Technology of Surface Coating
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Lecture No 11
Vacuum Evaporation Depositions

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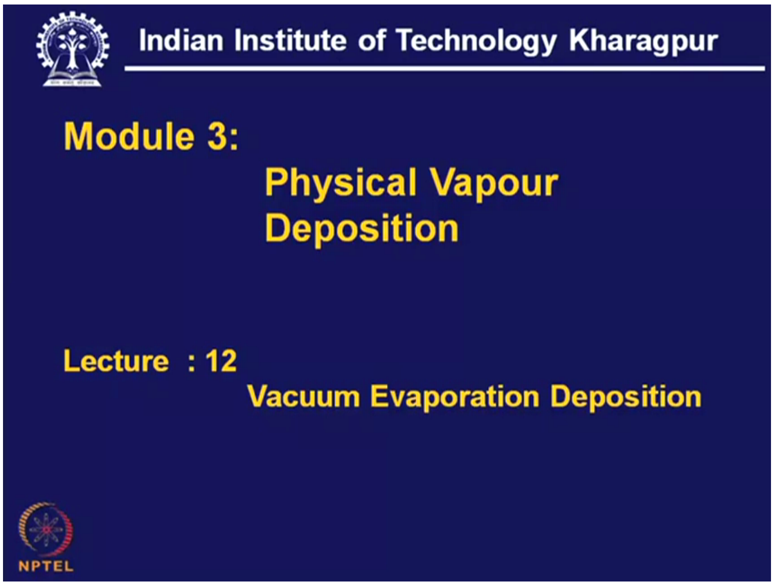

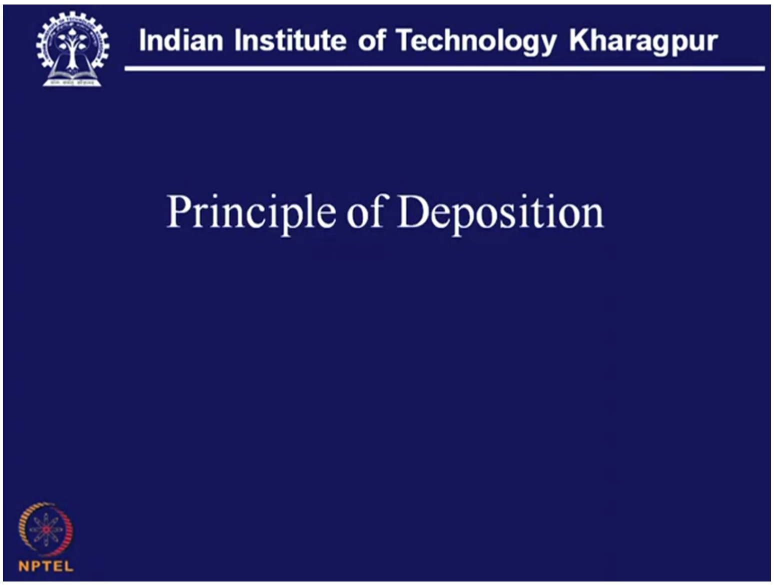

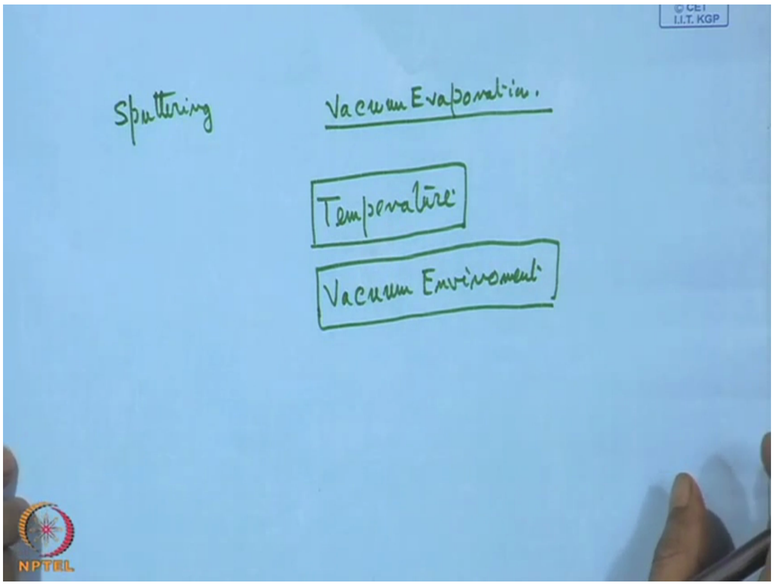

Deposition by vacuum evaporation. Now we have various types of depositions and what we can see here physical vapour deposition and chemical vapour deposition and in the physical vapour deposition basically it can be broadly classified into sputtering and vacuum evaporation, so principal of deposition that we should understand. Here since it is vacuum evaporation that means material has to evaporate under vacuum environment, so for evaporation of the material we need a heat source that means we need temperature this is number 1 and number 2 a vacuum environment.

So when we put them together this vacuum environment and the temperature or the heats source then the materials, then the source material, it can be metal, it can be a ceramic, it may start depending upon its melting point or if it is sublime that means straightforward from solid to vapour phase, it may start sublimation, so it depends upon its sublimation temperature or the melting point that is the characteristics of the metal or the material it can be ceramic also ceramic material and at the same time what is the saturation vapour pressure at the point at the temperature under consideration, so vacuum environment means we have to have a pressure equilibrium vapour pressure which corresponds to that evaporation point and the materials will starts evaporating and then the whole task is like that we can have a quick look into this.

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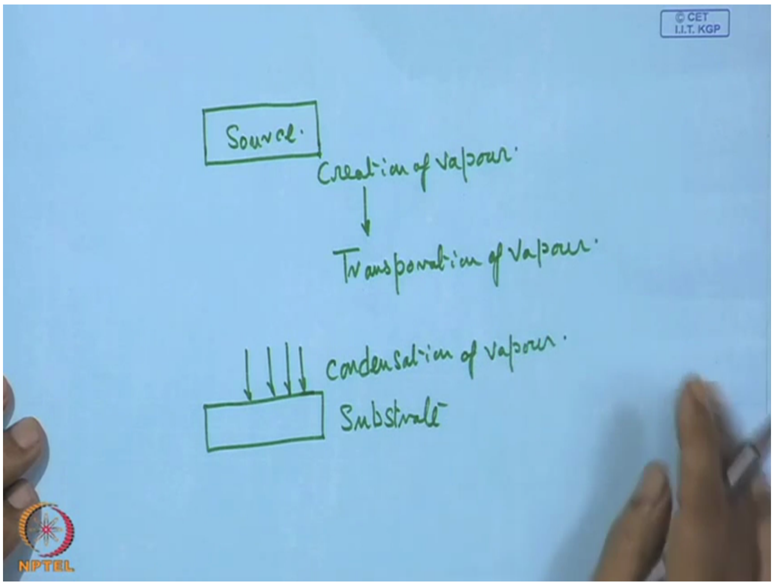

Say this is actually the source, source that means this is the source of the material for coating and here what we have, we have the substrate, so it is creation of vapour for that we need vacuum and heat source then there must be transportation, transportation of the vapour. It has to be carried and then finally this vapour comes over the surface and here it starts condensing, so generation of vapour, transportation of the vapour and here condensation of the vapour. So these are actually 3 steps in this deposition by vacuum evaporation, condensation of vapour.

