Technology of Surface Coating
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Lecture-04
CVD System

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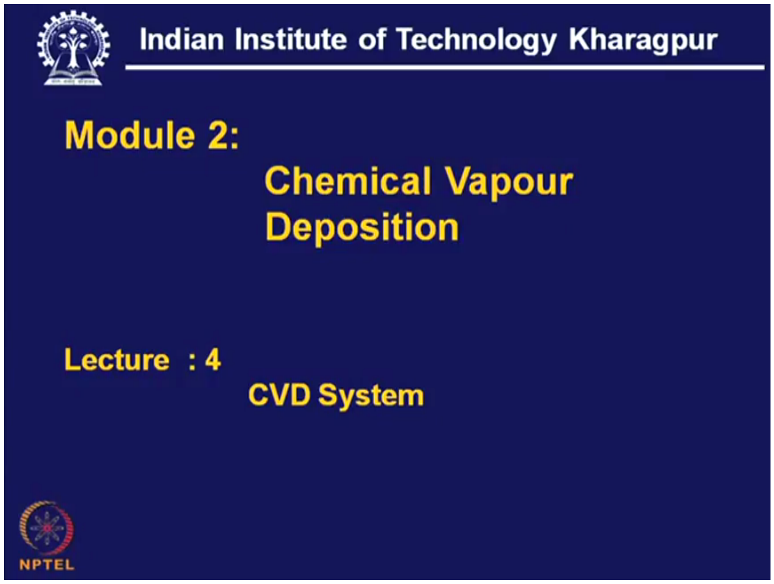

Chemical vapor deposition and CVD system we already know that this chemical vapor deposition technology can be conveniently used for deposition of metals, hard coating, soft coating and many more materials. However to put this principle of CVD in practice, we must have a whole CVD system and it is just not one component or one element of this system. It is the total modules, all the modules should be taken in consideration so that we can understand the basic technology of this chemical vapor deposition.

That means it is basically a chemical reaction which takes place in a vapor state with the result that we get one surface, one product in the form of solid and more specifically in the form of a film and the another one, that is the gaseous product. Now to make it happen, we need various modules in the CVD system and when we put these modules in harmony, then we get the total CVD system effective one.

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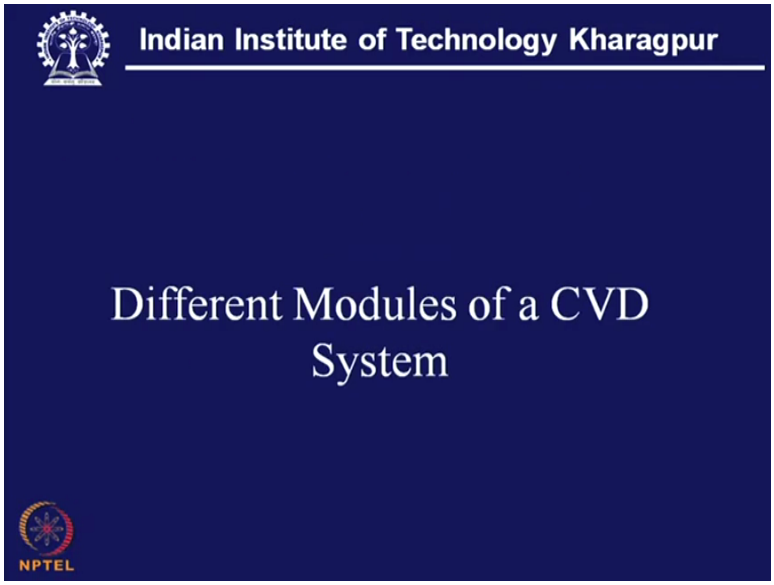

Now let us look into what are those different modules of this CVD system. Now here what we have, we have at least three modules.

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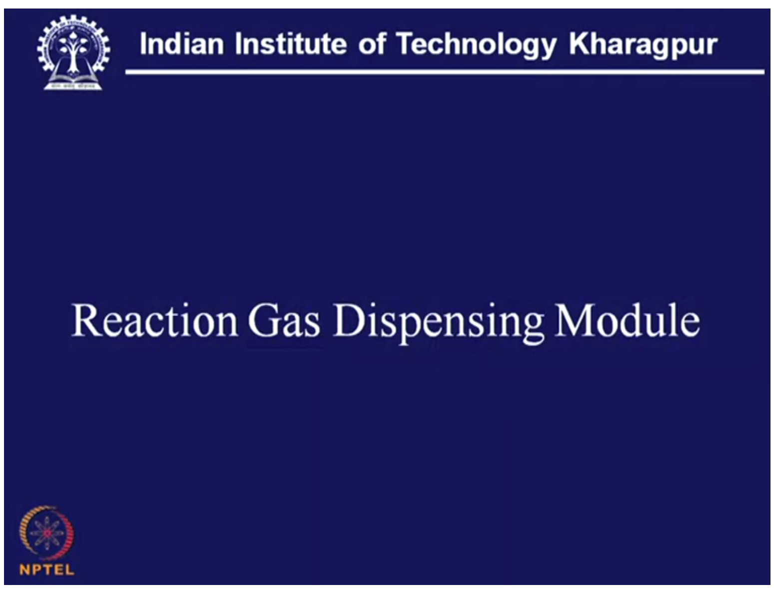

One we, what we can call here reaction gas dispensing module, that means it is actually the supply of the all the reaction gases which will participate in this reaction including that which is going to be a metal donor. So that is very important, others can be available in the form of gas but particularly this part which is mostly a metal donor, which is available in most of the cases in the form of one halide, that should be also available. And that should be available so that we can very easily use it. That means the ease of use, that is one important point here one has to consider. So this is reaction gas dispensing module.

