Chemical Engineering Principle of CVD Processes
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Module-3
Lecture 21
CVD Transport Phenomena: aerosol CVD (ACVD)

Good morning and welcome to the next section on our course on chemical engineering principles of CVD process. Starting in the last lecture we are looking at various types of CVD reactors and in the last class we looked at one system of CVD reactors that are known as catalytic CVD reactors or filament CVD reactors. This time we are going to look at a class of CVD reactors that are classified as aerosol CVD reactors.

Now normally as asset several times you try to avoid the formation of aerosol in a CVD reactor because they can interfere with the quality of this CVD film and make it final configuration very different from what you would expect based from vapor deposition only. For example when you nucleate aerosols of the gas phase they tend to be transported as the solid particles which means that the transport process is very different from the vapor diffusion process.

Vapor diffusion is essentially a random walk and so you can expect that the film will form uniformly on the surface whereas when you talk about particle transport it is very directional usually particles due to inertial forces, so they tend to separate themselves from the streamlines of fluid flow and follow inertial mechanisms for deposition. Also particles are discrete and so there is no self-assembly involved.

So when they deposit they will probably going to deposit as isolated particles, so the film will have more like dimpled look when you have aerosol deposition going on. The other affects the particles have is roughening of the CVD film. Instead of getting a smooth film if it was completely vapor deposition the aspect of a CVD film involving aerosol particle is a rougher surface.

Now why would you want a film that has those characteristics? 90 percent of the time you do not want that, you want a nice smooth and very uniform film on the surface. However there are situations where on top of an existing CVD film you want to incorporate certain features this could be for promoting certain functional characteristics, it could be for promoting a

vision, it could be for promoting certain chemical behaviour such as catalytic behaviour where these embedded particle can act as catalysts to drive a reaction.

So aerosol deposition in a CVD reactor is something that some industries want to achieve. So they try to design the reactor in such a way that aerosols forming the gas phase. Now how do you make that happen?

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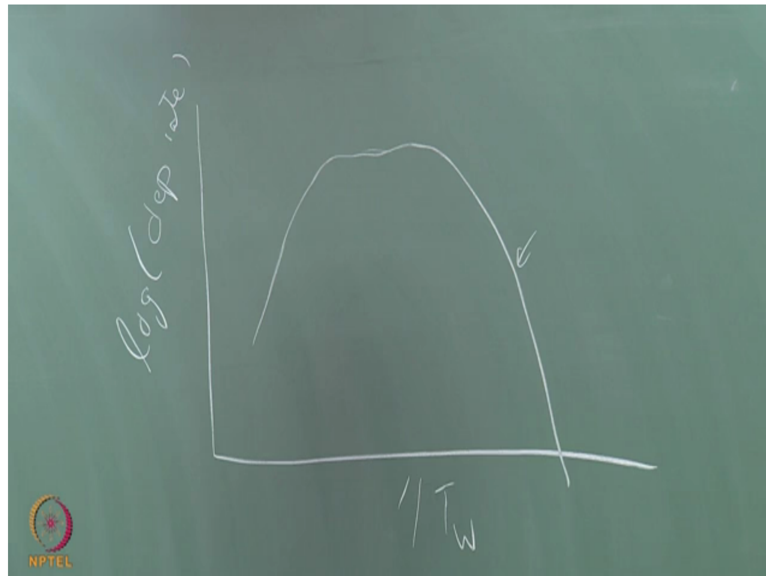

We go back to a diagram that we Drew you many many lectures ago which is looking at the effect of surface temperature on deposition rate. Remember when you plot the inverse of the substrate temperature against logarithm of deposition rate you get a behaviour that kind of looks like this, right? Where this region is that corresponds to low-temperature of the substrate where the heterogeneous reactions are actually rate controlling.

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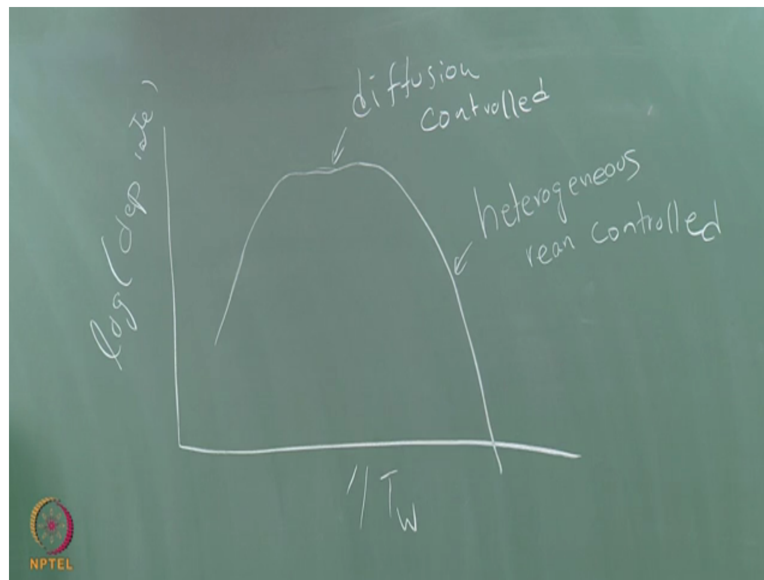

In other words the substrate temperature is so low that there are kinetic barriers to the formation of the film on the surface, right? And then you have an intermediate region which is virtually a plateau which is a diffusion controlled region where the substrate temperature has negligible influence on the deposition rate because it is not equilibrium or are kinetic that controlling the deposition rates but rather the transfer process through the boundary layer.

And then you have the 3rd region where we said that you can actually have a net reduction in the deposition rate at extremely high temperatures of the substrate because of parasite reactions in general which could be happening in the gas phase or it could be happening on the surface also.