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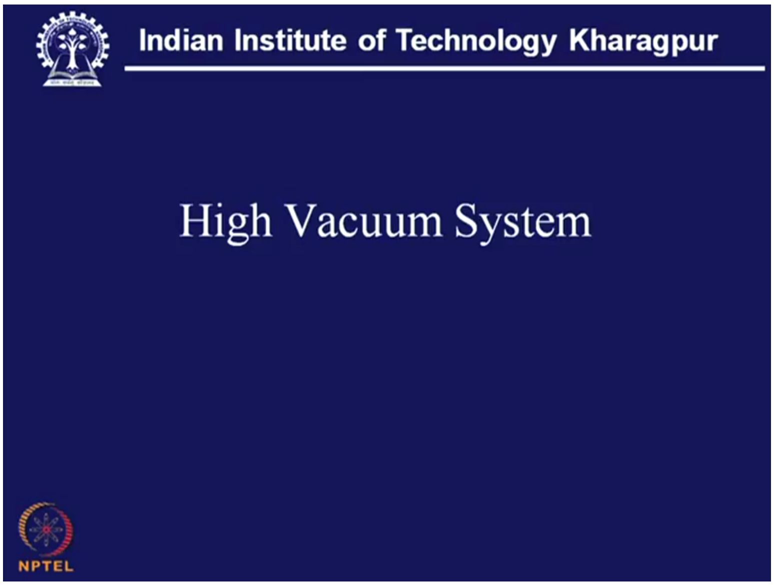

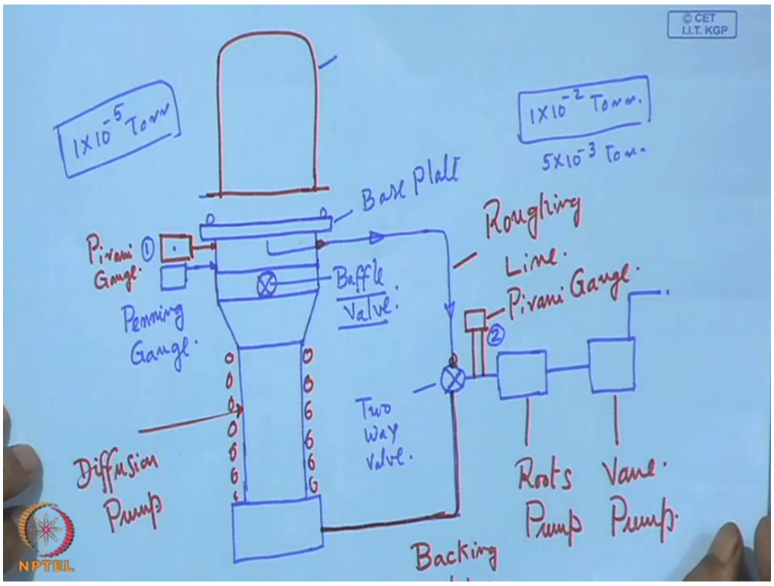

Now what we need a high vacuum system, so a high vacuum system means it is one equipment capable of making this high-volume environment. Say this is a chamber, vacuum chamber and just beneath it what we have? We have the inter-vacuum system, this is just a base plate then here we have a collar and then what we have further to this, we have here a diffusion pump. Now from this side too we have another line, so this is the plumbing work, so these 2 are jointed here and then we have 2 pumps in series. So this side from this point to this point this side we call it roughing line and this side, so from this point, so this side so this is actually a backing line and this is a diffusion pump, so which is water cooled, so here we have cooling coil, so this is actually the diffusion pump.

So here what we have roots pump, this is vane pump and what we have? Just here one pirani gauge, so these are all the peripherals of this vacuum system and here we have 2 gauges, so this one from this point we have one Pirani gauge and another that can be another one penning gauge. Now this things so this is the base plate and what we can have here this is actually a position where we can have one baffle valve. So what is done, when this dome, this vessel, this covered up this vacuum chamber that is on this with the help of this gasket on this one then what we can do, this baffle valve can be fully closed and this side this backing line and the diffusion pump this path is blocked.

So the air is sucked through this line and that is called the roughing operation and the vacuum will be shown by this pirani gauge one here and this is gauge 2, so this is Pirani gauge one and that is Pirani gauge 2, so that will show the pressure and when we have a pressure in the order of 10 to the power minus 2 torr that time this line should be activated that means here we have a 2 way valve, 2 way valve so either this passage can be opened or this one. It can also have one neutral position, so once we reach in this order or 5 into 10 to the power minus 3 torr, this line will be blocked and by that time diffusion pump will be warmed up and ready and so now the air will be brought down air will be sucked the residual through this line through this diffusion pump and this roots pump and vane pump these are these are the backing support.

So that will give the required support to this diffusion pump to take and with that a pressure reasonably, a pressure greater than 10 to the power minus 5 torr should be obtain with a good system with minimum lit. It is not difficult to attain a pressure vacuum better than 1 into 10 to the power minus 5, so this is actually a high vacuum system what is necessary for evaporation. Now inside this chamber all this evaporation or heating of the source material, then creation of the vapour, transportation of the vapour and condensation of the vapour that can be done within this chamber and this can be done.

So the material can be source material can be say it can be granule, it can be powder, it can be even where it depends upon the way it is available and also at the same time does heats source, heat source can be a resistance type, it can be inductance type, it can be leisure or it can be electron beam gun. So these are the various technological issues which can be handled depending upon the availability and suitability for a particular case particular material and it is why case one has to make the right kind of judgement to have the all the peripherals properly chosen and so that this process can be conducted in a very convenient way, so this is actually the high vacuum system and then what are the characteristic features of evaporation that also we should little examine.

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Number 1 it is actually one of the oldest and one of the simplest technology no doubt. So only to have vacuum and then heated up then at a particular pressure depending upon the vapour pressure each material has its own saturation vapour pressure at a particular temperature. Say for example silver has a saturation vapour pressure at 1000 degree of 1 into 10 to the power minus 1 torr whereas titanium as a saturation vapour pressure in the order of 1 into 10 to the power minus 5 torr at 1000 degree. So this way one can have the proper mapping of the whole history of each material and the equilibrium vapour pressure and temperature that relationship can be followed and accordingly the process can be conducted.