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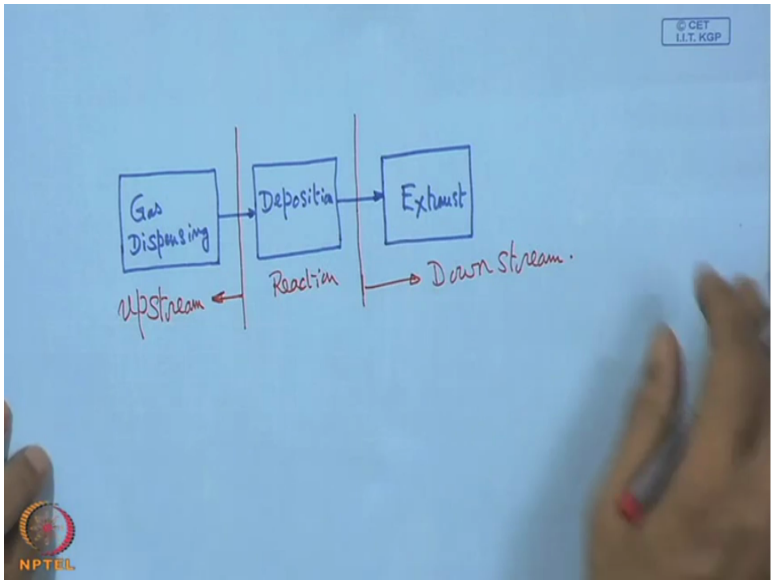

So here what we can see that we can have, say for example three modules are there. We can put this way. This is all gas dispensing, either gas or vapor, gas dispensing. So in simple language, it is gas supply. And then the whole thing goes here, what we call that deposition module, so this is actually deposition module. And then the last one, that is actually the exhaust. So we have all together these three modules: gas dispensing, deposition and exhaust.

So what we can see here, the reaction will take place here. So this side we call, this is actually the upstream and this side, we call it downstream. So gas supply, this reactor and then the exhaust. Now in this what we find, we can go to this reaction gas dispensing module. What are the things we have here?

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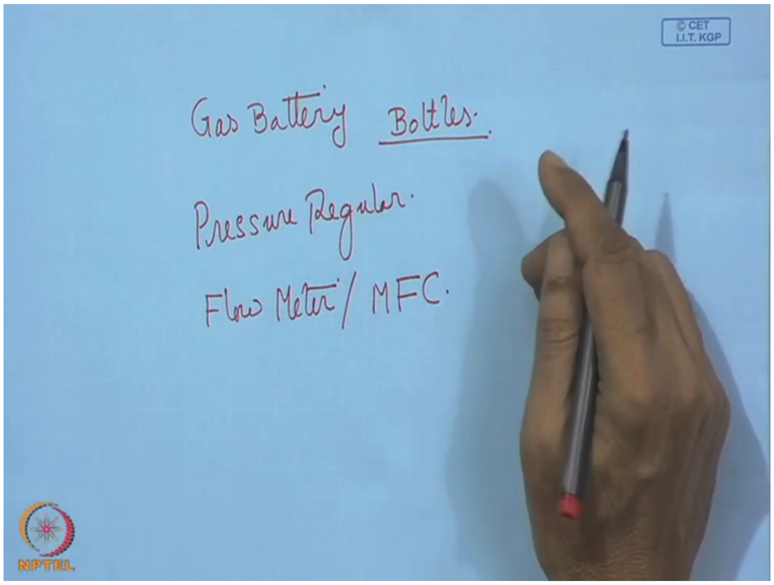

We have all the gas battery, that means here we have the gas battery, that means all the gas bottles, that means it is the gas bottles. Then we have the pressure regulator, this is also very important, followed by what we have, we have here either a flow meter or one MFC, Mass Flow Controller. Now with this, we can create this gas dispensing module.

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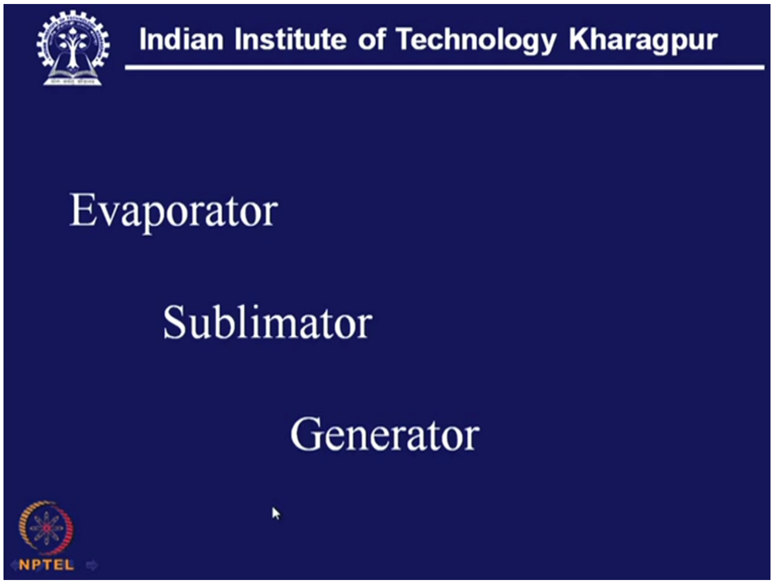

And here what we have in addition to that, we have to couple with this gas supply either this what we call evaporator or sublimator or generator. Either of these, any one of these will be useful in that.

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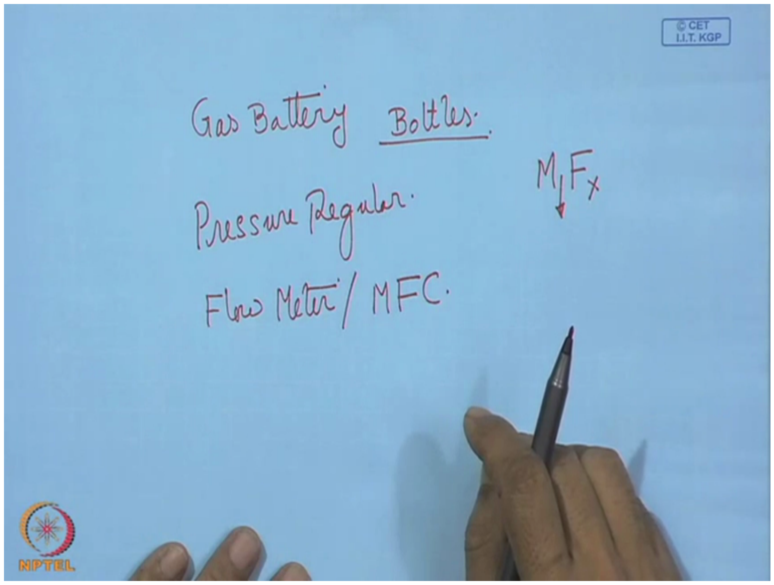

Because if it is a metal halide, if it comes in this form, then this metal has to be, metal has to participate in this reaction. So this is available either in the form of a solid, then it has to undergo sublimation, that means solid to evaporation. So we need the vapor of this halide. Or it can be commercially available in the form of a liquid, so that can be straightway useful. We can use it but in certain cases, this is just not available commercially or there are certain difficulties in use of those as a direct product, direct source.