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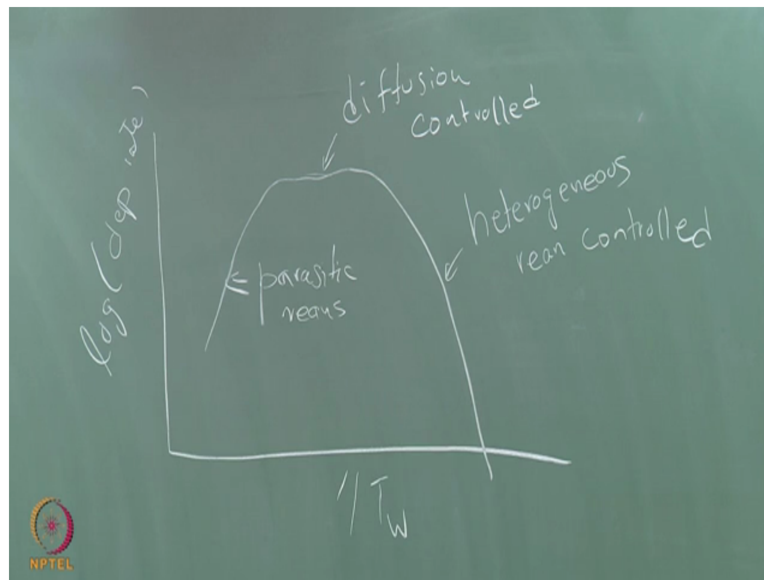

So in general parasitic reactions which essentially function as a sync for the material that suppose to deposit as a CVD film. So for example if you are trying to make SiO2 film the silicon may actually get sucked into Si3N4 something like that, so that there is a net reduction in the amount of silicon that is available for deposition as SiO2. Now if you look into this more carefully the reduction in the deposition rate can happen actually for multiple reasons.

One is competing reactions in the gas phase the other is that at extremely high temperatures it can actually start desorbing the absorbed material from the substrate. So the absorption process or the film formation process itself can become reversed and also the heterogeneous reactions that are going on can themselves become reversible as well. So extremely high surface temperatures reduce certain anomalies which can lead to a net reduction in the deposition rate.

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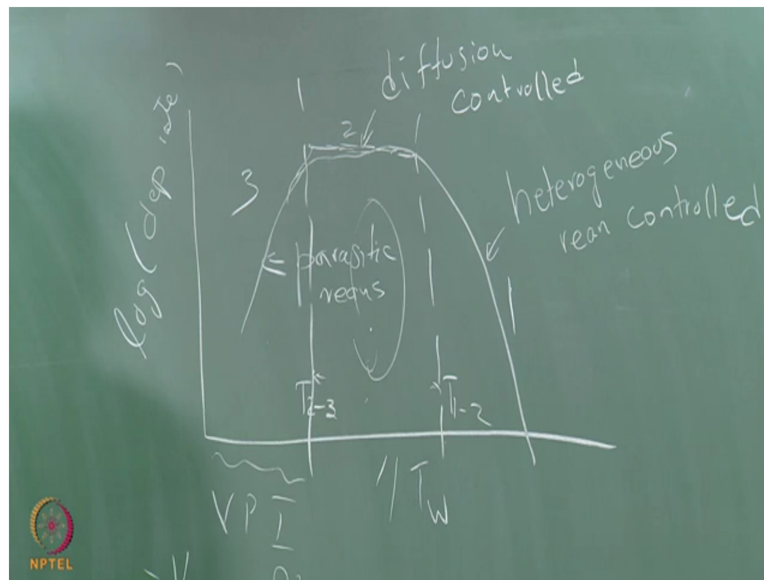

So this temperature correctly very interesting, right? The temperature at which you get a transition record this region 1, if you call this region 2 and you call this region 3, this T1 to 2 and T 2 to 3 temperatures are very interesting because this temperature represent the transition from a regime that as heterogeneous reaction control to diffusion control whereas this temperature T2 to3 represents a transition from a diffusion control regime to 1 that is controlled by primarily by the homogeneous reactions in the gas phase.

And what is happening in this region is called VPI which stands for vapor phase ignition that is essentially that is what is happening the vapor phase is getting ignited reactions are now starting to take place and because the substrate temperature is so high the adjoining gas temperatures also increase, so that even in the diffusion sublayer the temperatures become high enough that there are significant chemical reactions that will start taking place in the gas phase itself possibly leading to the formation of solid particles.

So this is also the regime that aerosol deposition or aerosol formation and deposition can be initiated. Now in a conventional CVD system where would you like to operate? Would you like to operate in this regime or in this regime or in this regime where you are trying to make a nice smooth skin CVD film? 2 but then you have to control it very carefully, right? You have to make sure that you do not get close to either boundary.

The reason is if we get close to this boundary even a slight variation in the substrate temperature can make a huge difference to the deposition rate and you do not want to be in a

situation where you have to control substrate temperature to plus or minus 1 degree or whatever or 0.1 degree. So you would prefer to be somewhere in the middle of this.

On the other hand, actually the way I plotted it is totally flat but actually the way it looks there is a slight upslope. So, you know, increase in temperature even in the diffusion control regime does result in a small increase because the diffusion coefficient varies as T to the power 1.5 or so. So there is an increase in the deposition rate, so that says you want to operate as close to this boundary as possible but we do not want to tip over into the other side.

So the ideal operating condition for a CVD reactor to maximize the CVD rate while minimising formation of nucleated aerosols the gas phase is right around this temperature T2-3, so if you could actually find a very calculated you could use it as a design parameter in your system. You could basically say that you want your substrate temperature to be held somewhere between T1 to 2 and T2 to3 preferably as close 2 T2 to 3 as possible. So that is your ultimate challenge.

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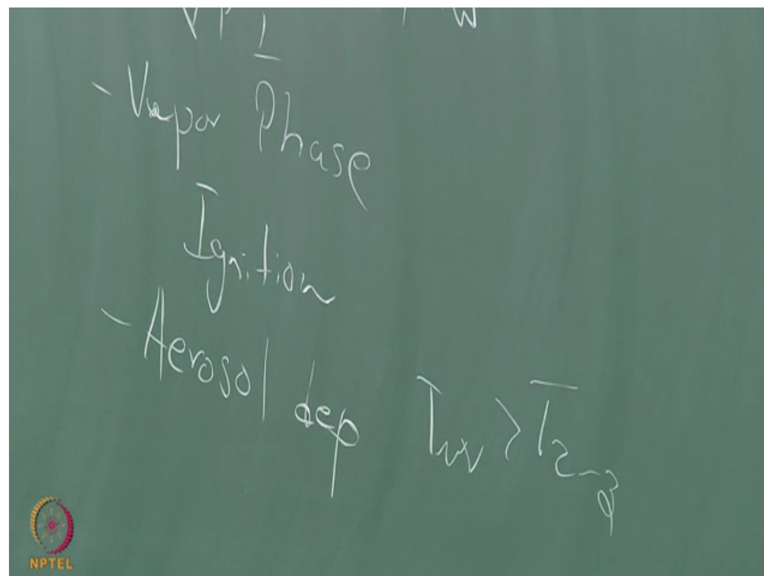