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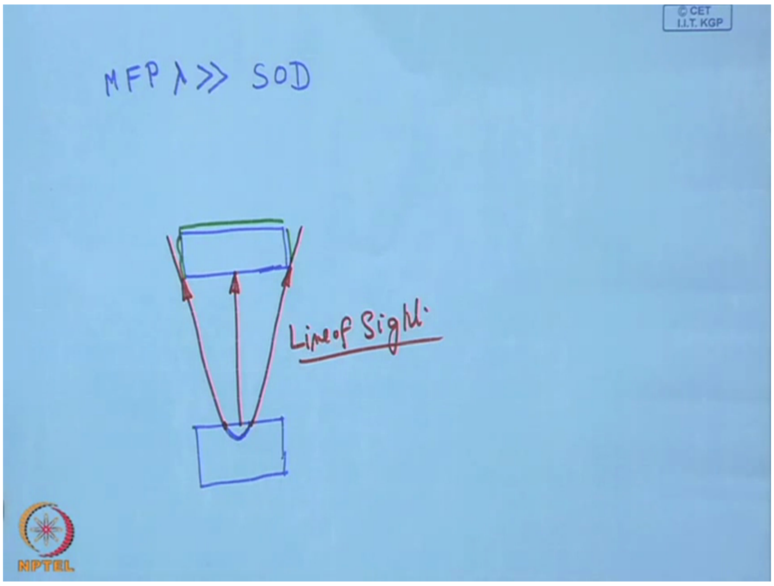

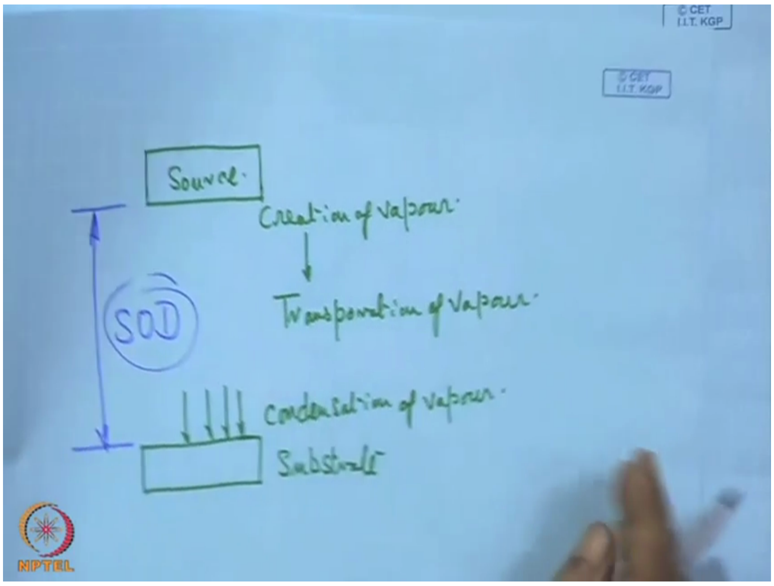

So characteristic feature of vacuum evaporation, it is that in this case main free path of this particle which are on their flight from the source to the substrate its main free main free path that is main free path that this lambda that is greater than stand of distance SOD that is between the substrate and the source, so that means this one we can immediately referred to this, so source and substrate this is actually this SOD, SOD so this is actually much less compared to main free path. Then 2nd one the energy with which this incoming spaces which arrives on the substrate surface their energy level is rather low in comparison to (())(16:05) particles which are emitting from the cathode surface and arriving at the substrate surface, so their energy level is higher.

So with this what we can expect that material which is arriving on the surface, they cannot be having high density of packing during condensation and at the same time adhesion is also not expectedly very high, so density of the film through this process of evaporation and condensation plus its adhesion to the substrate surface, they are not remarkable at all and at the same time more importantly from the technological point of view one has to also pay attention to this that means the flow of the material it is actually the line of sight. So we can illustrate this point say here we have a source material and this may be the heater and this may be a Crucible and over that what we have material a substrate which is facing this source and we expect stream of evaporated material that will move and if we follow these other 2 extremities.

So it will go like this and it will go like this and will be here, so this is the flight path, so what we can see, so this is going to be a line of sight, line of sight that means whatever it can see that part will be only covered, so what we can conclude very easily neither the back it is out of question even in the side this cannot be also coated, so this is also another shortcoming from technological point of view but it is very simple, so for quick metallisation of the ceramic surface or for some coating for some very quick preparation of some specimen this is one of the very simple process of doing the thing of course by modification of this process and by incorporating other technologies within the basic with the basic principle of vacuum evaporation we can also upgrade this technology to suit a particular commercial use and industrialisation of this thing and be also possible, so we have seen this various characteristic feature

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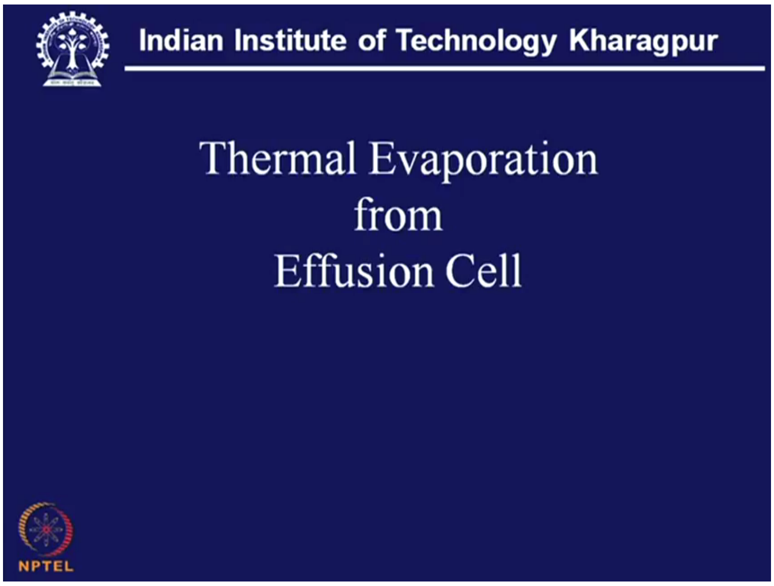

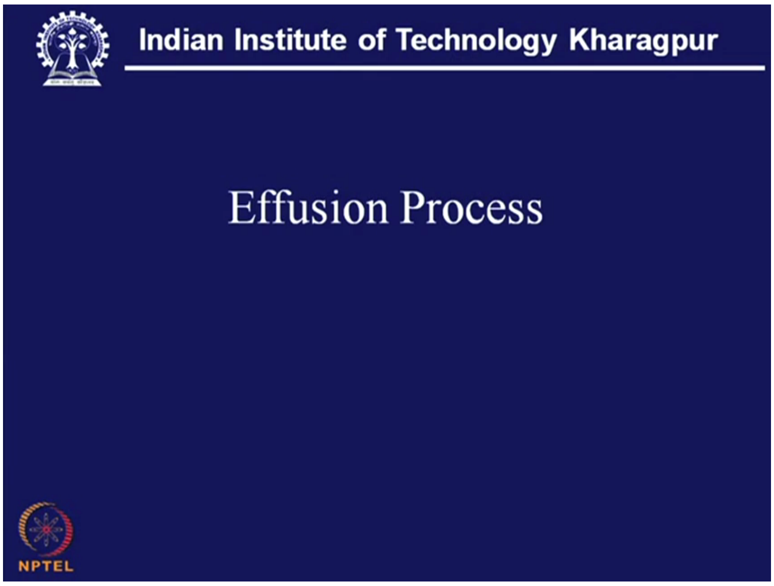

Now thermal evaporation from effusion cell, so this is the if you like to understand this basic principle of evaporation, we have to look into this very concept of effusion cell, so what is this effusion cell or what is this effusion process. Now effusion process is a process of evaporation of the material that means in this case from this cell which is nothing but a container and from this container, the material, the evaporate material will keep on escaping and during this escaping there should not be any clash that means it is escaping in a clash less or collision less fashion or manner and that is actually called effusion process that means escapement of this vapour in a collision less fashion and that is effusion process.