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In that case, what we have to do, we have to use what we have mentioned here, this generator. That means in this generator, we have to create the vapor of that metal halide.

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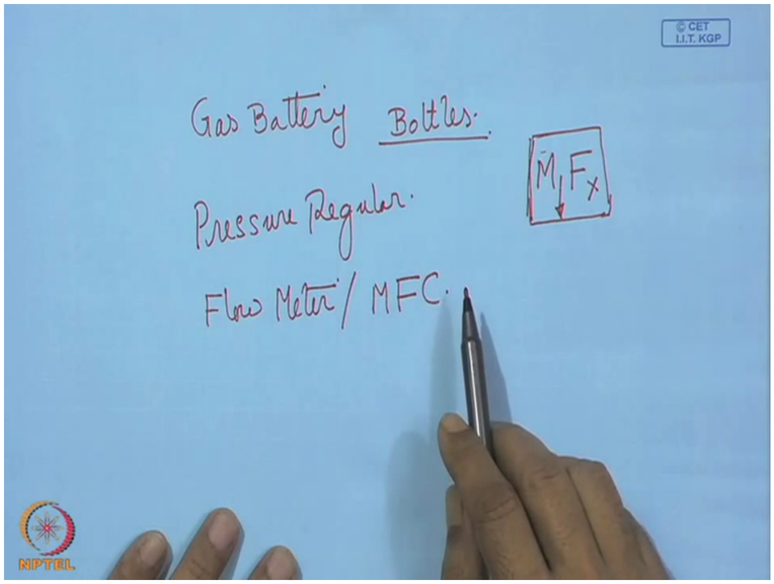

So these are the three, I mean subsystem. These are the submodule of this gas dispensing module that will be coupled with this one to make a complete gas dispensing system in the upstream side of the CVD system.

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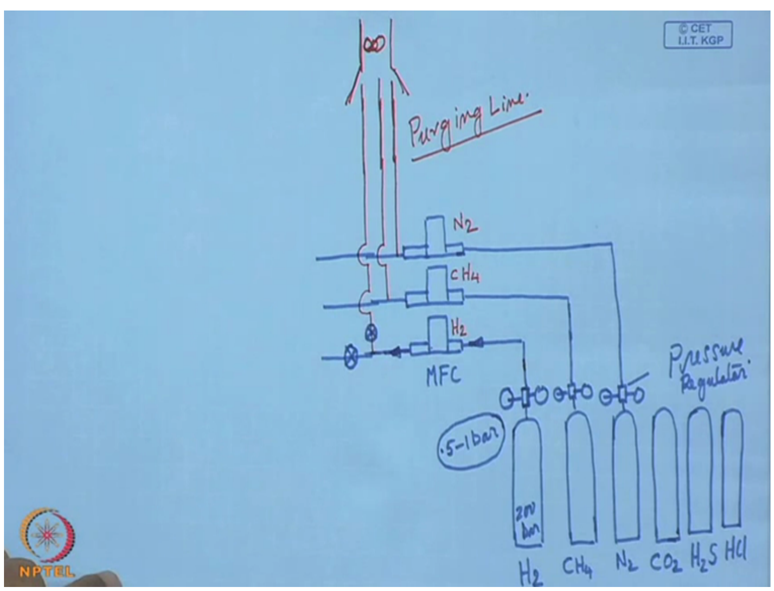

Now let us have a quick illustration how the whole thing will be built. So here say for example, we have say four or five bottles. So these are the bottles, maybe this is hydrogen, this is CH4, we can have nitrogen, we can also have CO2. We can have also say for example H2S, all are useful to conduct a particular CVD. We can still have another bottle containing the vapor of HCL. All are essential, needful for successful operation of one particular CVD.

Now from this what we like to have, from this actually here we must have one pressure regulator. So this is the pressure regulator and this one will be common to all of those. So these are the pressure regulator. So this way, we can have the regulator for each cylinder. Now the purpose of this pressure regulator would be to set a particular working pressure. The bottle pressure is few 100 atmosphere, say 200 atmosphere, 200 bar but the output here will be say maybe 0.5 to 1 bar.

So the working pressure just after this reduction, that will be say 0.5 to 1 bar, we can set to a value. But what is very important here that this pressure will remain all along constant though the bottle pressure will keep on falling with passage of time because the gas is going to be consumed. So naturally this bottle pressure will fall but this reduced output pressure will remain constant. So that will be the role of the pressure regulator and no intervention is necessary, so it will keep this reduced pressure constant throughout this operation.

Then comes important thing, either we can have one MFC or one pressure regulator or a flow meter. So here we can show one MFC symbolically. So this is one MFC to which it will be connected and it will have the output. This is the inlet and that is the outlet from this MFC. So similar MFC we can have for methane also if we are interested in deposition of titanium carbide. Definitely, we need to have such thing, so that will be also connected.

If we are further interested in titanium nitride deposition, then too we to have another MFC. So this way, we can make this connection. So this is how the system will be built. So here we have the outlet for each one. However what is necessary? So we have hydrogen, methane and nitrogen and these are the bottle, and here we have the MFC. But from this we have branching, so from this we have a branching, it is something like this.

We have another branching, it is connected to this one. And then we have further to this, we can extend this line. And further to this, we have another one. So this is actually connected to this hydrogen. So this is hydrogen, this is CH4 and that is nitrogen. So here also we have all these thing which are connected here. So we have three parallel tubing which goes to ventilation. And this is actually a ventilation which goes here. So here we must have a fan and it will keep on clearing this residual gas.