Now if you want to operate it as a CVD reactor than obviously your Tw must be kept larger than T2-3 or alternatively you have to essentially seed the reactor with nucleates. The presence of an existing heterogeneous nucleus promotes the formation of additional heterogeneous nuclear, so you can actually disturb the equilibrium in the system by injecting a small amount of seed aerosols and that will promote, you know, growth of more aerosols it's just like seeding clouds, you know, the reason that makes rain possible is because of the same principle.

So is there a simple way to calculate these transition temperatures, now one of the things that you need to look at here is, you know, if you look at the slope of this curve that actually corresponds activation energy for heterogeneous nucleation over some RTw where e heterogeneous is the activation energy associated with the heterogeneous reaction.

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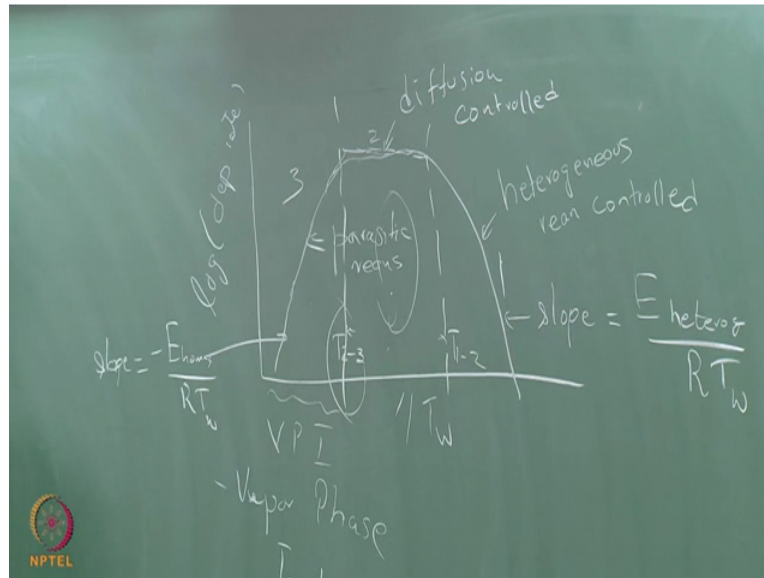

And similarly here the slope is some minus E homogeneous times RTw this is a negative activation energy because as temperature increases deposition rate drops, right? So these are obviously important parameters the activation energy by the homogeneous reaction on the activation energy for the heterogeneous reactions.

If you are to take a look at this and try to develop a model for it, and of the things that helps us in terms of doing the analysis is to use what is known as an asymptotic analysis. Typically the activation energy for homogeneous reactions is very large and that is the reason why homogeneous reactions do not occur as (()) (12:29) heterogeneous reactions. So you could assume that R times Tw over E homogeneous is much less than 1 it has actually assigned a value of Epsilon.

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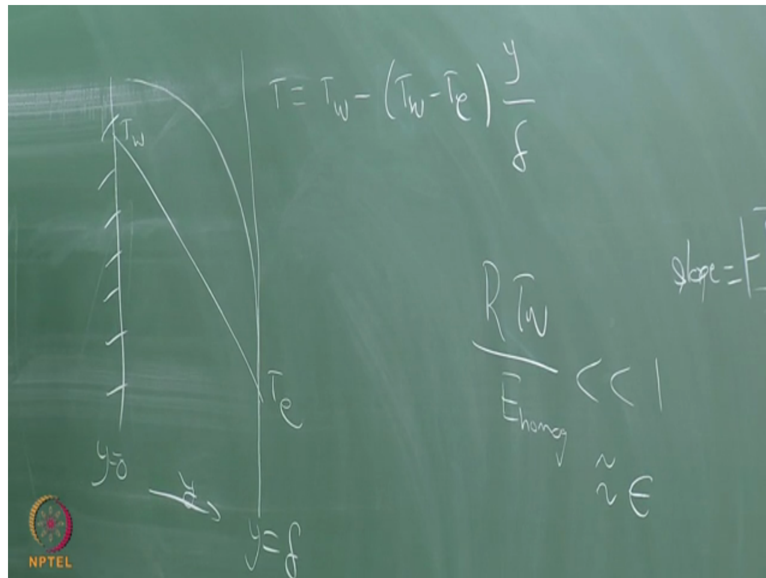

The reason that is useful for you is because now you can take look at their region near the substrate where you have the substrate here and you have a laminar sublayer, right? I mean that is a conventional model of a diffusion process, so this is y equals Delta and this is y equal to 0 where this is the y plane, so the substrate itself is at y equal to 0, the boundary layer edge is at some y equals Delta value.

And if you look at the temperature profile for example it will be fairly linear, so if this is Tw and this is the temperature at the edge of the boundary layer you can assume a relationship of the kind T equals Tw minus Tw minus Te times y by Delta. Now normally what we have been assuming in all of our analysis is that within the sublayer there are no chemical reactions happening, everything is chemically frozen and only diffusional transport is taking place and that is okay in regions 1 and 2.