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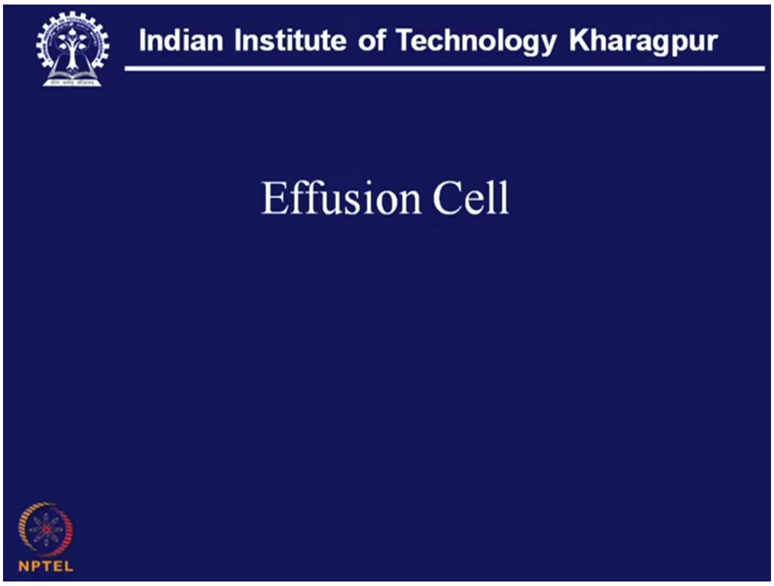

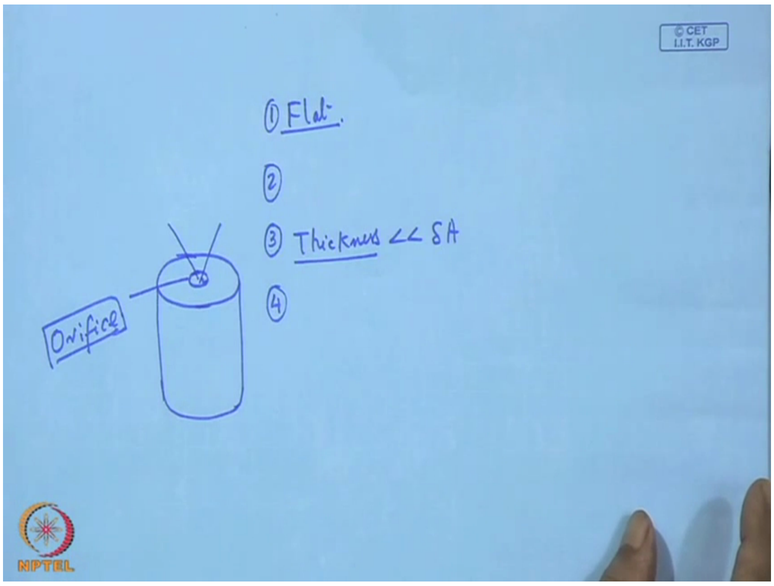

Now what is effusion cell, fusion cell it is almost like a container, say this is one container and the top of that we have a small opening that is say an orifice and this orifice through this material will escape without any collision that is actually effusion cell and another thing size of this orifice is less, much less compared to the size of this container. So without any collision the material will keep on skipping emerging out and this orifice size is very small compared to the overall size of this container containing this evaporating materials.

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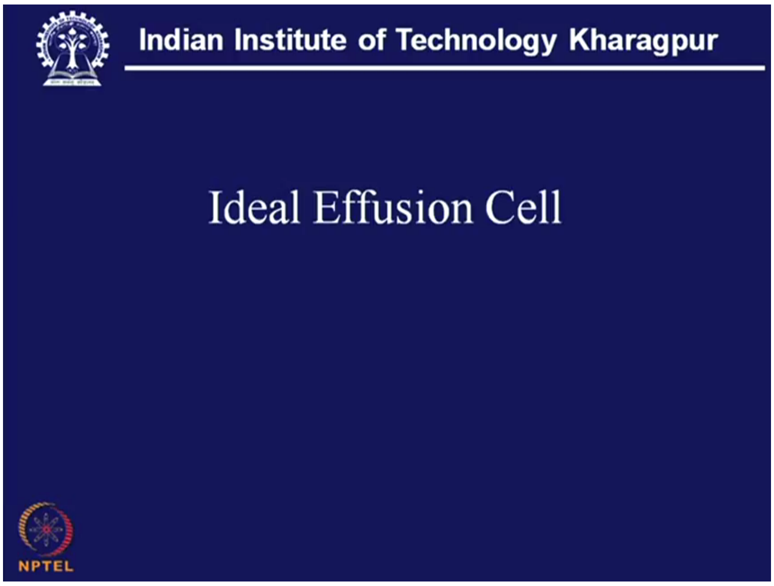

Now ideal effusion cell, ideal effusion cell means here this size is rather small then that surface of this effusion cell that is also flat that means this effusion cell the surface of this effusion cell is flat, surface of the effusion cell that is flat. Then this size this orifice sizes very small compared to the size of us cell. Number 3 system number 1 and number 2 is the size orifice which should be very small, number 3 is thickness of this container this thickness. Thickness of the container is very small compared to the size of the orifice, so this is actually another condition number 4^{th} that is what we say that within this effusion cell, the vapour is in equilibrium with the liquid with the belt, so within this where which is the source of vapour generation at means this vapour is in equilibrium with the melt or the with the liquid. So when this conditions are fulfilled then we can call it an ideal fusion cell.

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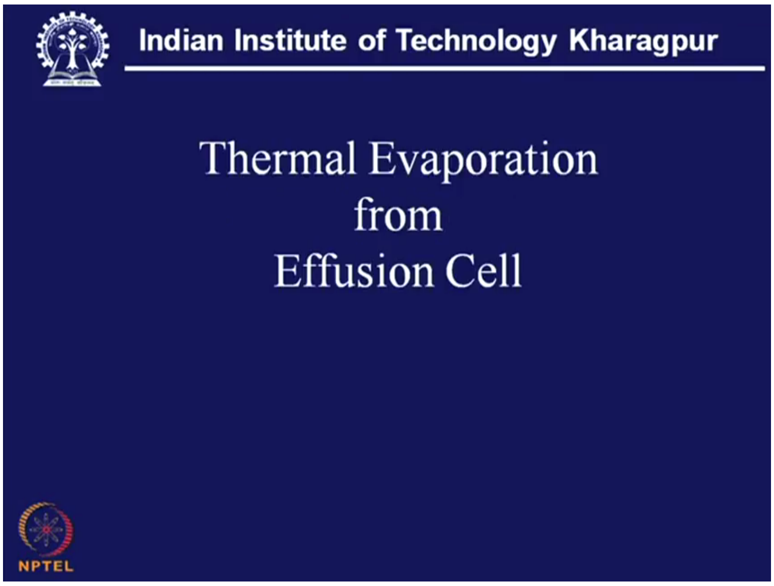