Now why this bypass is necessary? Actually this is called purging and here what we have, in each case say for example we have to have one this valve. So this is just like a stop valve on this side. So we have flow either in the, one flow towards the reactor, another flow directly to this ventilation. So this way we have two stop valves, that is the isolation valve for each line which we can put here. Now this ventilation is necessary.

This is exactly what we call, these three it is actually the purging line. So this line escape for purging, so we must, at least this is essential for hydrogen to improve its dew point. And this dew point is a representation of the moisture content. So a dew point greater than minus-51 or even greater, that is actually the standard routine practice. It is the routine quality control check that this hydrogen should be sufficiently dry and there should not be any further moisture content. So it will be just purged before it is fed to this side in the downstream side. So this way this purging has to be carried out. And there is one dew point meter which will record this value of what is the dew point at that particular condition.

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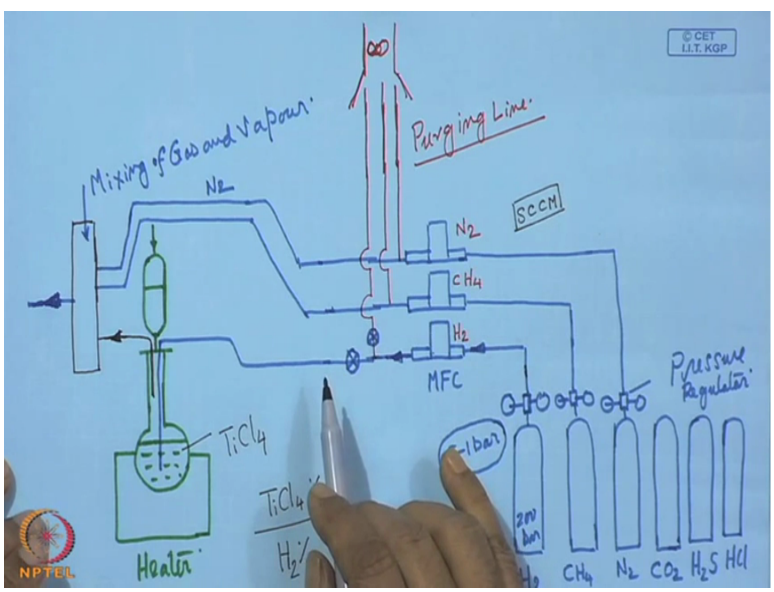

And now here what we have to connect, this can be, say in this case we can put one evaporator and this is just like a bottle. This is just like a bottle, so here it is blocked and what we have, we can have a supply of this liquid from this line. So this will be the supply line and this is actually the liquid and whose temperature is controlled by this heater.

So this is actually the heater and what we have to have here, it is just bubbling of hydrogen through this line. So it will be straightforward it is going to be bubbling of hydrogen. So this is, this will be dipped into this one. And then we can have, this is the line. And what we have from this, this will be the vapor which will be carried. So this is the exit from this evaporator, so that is a flux containing say for example titanium tetrachloride. And this is hydrogen line, so it is actually, in principal this is actually saturation of hydrogen with $TiCl4$ vapor.

So what is important? This evaporation rate of $TiCl4$ or anything, that will depends upon the temperature of this liquid, then the pressure in question and also flow rate of this hydrogen. So on those thing will depend how much $TiCl4$ is actually passing through this line. So it is actually said that it is given by $TiCl4$, percentage of $TiCl4$ in comparison to total or percentage of $H2$. So this way, we can find out how much amount is being fed.

And here this mass flow controller for methane, nitrogen, all are actually rated by a term what is called SCCM and that is actually standard cubic centimeter per minute. And this is used as a parameter, process variable how much is being fed into the reactor. So from this what we have, here we have one mixture, this is for mixing. So this way it can go like this. This is one line, this is for nitrogen. We can have another line for methane. So this is actually the mixing of gas and vapor. So this tetrachloride is saturated.

Now from this side, now here we have this is the outlet, that is going to be connected to this reactor. So this whatever we have shown here, that is the detail of this upstream side and this module is called the gas dispensing module. Here we have gas battery, pressure regulator, mass flow controller, purging line. So for all these, we must have this kind of isolation valve, so the gas can be either diverted to this ventilation or it can be diverted further down to this, towards the reactor. So these are the two possibilities.

This can be either manually operated or it can be automated or nowadays all the things are programmed and microprocessor controlled. So in a sequence, all this operation will be conducted and all those things are well standardized for practicing in the actual field.

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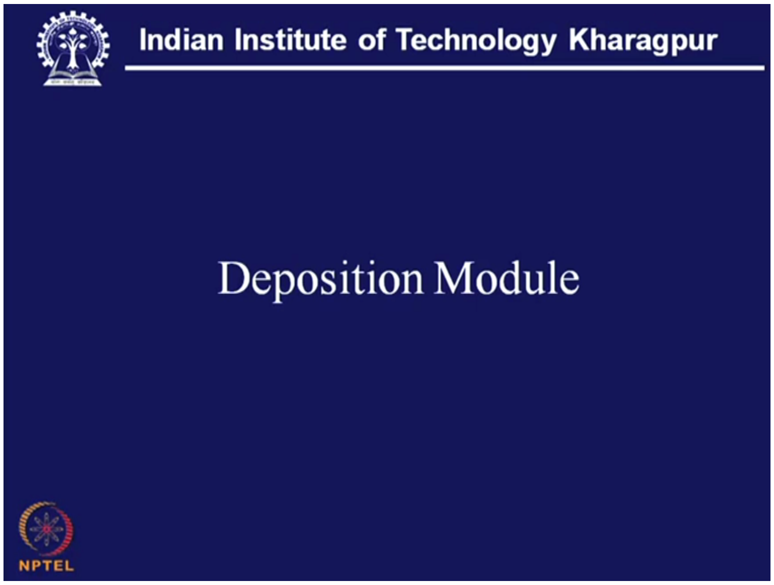

So from there what we see now comes deposition module.