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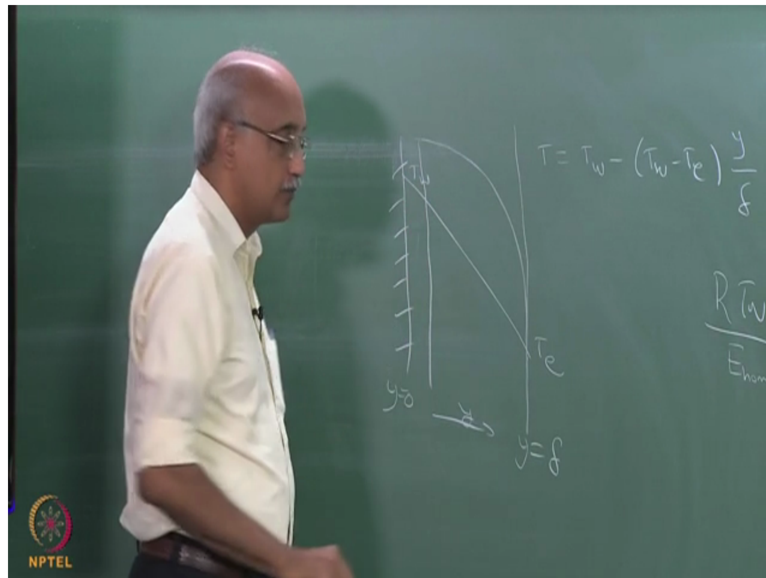

But in region 3 because the temperature of the substrate is so high that assumption breaks down. You cannot assume that the entire sublayer has 0 chemical reactions but this asymptotic analysis enables us now to define another small layer within the laminar sublayer which is called the chemical sublayer or CSL which is equal to this thickness is equal to Epsilon times Delta where Epsilon is equal to RTw by the activation energy for the homogeneous reaction.

So now the new assumption is that within this chemical sublayer reactions are taking place but outside of this chemical sublayer you can still assume that the layer is chemically frozen. So now you essentially do your transport analysis on the basis of these assumptions that there is a chemical sublayer present whose value whose thickness is equal to Epsilon times the sublayer boundary layer thickness and within that chemical sublayer homogeneous chemical reactions are happening and outside of that within the rest of the boundary layer there are no chemical reactions happening and you can assume that it is chemically frozen.

So you essentially solve the problem in 2 segments and try to match the solution at the interface between the 2 regions and you can get a closed form solution for this problem in that sense. So how actually would you write the transport equations governing this system because previously if you recall we had looked at 2 situations, one is where the heterogeneous chemical reactions are happening so quickly, that the deposition rate is purely diffusion limited?

And then we put in a correction factor to account for the fact that heterogeneous chemical reactions may be occurring at a finite rate, you know, we use the Hatta factor and developed a correction factor for the deposition rate but now we have a situation where potentially you have both homogeneous chemical reactions happening at a finite rate and heterogeneous chemical reactions happening at a finite rate and you have diffusion going on.

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So you essentially have to solve this problem by applying all 3 conditions. So if we go back to our definition of the deposition rate Jiw dot double prime equals rho times D times minus gradient in Omega i plus alpha T omega i times minus gradient in temperature over temperature this is how our diffusion equation including Fick diffusion as well as thermal diffusion or soret diffusion. So this is the Fick diffusion part or this is the thermal or soret diffusion path.

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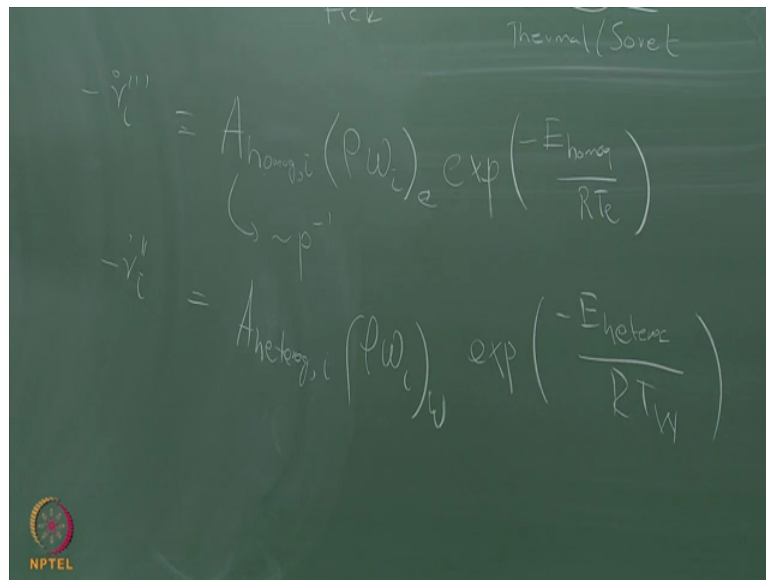

Now for the homogeneous chemical reactions you can now define a consumption reaction rate minus ri dot triple prime because it will be for unit volume and that will have an Arrhenius type of relationship, so you can write at A homogeneous i times rho omega i E times exponential of minus E homogeneous over RTe where this is a pre-exponential factor which will depend on pressure will roughly go as P to the power minus 1 and then you have omega a is the mass fraction of the depositing species i, so you know it is a typical Arrhenius type of expression, this represents the rate of the homogeneous chemical reactions.

Similarly on the surface you will have heterogeneous reactions taking place which you can write as minus Ri dot double prime because now it is going to be per unit area and this will have units of A is can be written as A heterogeneous i times rho omega i at w times exponential of minus E heterogeneous over RTw. So these are the reaction rates governing the homogeneous chemical reactions and heterogeneous chemical reactions.

So how do you close this set of equations, you have to write mass conservation equations, right? So in the gas phase if you remember assuming steady-state operation the 2 terms will be that when you have and again these are equations only within the laminar sublayer, so convection is also (()) (19:50). So essentially the left hand side of the equation both terms are 0, so the diffusion term must exactly balance the reaction term, right?

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So how do you write that? Let us also assume that instead of using gradients let us do this as one-dimensional analysis. So you can write the diffusion equation as rho times Di times you are not taking the divergence basically formula we are using is divergence of di, w dot double prime must equal ri dot triple prime, right? So when you take the divergence of that, that becomes rho Di times d squared omega i over dy square plus Alpha T times d by dy of omega i times dT by dy equals the expression that we have written for ri dot triple prime.

So this is the conservation equation for species i taking into account diffusion and homogeneous chemical reaction, how do you bring in the heterogeneous chemical reaction here? You include it in the same conservation balance or do you operate separately, how do you incorporate your heterogeneous condition? You do it as a boundary condition, so there are 2 boundary conditions for this problem because it is a second-order differential equation.

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So the boundary conditions are, the first one is omega i equals omega ie at y equals Delta. In other words the mass fraction of the right species will be equal to omega ie when the y equals the thickness of the boundary layer. The second boundary condition is where you bring in your heterogeneous reaction, how do you do that? Now when you look at the surface the flux of material to the surface is consumed by the reaction taking place at the surface.