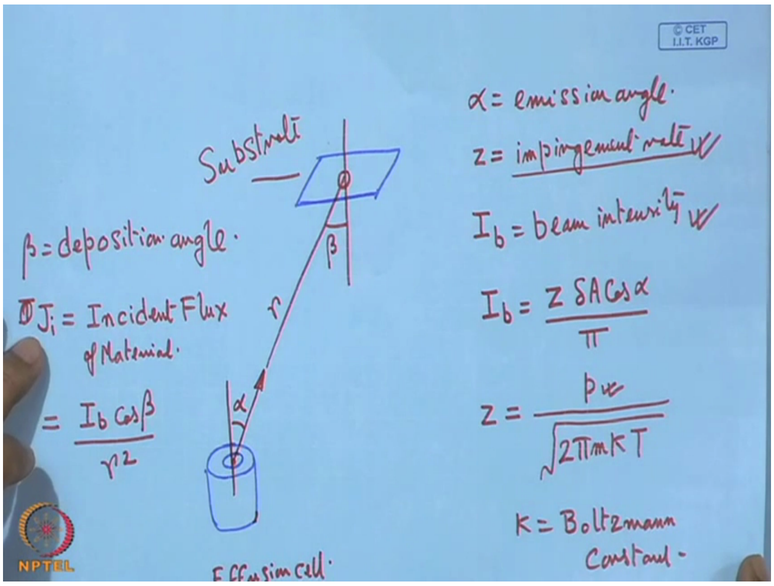

Now here comes if we go back to thermal evaporation effusion cell there are we can illustrate this thing by this a simple diagram, so let us have a look into this diagram, so here we have one cell, so this is the cell effusion cell and here maybe we have the substrate. Now what is we have few terms number 1 what we call here Alpha this is called Alpha emission angle and this is actually a flux, so we have another term z that is called impingement rate, impingement rate means number of particle emerging or escaping or number of particle striking the valve of this effusion cell per unit area per second that means z is the number of particles which are striking a valve of this effusion cell per unit area of the cell and per second effusion cell.

So then this is called z, then we have another term I b that is called beam intensity, beam intensity means number of particles which are moving or flowing per unit solid angle per second, so actually this I b that depends on z and this will be given by this I b is equal to actually z into delta A into cos Alpha divided by pi, so that will be the relation, so z is the impingement rate and z can be also given by this from this kinetic theory that means this is actually 2 pi m KT that means this is the prevailing pressure inside the effusion cell, m is the mass of the particle, T is the temperature of this effusion cell and K is the Boltzmann constant. So we can have and this is called a mission angle now with this beam intensity that is going to reach the substrate surface, substrate surface.

So it is going to reach this way and here we have few more terminologies to be to get familiar with. Now here we can have another angle beta and this beta we call it beta is actually deposition angle and then what we called incident flux, incident flux of material, flux of and that will be given by this I b into cos beta divided by r square. So we can step-by-step understand impingement rate, beam intensity and now incident flux and here what is r, r is actually the distance between this point of interest on the substrate and from this point on this effusion cell and r is the distance between this so that is the distance of this flight path, so this is the relation. So it is the incident flux of material which is arriving on the substrate surface. However what we are interested in, we are also interested in the, we are interested in this condensation flux that means incident flux is the number of particle arriving per unit area per second of the substrate but condensation flux is different and it is not necessarily equal to J i rather it will be less than that of J i and we shall also consider that.

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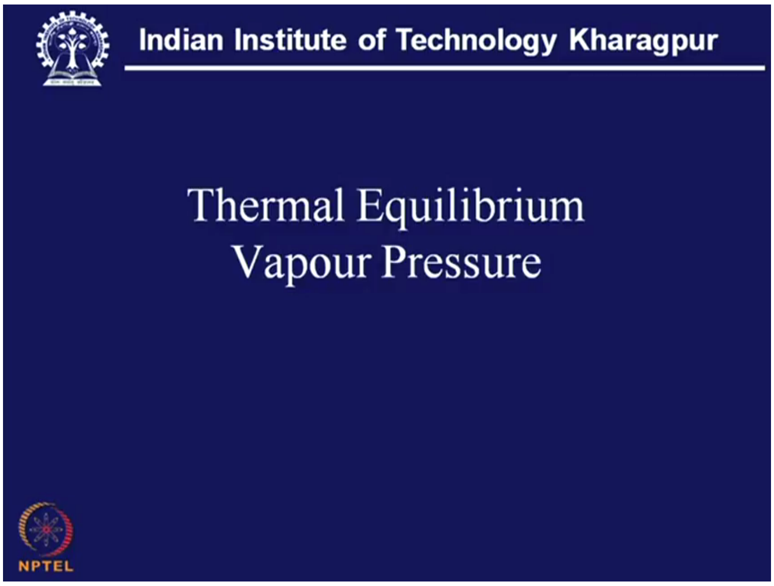