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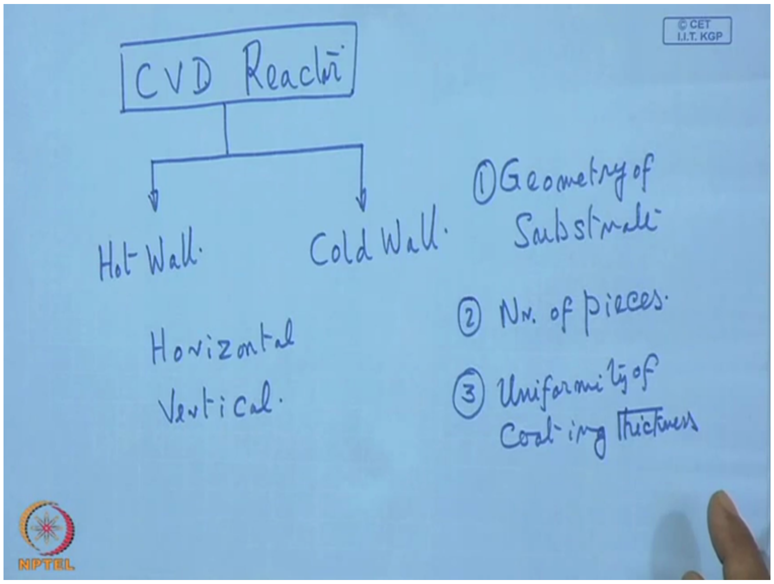

So deposition module is actually the reactor, CVD reactor now CVD reactor, we can have CVD reactor. One thing we can have say hot wall reactor, hot wall and then we have also cold wall. It can be horizontal, it can be also vertical. Now the question is, which one is the best? Now to address this question, what we have to tell here that the geometry of the substrate, it is actually geometry of substrate, number one.

Then number of pieces, that means the quantity. Number three, what is very important here, uniformity of the coating thickness. Uniformity of coating thickness, that is also of immediate attention. That means it is just not a single piece, it can be several thousand pieces are being coated simultaneously and all are arranged either along the length of the reactor, placed horizontally or down the length, height of this reactor. So this axis of the reactor, that can be also placed vertically. So their position will be different considering the entry point of this.

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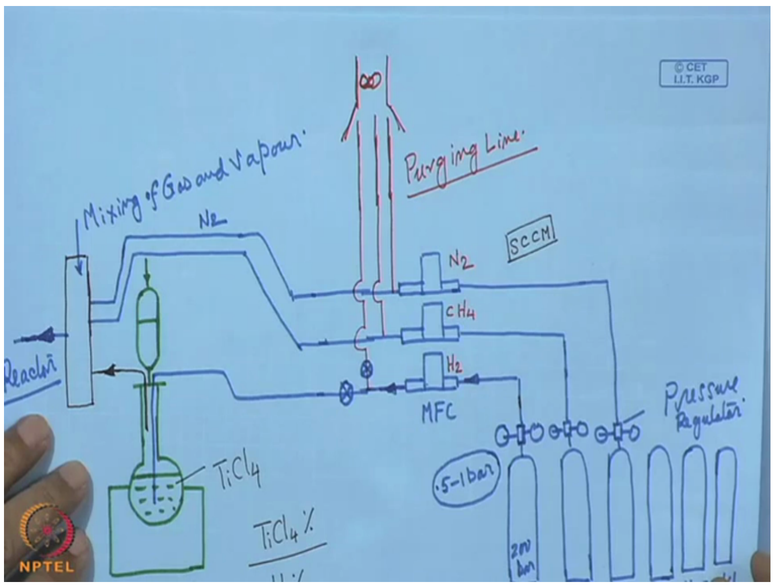

That means this, if we refer to that diagram, this entry point that will be very important where we are going to connect this line. So that is going to reactor, so how we are going to connect, whether it will be from the bottom and the gas will move up or whether it will be from the top to bottom, it will move vertically downward so all these things are to be considered while addressing this particular issue.

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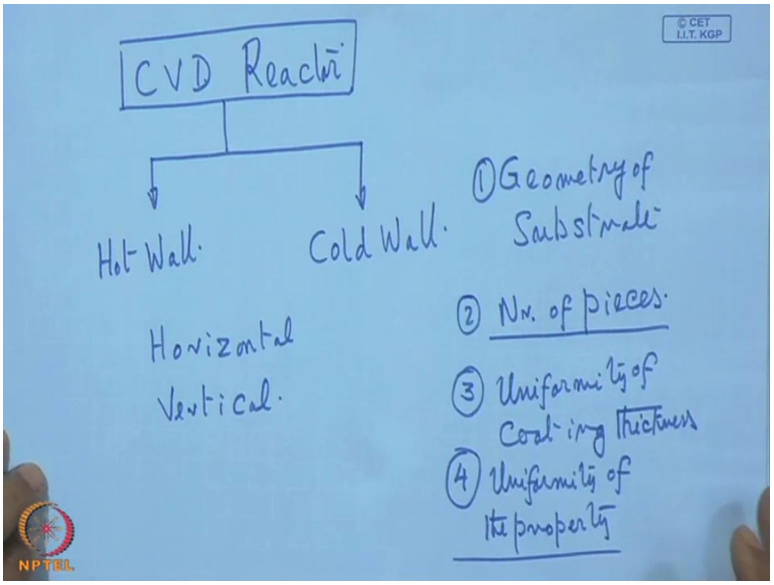

And fourth one, obviously uniformity of the property uniformity of the property, coating property, coating quality, uniformity of the property, these are at least one has to look. Geometry of the substrate, whether it is simple or complicated? Number of pieces means mass production or small batch production uniformity of the thickness and uniformity of the property. Now in this respect, we can also say horizontal, vertical, hot wall and cold wall.

But there are also type of reactor say for coating a substrate which is like a grain. Grain coating, that is also very important in terms of making abrasive product or abrasive surface or wear resistant surface. That can be used for various purposes. So grain coating, what should be? So it is going to be a very special type thing. Suppose we like to coat the inner surface of a vessel, a cylindrical object whose inner surface need to be coated and in that case how to coat that surface, that should be also taken into consideration. So these are the few things.