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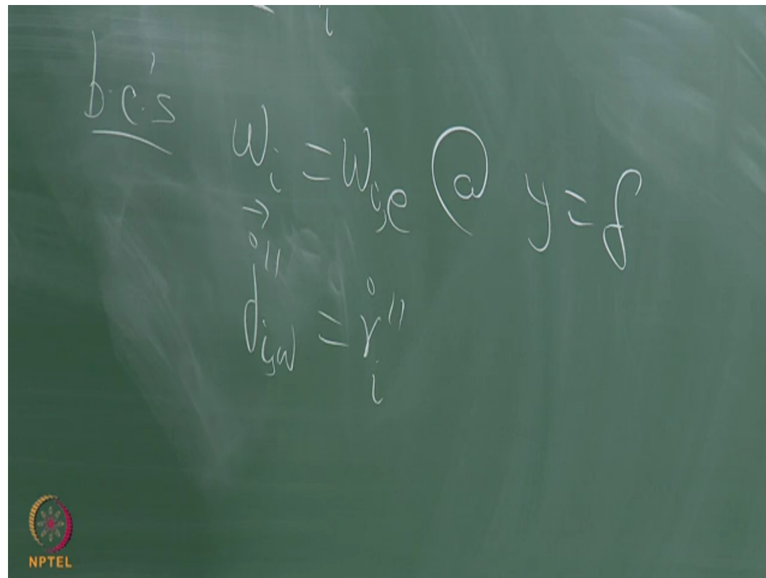

So the second condition which is at the substrate is simply jiw dot double prime should be equal to ri dot double prime, okay. So the fluxes have to balance each other and this is the same formula if you remember that we used when we were trying to calculate the correction factor for the effect of heterogeneous kinetic's on deposition flux. So when you write that, when you expand it and write it.

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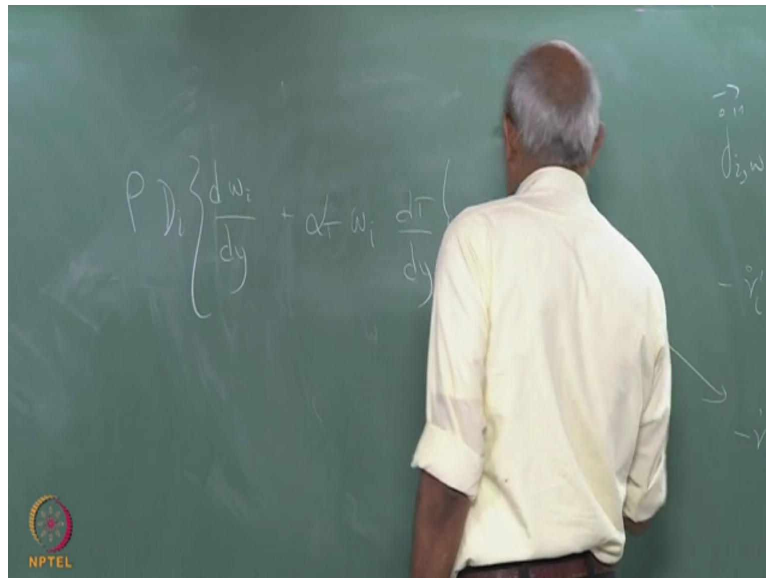

It will look like rho times Di times simply d omega i over dy plus Alpha T omega i dT by dy equals this expression for ri dot double prime, okay. So in the same set of equations you have included your homogeneous kinetic's as part of the mass conservation balance equation and you have incorporated your heterogeneous candidates is a boundary condition based on what happens at the substrate interface, okay. So then these are your new governing equations for deposition which account for all 3 homogeneous reactions, diffusion and heterogeneous reactions.

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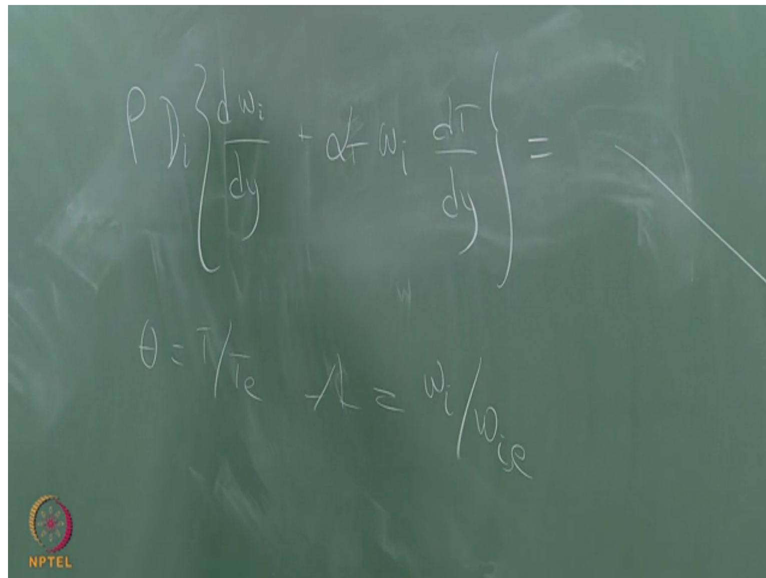

Now you can do non-dimensionally isolation you can write theta equals T by Te, you can write some capital omega equals omega i over omega ie and you can reformulate the equations. Obviously the Damkohler numbers as you can expect will become important, just like when we were dealing with the homogeneous reactions and heterogeneous reactions as correction factors if you remember we use the Damkohler numbers.

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Similarly in this case also you have to define a Damkohler number homogeneous which will be equal to A homogeneous this is all for species i times Delta squared over D times rho omega e to the power minus m where m is the order, sorry I should have included this should be rho omega i to the power m where m is the order of the reaction, the equation I wrote is only true for the first reaction.

For a general reaction you will have to the power m here and similarly you will have power n for the heterogeneous reaction. So your definition of the Damkohler number will then include, it is actually not minus and it is m minus 1 and similarly the Damkohler number heterogeneous will be A heterogeneous times Delta over D the difference of course here is its Delta square and here it is it is Delta because the units are different for A homogeneous and A heterogeneous, these 2 have different units, right?