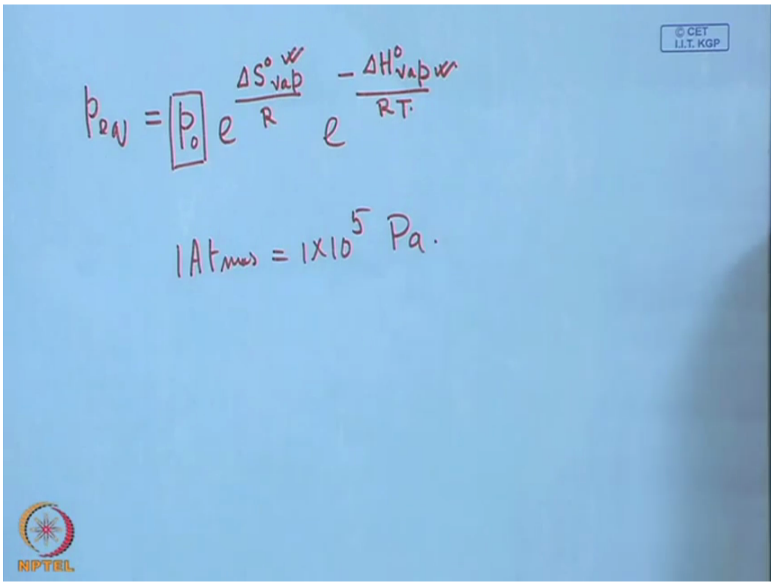

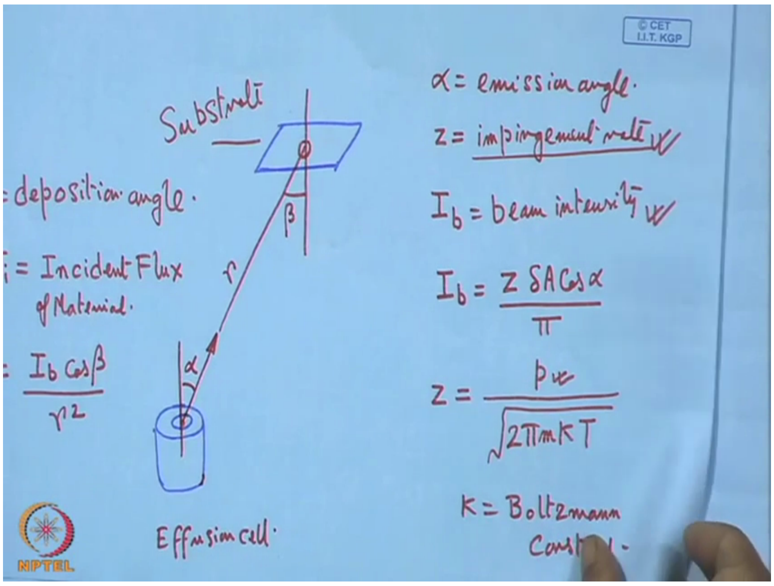

So here what we see thermal equilibrium vapour pressure, so thermal equilibrium vapour pressure that is actually given by this is P o into e to the power delta, so vapour to the power r into e to the power minus delta H 0 vapour by RT. Now this is actually the equilibrium vapour pressure and that is the standard pressure, standard pressure means 1 atmosphere is equal to 1 into 10 to the power 5 Pa. So that is and here this vapour pressure we can determining considering what is this, it is actually entropia vaporization, this is enthalpy evaporation and for a particular material this can be found out at a standard state and that is the temperature in question and from that this equilibrium vapour pressure can be determining and from this equilibrium vapour pressure we are in a position to determine this impingement rate, beam intensity if we know the value of Alpha and then also we can determining the incident flux and also now condensation flux.

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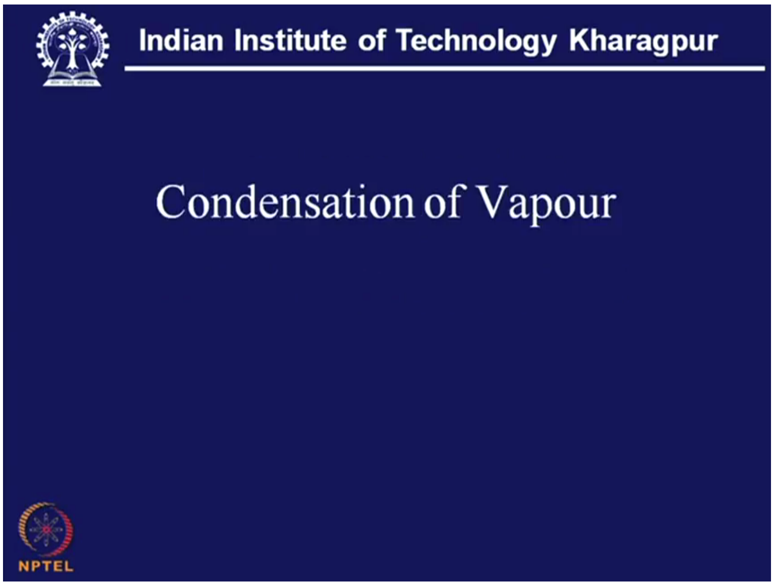

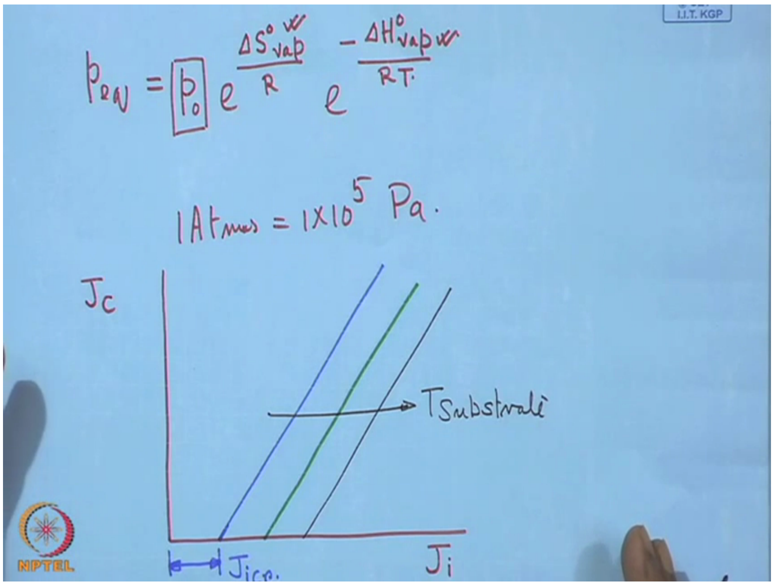

Now condensation vapour, now condensation vapour is actually what we can see that we can illustrate with the help of this diagram here, so this is actually J incident flux J i and this is J c J condensation and they are actually co-related but the line if we draw a line is line does not pass through 0 but it is a little bit off set, so here this is the line that means this is actually critical J i critical this is the minimum quantity, this is the minimum quantity necessary to initiate the condensation process and this line what we have drawn here, this line can also shift, so for example this line can be also shifted or it can have further shift.

Now on which does it depend actually what happens, this condensation is taking place on the substrate surface, so it is the substrate surface temperature that matters, so when we see this blue, green and black line this indicates this is increasing substrate temperature. So the substrate temperature, if we increase the substrate temperature then the requirement then the amount of incident flux that is necessary will be more that means the quantity of incident flux which is necessary to start the condensation that quantity will increase obviously if we like to have lower critical value, the substrate temperature should be very low compared to the source temperature in this case it is the effusion cell temperature.

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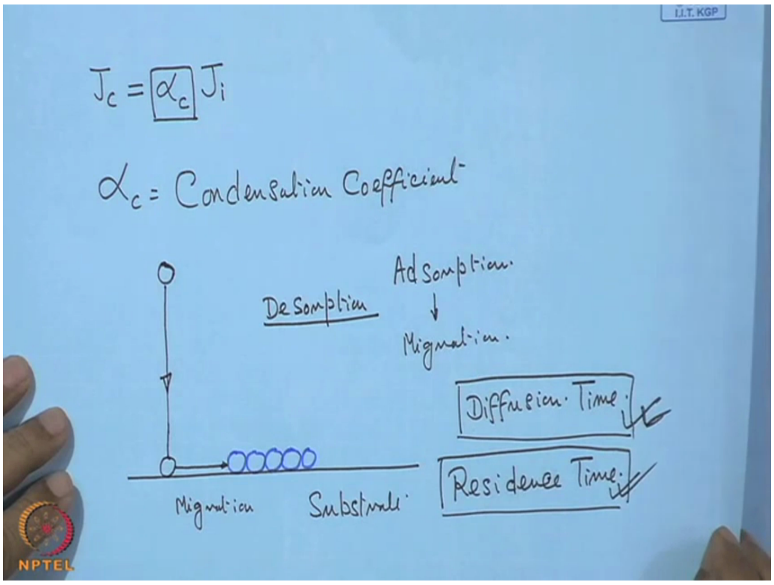

And this can be also illustrated this way that condensation flux and condensation coefficient. Now what we can say here that condensation flux, so condensation flux J c equals to actually what we write Alpha c into J i, alpha c into J i that means here we have introduced one term and this is called condensation coefficient. So this condensation coefficient it depends upon the initial reflection and also the ability of this material to get absorbed. Say for example this is the substrate surface and one material that is in its flight, it gets it arrives here.