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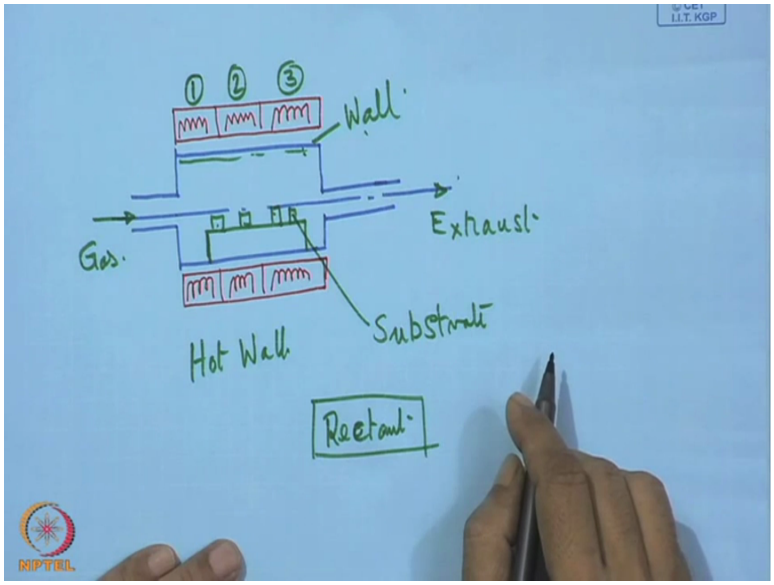

So what we can see here, hot wall reactor. So we can have just a very simple diagram to show this. Say this is one horizontal tube like thing and this can be either hot wall or cold wall reactor. Now since it is hot wall reactor, we must have the heating elements here. These are the heating elements. And we can have segmentation, that means this is one heating coil, this one too. So this is just one annular thing, so this is also continuation of that coil.

Here we have also one coil and this is also another zone. Say for example this is actually Zone 1, this is Zone 1. This is 2 and this is 3. And here maybe we have a table for holding the substrate over this. This is the incoming gas, gas supply and that is the exhaust. So in that case, if we use a hot wall reactor, what happens in this case, it is actually it is getting heated through this radiation of this, from this wall and partially convection and conduction through this gas.

So these are the main sources. But here what happens, this wall and the substrate, these are the substrate, these substrate and this wall, they are at the same temperature. So it is actually hot wall, so this wall and the substrate, so wall temperature and substrate temperature, they are same, they are at the same temperature. Now what is going to happen? It has certain, definitely certain advantages. Advantages is that we can have a high productivities. That means we can have a very high loading factor for this type, hot wall type of reactor because we can maintain a uniform temperature.

And another advantage of this, if we like to have different temperature gradient, that is also possible just by having different power supply to various zones of this heating coil. That is possible but one disadvantage that is that this gas can also react with this wall. So with this wall, it can react, that reaction product may have some kind of contamination, it can contaminate the whole CVD atmosphere.

And that can have a negative influence on the quality of the coating on the substrate. So this is one of the disadvantage. And at the same time, another thing we must also note that in this case what is going to happen? From this point to this point, if it is reasonably long, in that case this gas will keep on participating in the CVD reaction and there will be depletion of this reactant. That means reactant must have certain concentration and if there is some consumption of these reactants here which is not at all our objective or desire, there will be some shortfall as it moves on this side.

The result will be that growth rate of the coating on this point and which is very close to the exhaust, at the point of exit, they may not be the same, so this is also another thing one has to look. But in this case perhaps we can take some proper step and measure so that finally our main goal, that means to have uniformity of the coating thickness and also the quality of the coating all along this length of this, that is properly maintained. So this is one thing one has to also consider.

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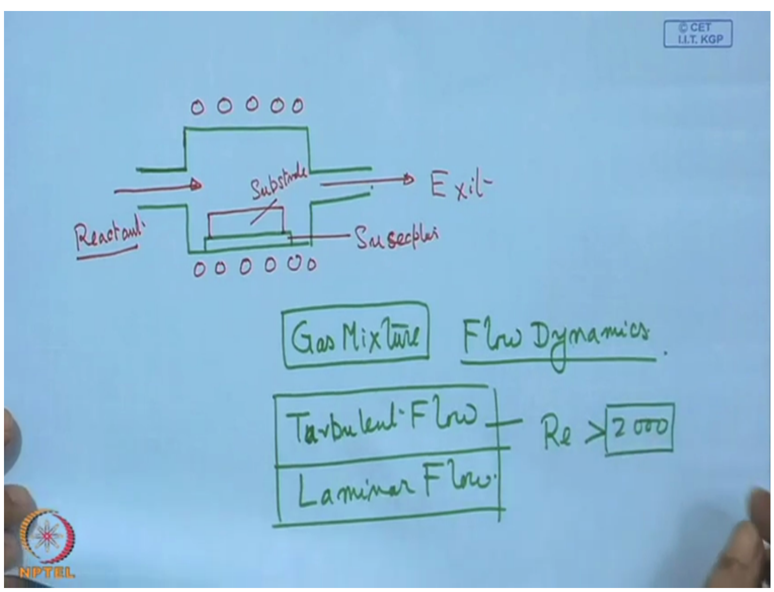

Now hot wall reactor we have seen but then we can also have a cold wall reactor. And in this case, what is going to happen? Let us have a quick look. So here what we have, we have the substrate. This may be the substrate table for resting, supporting the substrate. And here we may have some induction coil and this is going to be the susceptor. So this susceptor actually will be heated by this induction coil which will actually heat this particular substrate.

So in this case, what we can see that this wall is not heated at all. So there is incoming fluid, incoming gas and that is the exit, that means the byproduct. And this is the incoming, so that is the reactant. So in this case, there is no interference of this wall of this reactor, so it can be just one transparent quartz tube also. It can be a quartz tube and through that we can also have this, through this susceptor we can have this heating of this particular substrate.

But what is important here to know that in most of the cases, what we find that this gas mixture has some flow dynamics. This is also one important issue, flow dynamics. That means the question is, whether it is turbulent flow or laminar flow? So we have to make a choice here, whether it is turbulent flow or laminar flow. So what we can see in this case that from mixing point of view, for good mixing amongst this gas mixer, a turbulent flow is always desirable. That means Reynold number should be greater than 2,000, but it has certain disadvantages.