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Because one is volumetric and other is surface coefficient times rho times omega e to the power n minus 1, so these are the corresponding Damkohler numbers and as you can expect the Damkohler numbers are related to those temperatures that we talk about T, T1-2 and T2-3 by evaluating the Damkohler numbers you can estimate those temperatures as well. Okay, so what you can do now, you know, now that you have defined certain dimensionless coefficients you can actually rewrite those equations also in nondimensional terms in terms of the theta and omega.

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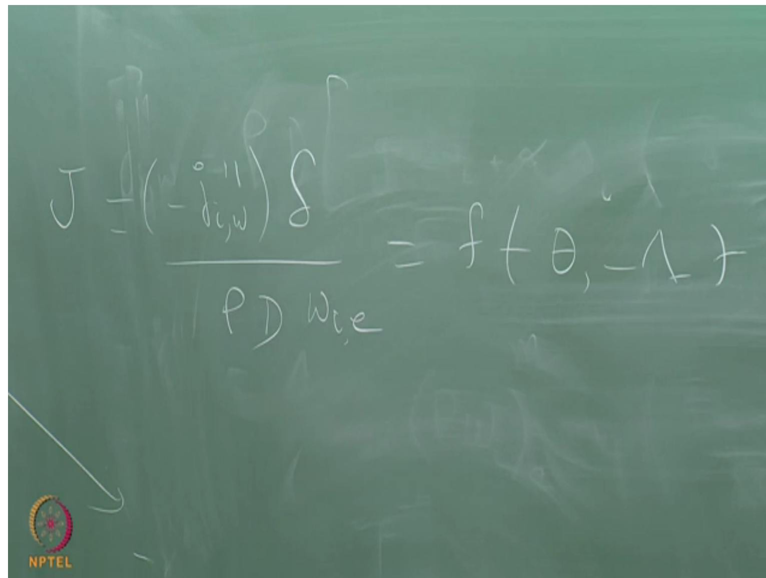

You can also nondimensionalized the deposition rate as J equals minus jiw dot double prime times Delta over rho times D times Omega ie and you can relate this J now as a function of Theta and omega, okay. So by the way again I will send you some papers that talk about this system, so you can get the actual rate expressions and so on but more important to understand the concept, you know.

Essentially what we are seeing here is that when you have all 3 phenomena operating simultaneously you have to make sure that you incorporate all of them into your conservation equations, so that we can get a solution that accounts for both homogeneous as well as heterogeneous kinetic's. So when you go through these then you actually obtain the final expression for J and then you obtain conditions at which diffusion control happens.

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So how do you estimate the temperature at which the transition from region 1 to region to happens? Is there a simple way where you can say that this is the temperature at which your transition from being heterogeneous kinetics control to being diffusion control, what would that depend on? It should be based upon the Damkohler number, right? If the Damkohler number exceeds or falls below a certain value but which Damkohler number?

When you look at the transition from 1 to 2 which one do you think is the crucial Damkohler number? Heterogeneous, right? And similarly the transition from 2 to 3 will not be influenced by your heterogeneous Damkohler number but by your homogeneous Damkohler number.

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So you can obtain an expression for T1-2 which is a function essentially of the Damkohler number heterogeneous and similarly you can obtain an expression for T2-3 which is a function of Damkohler number homogeneous and at each of these regions you can obtain the actual deposition rates using these non-dimensional relationships that we have derived.

Now it turns out that if you want to drive the process to conditions where homogeneous nucleation's or aerosol formation becomes possible then the ratio of the Damkohler numbers does become important. In fact the temperature or the conditions that favor the formation of nuclei in the gas phase have to do with how rapidly are the homogeneous reactions proceeding versus how rapidly are the surface reactions proceeding.

A simple way to look at it is if your homogeneous rate kinetics are very rapid compared to are comparable to the heterogeneous rate kinetic then you have to take both into account essentially in the extreme case there is no difference between the substrate and the gas phase in terms of promoting chemical reactions. Now that is unlikely to happen the energy barrier or the activation energy in the gas phase is always going to be larger than the activation energy on the substrate.

Unless like I said you deliberately inject solid surface in which case you are essentially introducing small form factor substrates into your gas phase. Your kind of making the gas behave like a deposition substrate but unless you do that, there is always going to be a higher barrier for gas phase nucleation compared to heterogeneous nucleation and so you can

establish a condition which will be based upon ratio of the Damkohler numbers for heterogeneous and homogeneous nucleation as a ratio.

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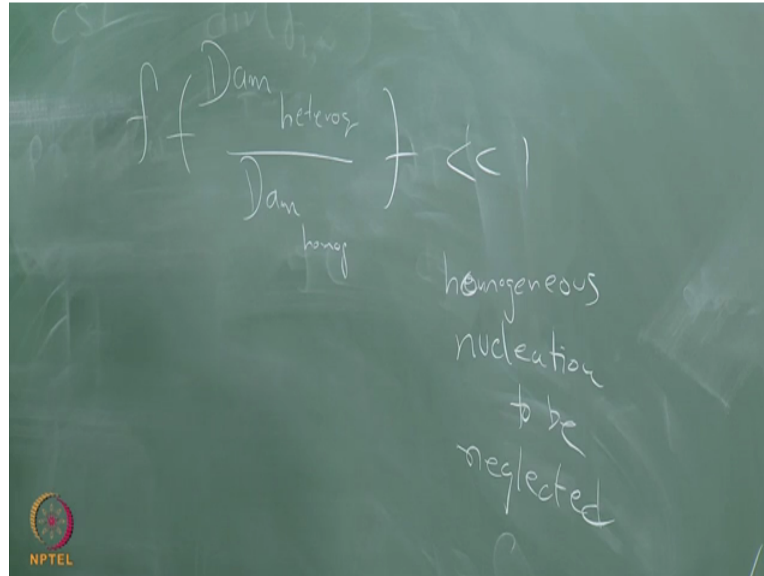

So again you have a function of the ratio between the Damkohler number heterogeneous to the Damkohler number homogeneous which has to be much much smaller than 1 in order for heterogeneous or homogeneous nucleation to be neglected. So here again from a design viewpoint have a lot of parameters you can control, you can by choosing appropriate surface temperatures can choose which regime of deposition you want to operate in, region 1, region to region 3.