So it is actually in the flight so it arrives here and if we have already sum of the particle which are already got attached strongly, so then the tendency of this one will be to move in this side and to get attached on this side, so this is actually the moment migration, so this is actually migration and this will get diffuse to this. So 1st adsorption followed by migration, migration to some of the particles or particle or atom already attached to this substrate surface, so this attachment that needs some time and that is called diffusion time and on the other side what we have? Residence time.

So naturally this diffusion time and residence time these are the 2 things one should look in otherwise this material can also it we can lose this material by desorption and that is why we have this condensation coefficient it depends upon also contamination of the substrate and also one thing is adsorption that is aided by aided physically or it can be aided chemical by some chemical affinity. So when it is physical adsorption substrate cleanliness virginity of the substrate is extremely important otherwise there will be this condensation coefficient that value will be rather low. However apart from this what we have to also look in that is actually called re-evaporation, re-evaporation of the material from the surface.

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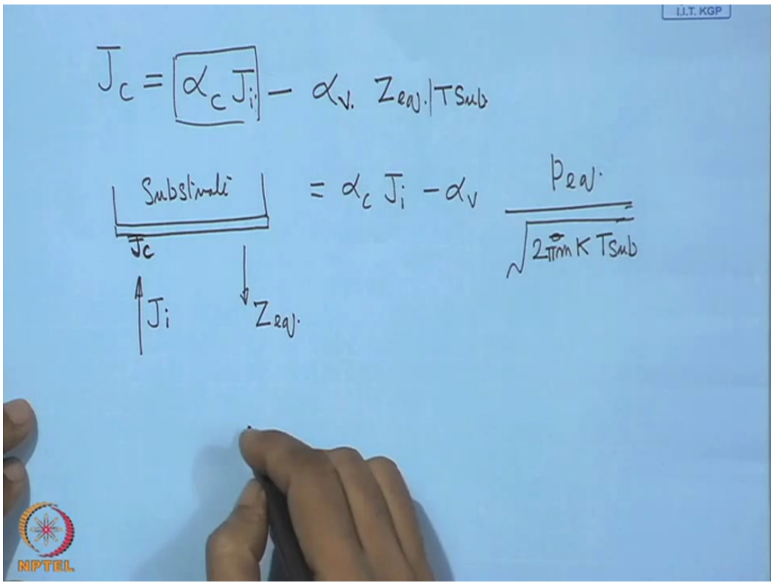

So what we can say in this case that re-evaporation flux and re-evaporation coefficient, so what we can write here condensation flux actually we should write it into J i minus Alpha v into z equilibrium in actually it should be T substrate, so T substrate means this is this is impingement rate, impingement rate from the substrate surface and this impingement rate depends upon the temperature of the substrate and the pressure prevailing nearby, so this 2 will determine what will be the impingement rate from the surface and at the same time you have to also multiply with another term what we call re-evaporation coefficient that means if we see one flux which is incoming, so this is the substrate.

So one is actually J i that is on this side and another is Z eq. that is the impingement rate that is really re-evaporation rate and finally what we have here on the surface that is actually J c. So that is the condensation flux, so this is incoming and this is outgoing because of this re-evaporation and this can be further written just like Alpha c J i minus Alpha v and this one we can also write p equilibrium divided by 2 pi m K into T substrate, so it is actually 2 pi m K into 2 pi m K into T substrate that is the substrate temperature and from there we can determining this value and so always it should be greater than 1 that means this difference should be greater than 1 or what we can write just one expression that means the expression what we call supersaturation.

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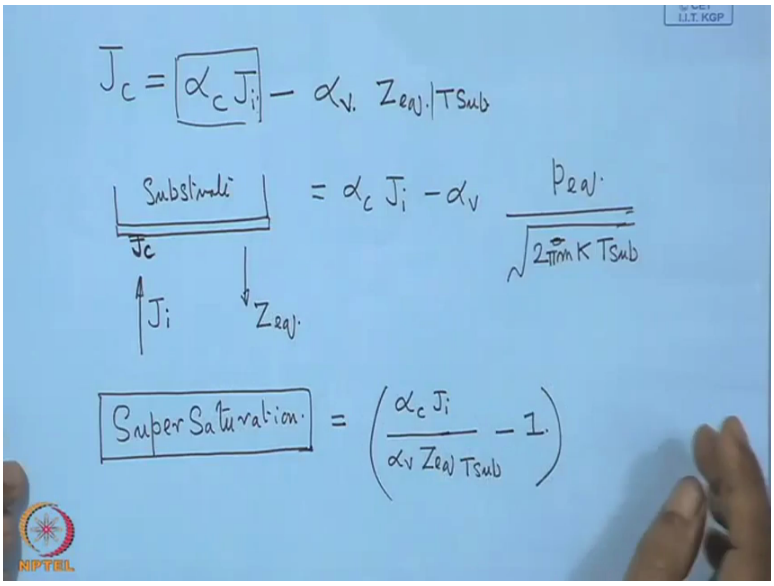

Supersaturation that means in this case we have to right this can be written as alpha c J i by alpha v Z q T substrate that is minus 1 that means this is actually called supersaturation and supersaturation means the pressure above the substrate, pressure about the substrate must be moved greater than the equilibrium vapour pressure to have reproducible condensation that means if we have a higher pressure just about the substrate surface then the question of re-evaporation of the material, the chances will be removed. So this is actually called the super saturation, what does it mean?

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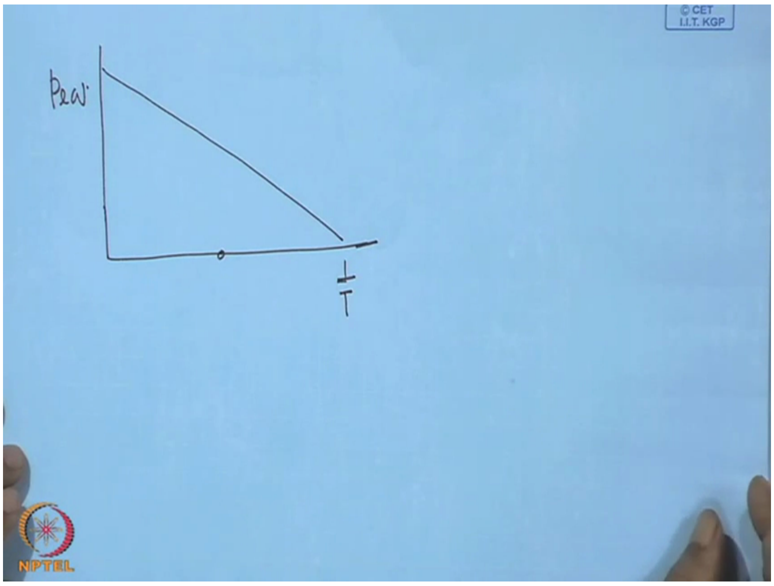

That means if we have p equilibrium we can put as upon 1 upon T so that is called the equilibrium vapour pressure, so for a particular value of T the pressure above the substrate if it is more than that p equilibrium, so on that side there will be no re-evaporation and chances of rather condensation of the material that will be certain and this can be maintained just by keeping low substrate temperature and having a higher pressure over the substrate surface. Now this is what we see as the principal of evaporation and in this case what we find that though evaporation follow certain principal and basic rules.