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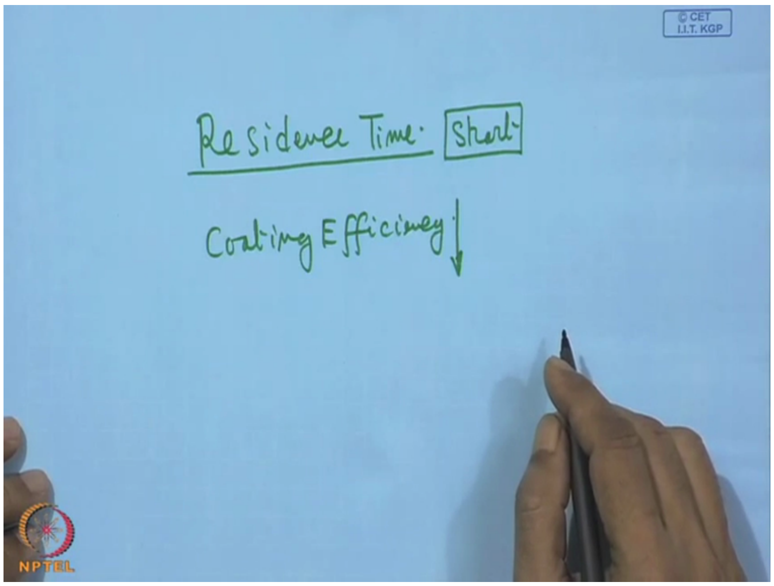

And disadvantages are, that means in this case the residence time on the substrate surface, that will be too short. And as a result of that, what happens? Coating efficiency is very low. That means in simple language the material which is, which has gone inside the reactor, only a small percentage is utilized in the conversion, in the reaction. And most of the reactant just exit without having any conversion. So it is a low efficiency process.

So this is one thing one has to also consider. But when it is a laminar flow, in that case the problem is that we have a problem of getting a uniform coating all through the length of the reactor. So all through the length of the reactor, how to have a better uniformity of the coating property? And for that, a design parameter that is considered.

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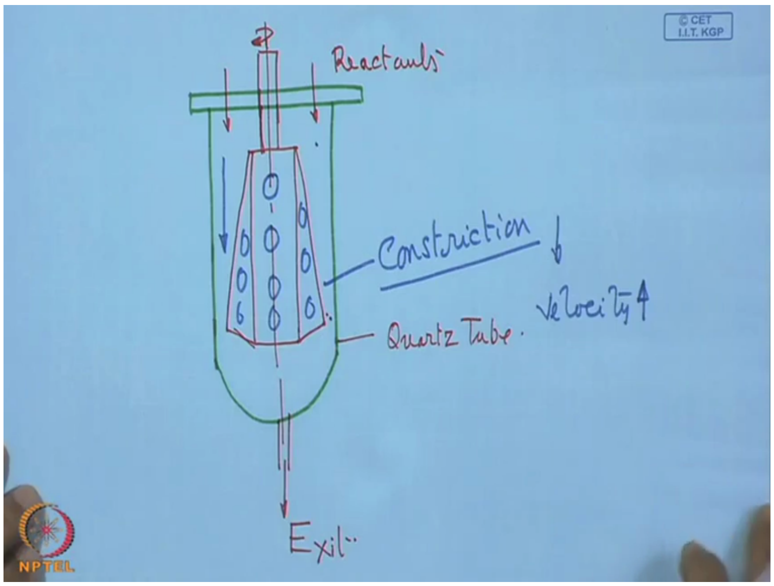

Say for example, we have a reactor of this fashion and here it is almost like a bell jar. So what we can do? We have a barrel, the barrel is placed like this. So this is the axis of the barrel. So this is actually a quartz tube. And here we have incoming reactants and that is the exit. So the barrel is something like this. So it will be, it has a shaft which is extended outside. So this will have a rotary motion. And here we can put the substrates.

So on this wall of this, we can put the substrates. So it is not parallel, it is little bit tapered. So what we find here that as it moves in the downward direction, there is a constriction. And with the constriction, we have a high velocity of flow. Velocity will increase and as a result of that, the thickness of the boundary layer which is formed on the coating surface or on the substrate surface at the initial stage, that thickness of that boundary layer will be thinner.

It will be of less thickness so that in that case the diffusion of these reactants that will be possible, that will be easier across this boundary layer and that will facilitate in getting a deposition of the coating at the same rate right from this point to this point and we expect a better uniformity of the coating.

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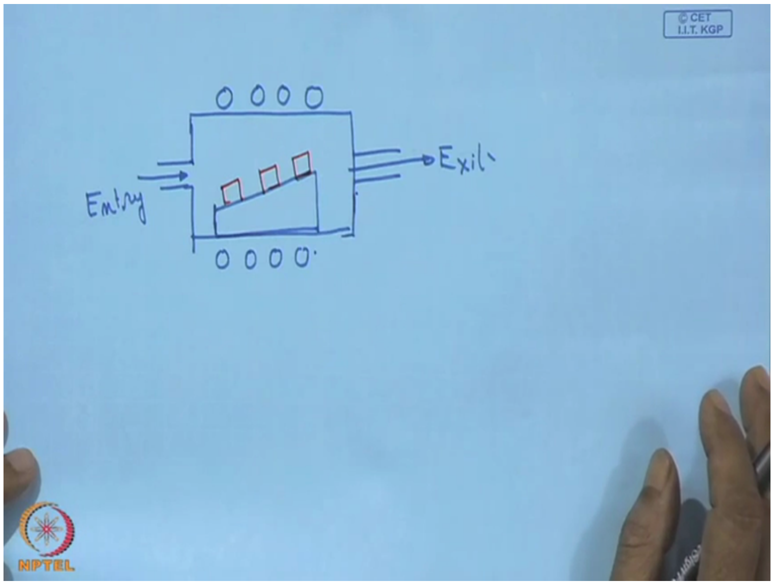

So this can be also done in a horizontal reactor. So this side, that is exit. That is the entry and here we can have a slope like this. And this can be also heated, this is just like a susceptor and it can be one induction coil that can be used. And here we can put some sample and in this case also we can have such kind of effect as we have mentioned in the previous example. So this way we can have radiant heater or we can have induction heating with some susceptor which will be heated by that coupling. And at the same time, we can have constriction of the flow passage. And with that flow passage constriction, we can expect better uniformity in the coating thickness.