And also when you are in region 3 by suitably designing your system to have certain desired ratios of the Damkohler number for the homogeneous reaction versus the Damkohler number for the heterogeneous reaction you can further control what percentage of your feed material you want to go into homogeneous nucleation versus heterogeneous nucleation. Again simple way to think about it is the higher the temperature the greater will be the propensity for more material to be consumed in a gas phase itself which is again why you should try to keep the temperature as low as possible to still achieve the deposition rates you are looking for.

It is good from the viewpoint of preventing homogeneous nucleation but it is also good from the viewpoint of minimising your energy expenditure, right? The less you heat the substrate the less energy input you are providing to the system but it is not something that you can do empirically, the message that I am trying to drive home is that there are ways in which you

can systematically analyze a CVD system and actually precisely identify these temperatures that you need to set your substrate at.

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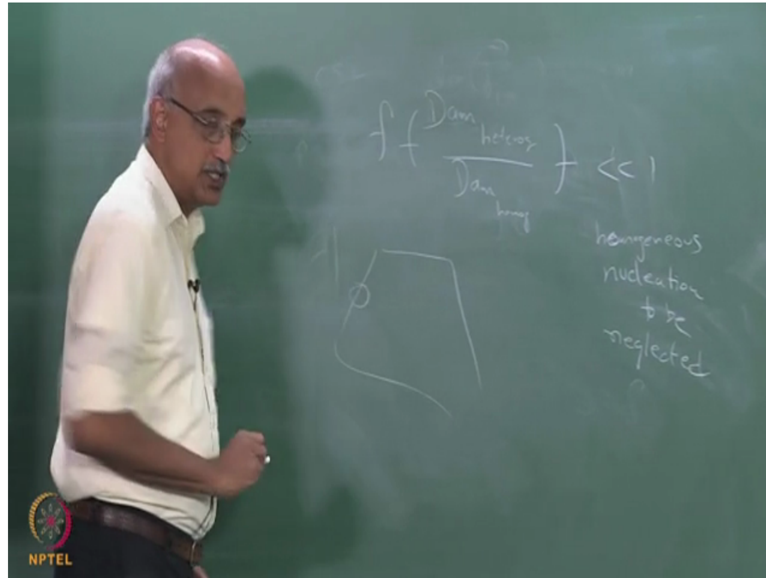

Now supposing you have decided you want an aerosol CVD reactor and you have chosen the condition so that, you know, you are in this region somewhere and you have chosen your Damkohler numbers, so that yes there is a high probability of homogeneous nucleation happening and producing these fine particles then there are 2 challenges, one is how do you manage temperature gradient?

Because if you remember thermophoresis is the phenomena that can drive particle transport quite effectively but because particles are always heavier than the carrier gas when you apply a thermal gradient the Phoretic motion of particles is always from hot to cold and in a CVD reactor the substrate is always at a higher temperature compared to the surroundings even in a hot wall reactor of all temperature maybe close to the substrate temperature but it is not going to be exceeding the substrate temperature.

But what that implies is, thermophoresis is always acting against you if you are trying to deposit aerosols because aerosols will be rejected by the hot substrate and driven towards the cold walls of the chamber and that is one reason why aerosol CVD reactors are very difficult to design and operate because thermophoresis which can actually be (()) (35:31) of magnitude stronger transport mechanism compared to the Fick diffusion for Brownian diffusion in the case of particles.

It can really result in a net depletion away from the substrate rather than deposition on the substrate. So how do you handle that? The first thing is an aerosol CVD reactor is always designed in a hot wall condition, you just cannot have a cold wall aerosol CVD reactor because the amount of particles depositing on the cold walls will just be enormous. Virtually 100 percent of your aerosol production will move towards the cold wall and deposit there.

So you do not want that, so you want to keep the substrate it is almost the exact opposite of normal CVD reactor in a conventional CVD reactor you want to keep the walls as cold as possible to prevent formation of deposit by vapor deposition but in the case of an aerosol CVD reactor because of primary mechanism of deposition there is actually 2 of them, one is thermophoresis and in order to defeat thermophoresis we have to keep the reactor wall temperature high to make sure that the driving force for thermophoresis is kept as low as possible.

The driving force is the temperature gradient, so the smaller the temperature gradient between the substrate and the reactor walls less the propensity for Russell particles to be directed towards a wall for deposition, so that is the first thing but the second mechanism that leads to aerosol deposition is inertial forces inertial impaction.

Well again depending on if you are trying to promote aerosol deposition you want to provide essentially line of sight deposition. In other words inertial deposition happens most effectively when the source of the particles is located directly upstream of the substrate.

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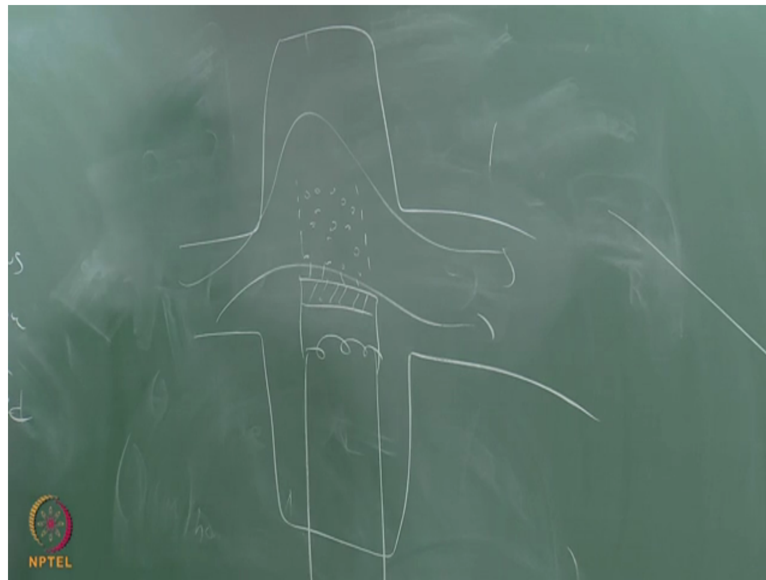

So for example in a CVD reactor, let's say you are using a conventional flat plate type of CVD reactor and you have your wafer here and the gas flow, you know, is like this. If you want to use this as an aerosol CVD reactor, the aerosols should as far as possible be formed in a region that is directly above the substrate, so that they will naturally just by gravitational sedimentation other body force effects by inertial effects drop-down on the substrate and deposit there.