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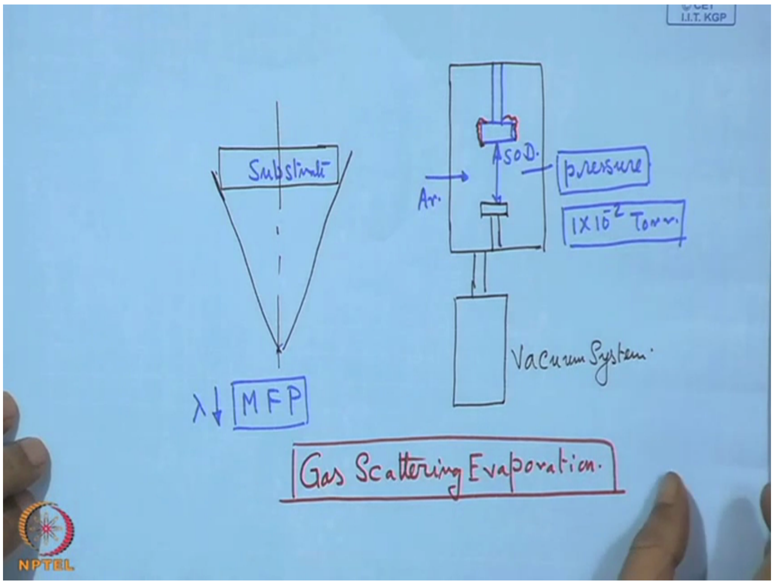

However this evaporation is limited as we have already mentioned that this is the line of sight deposition, line of sight deposition and with this line of sight of deposition hardly we can have any deposition on the surface. Now the situation can be little bit improved just by injecting argon inside that means inside this chamber which is connected to a vacuum system and if we have a source material here and the substrate on this side, so what we can put here argon and with this argon we can increase the pressure inside this chamber that means pressure inside the chamber that can be raise to say for example 1 into 10 to the power minus 2 torr and with that benefit we can achieve.

This is the substrate the benefit what we can achieve in this case this is actually the SOD stand of distance however if we can reduce the main free path, main free path length in that case what we have, more scattering of this material within because of this collision and if we increase the reduce the main free path there will be more collision and more scattering and this scattering it will no longer along a particular direction and it will have a random motion and with this randomised motion what we can have, we can have a flat chance of getting coating over the surfaces which are not exposed uhh, so favourably from this source point, so this is actually known as Gas Scattering Evaporation, Gas Scattering Evaporation.

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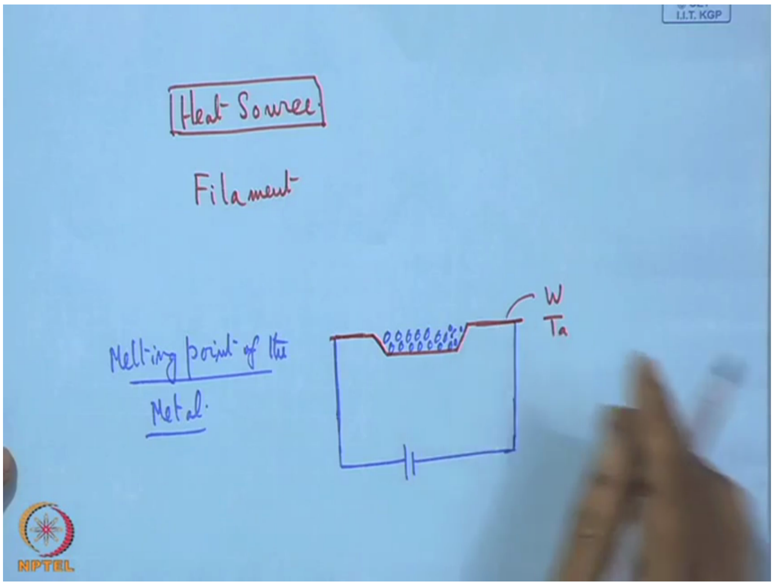

Now the heats source whatever we have mentioned the heats source, the simplest one to carry out this process it is the filament in a form of a boat that means this will be a boat like thing and this is just a tungsten or tantalum filament and where we can put the granules and this one can be used for heating, so this can be just a resistant sitting by giving the power supply with this, this is just like a heater and in that case evaporation is possible but the limit of this is that, it depends upon the melting point of the metal, so that we can, so that we can see whether the heat what is available here that can be good enough to evaporate this material.

Now in this case we can evaporate low melting point materials and this can be done by using this as the heat source however the main problem will be that, if this material reacts with this boat then the melting point of this one is lowered and in the next cycle this cannot be use because of the lowering of the melting point and it will itself will melt and it will break. So for that what is necessary? It is necessary to use a high-temperature heat source like can electron beam gun and here with his electron beam gun it is possible to concentrate high energy density over this material and the same time it is also possible to use this for production purpose for improving the productivity of the process.

Now whatever may be whether it is electron beam of this filament or laser. Now in this case the 2 things what we understand immediately that the limitation of the process is one is the line of sight deposition another is also what we find that the density of the coating and also the adhesion of the coating there are certain limitation and for that there are modification in the process and by which it is possible to have a better coating property with better hardness, better density and better adhesion.

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So with this, what we can summarise that the basic principle of evaporation to follow this principle in practice what we need? We need in principle a heat source and a good environmental or high vacuum and this can be done by using a vacuum system and the vacuum system basically consist of pumping system and the gauges is a low vacuum side and a high vacuum side for evacuating the system from the atmospheric pressure and at the same time the evaporation rate that depends upon the saturation vapour pressure and also the prevalent temperature.

So that means if we can reduce the pressure, we can also reduce the requirement of the temperature or evaporation of the material and at the same time what we have observed that is also the contaminated surface of the evaporant that means the source material or of the substrate that may affect the impingement rate, evaporation rate or also the deposition rate and that will be decided by also the adsorption of the material on the substrate surface and what is necessary in this case to have a reliable condensation of the material with a high rate of deposition?

It is necessary to have a low temperature on the substrate and a high-pressure which is greater than the equilibrium vapour pressure on the substrate this thing can be done either by using commonly a filament heats source or the electron beam source but further modification of this evaporation system is required in or that the coating obtain by this process can have a better property in terms of its density, hardness then growth rate and adhesion with the substrate.