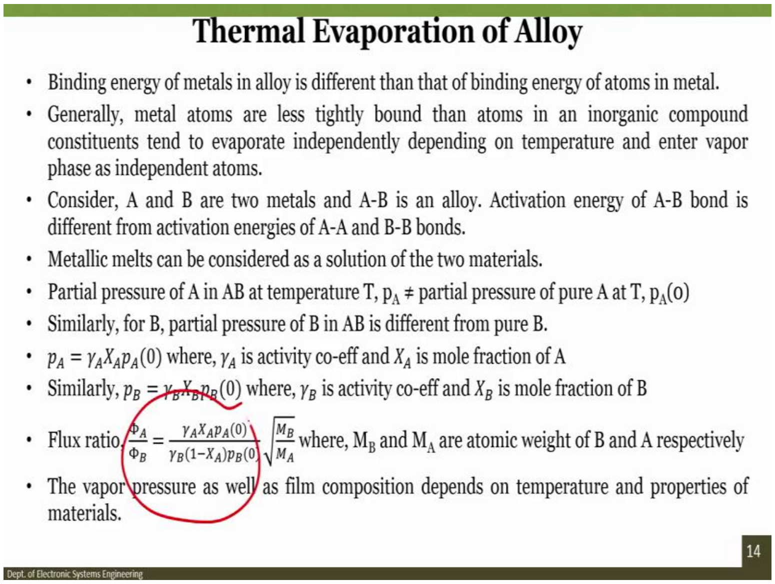

Thermal Evaporation of Alloy

- Binding energy of metals in alloy is different than that of binding energy of atoms in metal.
- Generally, metal atoms are less tightly bound than atoms in an inorganic compound constituents tend to evaporate independently depending on temperature and enter vapor phase as independent atoms.
- Consider, A and B are two metals and A-B is an alloy. Activation energy of A-B bond is different from activation energies of A-A and B-B bonds.
- Metallic melts can be considered as a solution of the two materials.
- Partial pressure of A in AB at temperature T, $p_A \neq$ partial pressure of pure A at T, $p_A(0)$
- Similarly, for B, partial pressure of B in AB is different from pure B.
- $p_A = \gamma_A X_A p_A(0)$ where, γ_A is activity co-eff and X_A is mole fraction of A
- Similarly, $p_B = \gamma_B X_B p_B(0)$ where, γ_B is activity co-eff and X_B is mole fraction of B
- Flux ratio, $\frac{\Phi_A}{\Phi_B} = \frac{\gamma_A X_A p_A(0)}{\gamma_B (1-X_A) p_B(0)} \sqrt{\frac{M_B}{M_A}}$ where, M_B and M_A are atomic weight of B and A respectively
- The vapor pressure as well as film composition depends on temperature and properties of materials.

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Now if you want to have thermal evaporation of alloy, then you already know that binding energy of metals in alloy is different than that binding energy of atoms in metals and generally metal atoms are less tightly bound than atoms in an inorganic compound, so if you take an example, so that what happens is one will evaporate faster than the second one. So, if we take an example, A and B are two materials and A-B is an alloy, and activation energy of A-B bond is different from activation energy of A-A and B-B bonds. And that means, a metallic melts can be considered as a solution of two materials. Partial pressure of A in AB at temperature T, let us consider as pA cannot be equal to partial pressure of your A at T $p_A(0)$.

Similarly, in case for B, so yet we can have equation as pA equals to we can have gamma A into XA pA or rho A(0), where gamma A is your activity coefficient and XA is a mole fraction of A. Similarly, we can have pB which can be equal to gamma B XB pB 0, where gamma B is activity coefficient and XB is a mole fraction. So, the flux ratio can be given by this equation which is shown here, of course this is a part of the equation under root MB by MA, where MB and MA are the atomic weight of B and A respectively and that mean that the vapor pressure as well as film composition depends on temperature and the properties of material.

In some cases it is difficult to deposit allow, there are some, there are techniques called two source evaporation techniques, two source evaporation technique, where both the sources are heated at different temperature, so that to evaporate the metals simultaneously and form the allow. However, like I said there will be cases where your deposition (temper), the material that you want to deposits, its temperature is extremely high compare to your source material, for example if you want to deposit silicon dioxide, silicon dioxide melting point is very high compared to tungsten, it is very difficult to deposit silicon dioxide in thermal evaporation.

Same thing goes silicon nitrite, very difficult to deposit in thermal evaporation and that is why there is another technique called electron-beaming evaporation, which we will see in the next module and we will go in detail about how the e-beaming evaporation works and how it can overcome the limitations post by thermal evaporation, where is the melting point issue would not be there.

So, till then you just look at this module, it is a very small module to help you out and not to confuse you with different PVD techniques. We will just look at PVD, we will not go into depth of CVD again the reason of teaching you PVD techniques, is that we use PVD technique for depositing metal and those metal can be patterned using lithography to form your electrodes which we can use for measuring the body vitals either it is ECG or EMG or EEG.