Now that is difficult to achieve obviously in a parallel plate CVD reactor because how do you control the formation of the nuclei to only the region above the plate above the substrate. So in fact more than 99 percent of aerosol CVD reactors use the stagnation flow configuration.

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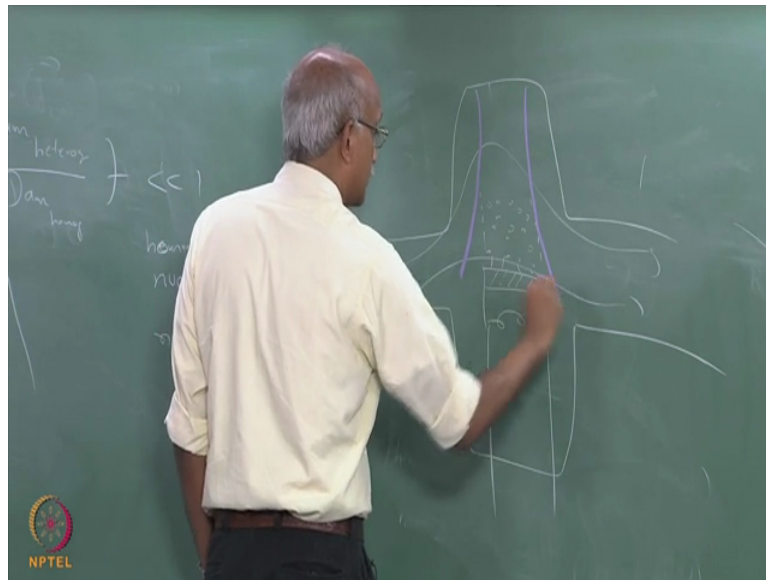

So instead of this type of driving mechanism you essentially use a flow field that looks like this then the nucleated aerosols are naturally confined to this flow region, right? So they are essentially driven to the substrate and made to deposit there, it is very difficult for them to escape along with the gas phase because they do not follow the streamlines they have a sufficiently large Stokes number that they essentially act as a second phase which is not coupled to the carrier gas phase.

So this inertial separation enables them to directly impinge on the substrate and form a layer there. Now that is one aspect of it which is directing the aerosols to the substrate. Now when you talk about sticking coefficients, what is that probability that an arriving particle say on the surface rather than re-bonding. Here again the considerations are very different for an aerosol CVD reactor compared to a vapor CVD reactor.

In a conventional CVD reactor you can assume that when a vapor molecules comes and strikes a surface as long as there is a site available for absorption the vapor molecules will get absorbed, right? But in the case of small aerosols striking the surface that is not necessarily true even if there is a vacant site and even if there is a chemical affinity between the particle and the substrate depending on the angle of incidence and depending on the velocity of incidence the aerosol can actually (()) (40:32).

And so the sticking coefficient or the sticking probability or the capture efficiency for a fine aerosol is typically much lower than for vapours. Now as the size becomes larger and larger again the capture efficiency starts to increase but in the middle region when the agglomerated

nuclei size or particle size is of the order of few microns the capture efficiency is at its lowest which means that your material investment has to be larger.

If you are using vapor deposition you can assume, you know, as we were discussing the other day in a filament CVD reactor you can achieve up to 40 to 50 percent capture efficiency or utilisation efficiency. In an aerosol CVD reactor unless you have very tight control over the size distribution of the nucleated aerosols the capture efficiency can be as low as 10 percent, so 90 percent of the material may just bounce off.

So here again the stagnation flow configuration helps because when the flow is this way even if an aerosol hits the substrate and rebounds it can again be made directed back to the substrate whereas in this type of a flow situation once it rebounds it is gone we have lost the material.

So simply by changing the configuration of the CVD reactor to a stagnation point reactor rather than horizontal flow CVD reactor you can improve the capture efficiency by 2 or 3 times because even if the first capture efficiency is only 10 percent the same material is again directed when it is done for 3 or 4 times the overall capture efficiency can increase up to 30 to 40 percent even in an aerosol CVD reactor but it also extends the time.

So in an aerosol CVD reactor the time required for building up a CVD film can be significantly larger than in a conventional CVD reactor because of this probability of multiple rebounds, multiple capture and so on. And it also means that you really cannot promise a certain level of uniformity of the film is going to be based upon multiple incidences and multiple rebound events.

So it is hard to predict unless again you have such a tight control over the approaching trajectory of the particles, if you can make sure that they always approach at the same angle and the same velocity every time it can become a little more predictable but most of the time an aerosol CVD reactor, you know, in a conventional CVD reactor it is possible to achieve plus or minus 1 percent uniformity and deposit thickness for example. In an aerosol CVD reactor if we get 10 percent accuracy or repeatability you should be happy.

On the other hand like I said aerosol CVD films have certain very distinct characteristics primarily the discrete particle presence which can increase the total amount of surface area that is available. So particularly when you are using when you are trying to prepare catalyst

materials, aerosol CVD reactors are quite widely employing to achieve certain distinct features of such materials.

Okay, so let us stop at that point, in the next lecture we will talk about another class of CVD reactors which are primarily used to make coatings on surfaces. These reactors have certain very characteristic requirements that we can discuss in a little more detail because CVD is you know of coatings probably accounts for close to 30 to 35 percent of all CVD usage in industry. So it is an important process for us to try and understand.

Okay, any questions on what we have talked about today?

“Professor -Student conversation starts”

Student: Another CVD examples…

Professor: Silicon oxide for example when you are trying to make silicon oxide coatings on surfaces for many applications particularly where there is erosion possibilities CVD film tends to get damage more easily than aerosol CVD film you can get much better hardness for example of the film when you use an aerosol reactor.

Also as I said when you are trying to make catalyst particles that are a primary use and again silicon oxide is one, titanium oxide is another when you are trying to make a thin film bed with embedded catalyst particles that is always done in an aerosol CVD reactor. Okay, thank you, see you in the next class.