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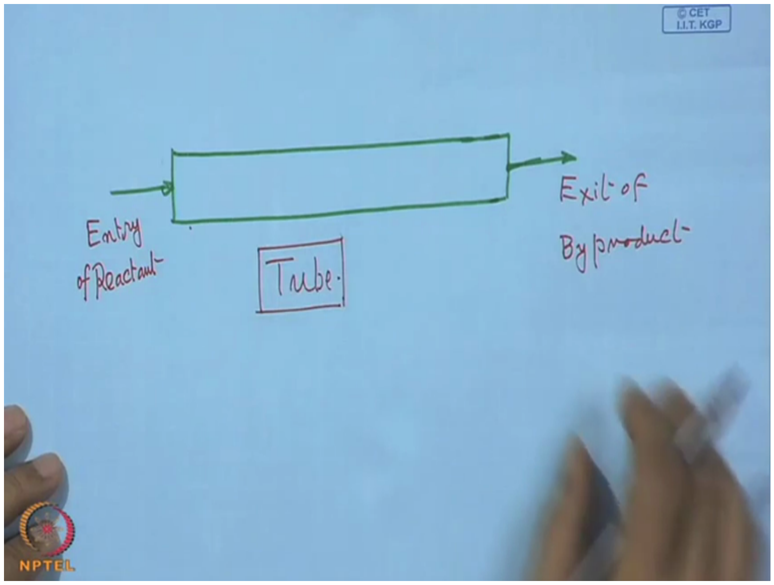

And then comes the substrate heating. Now another thing, say for example when we like to have coating inside a tube. So this is a tube, long tube. So in that case, the tube itself is the reactor and we can have, this is the entry of the gas, byproduct. And in this case, what we can see that it itself is a reactor but to have it is quite long. And what we have to do, that is a special technology. That means the entry point of the gas reactants while we have shown here or the heating arrangement, heat source, the way it is being heated, this wall is heated, that point can be shifted along the length or the entry point of the reactant, that can be also shifted.

In that case, what we can see that we can have a better uniformity of the coating over the surface. So in this case, the source of heat that can be also shifted or the tube can be also little bit shifted, can be shifted or it can also change its position to have a better uniformity of the coating over the entire surface. So this is actually a tube, coating of internal surface of a tube.

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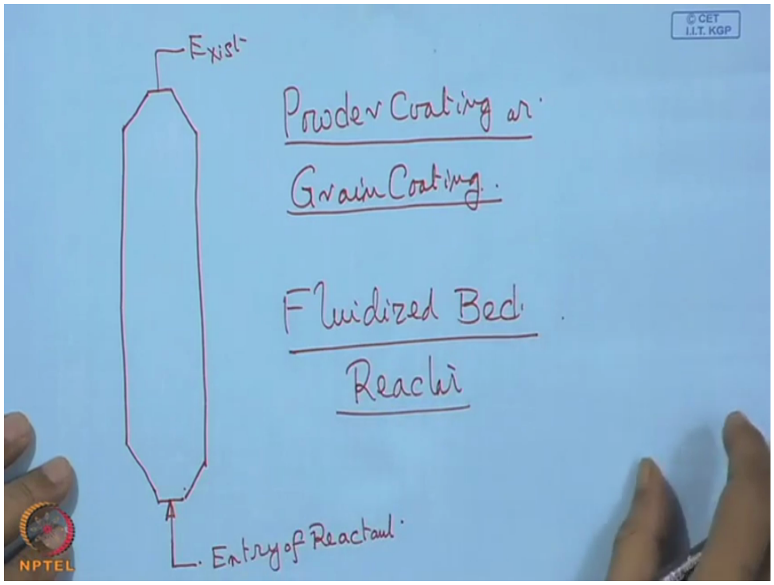

Now when it is powder coating, or grain coating, in this case we have to have a fluidized bed reactor. So this is the geometry of the reactor and we have the gas entry from this side. So that is the entry of the reactant gas and that will be the exit. And there will be sufficient buoyant force so that the grains will be in a floating condition and over, when they are in suspension, and (over) during the suspension, they will be coated and the entire surface will be coated. So it almost, so it is called the fluidized bed reactor.

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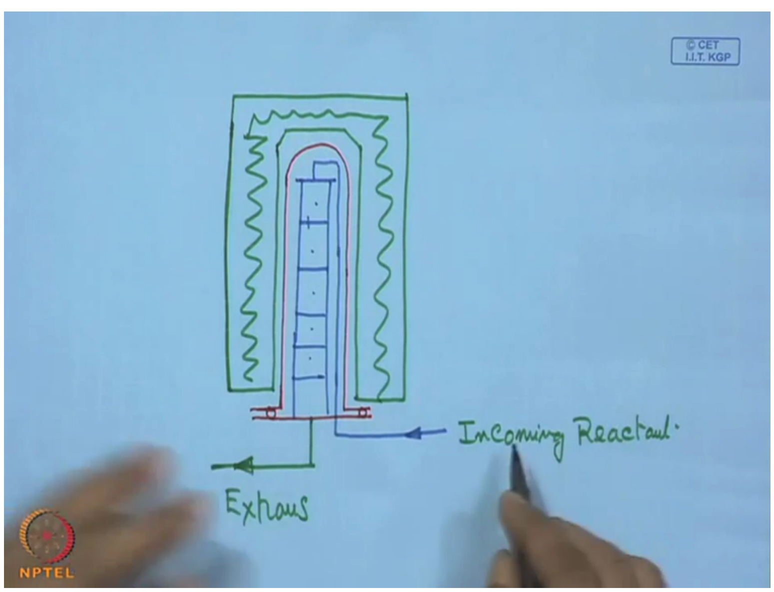

So for normal CVD what is done, it is mostly a vertical reactor. What we have seen normally, it is a flux and here what we have, this is the gasket and here we have a tower. So that means this is the tower. So this tower has various stages with perforations. So we have 1, 2, 3, 4, 5 stages and through that we have perforation. So here will be incoming reactant gas and which will be, which has an outlet and that will be connected on this side.

And what we have here, further to this we have here centrally one tapping point and that will be towards the exhaust. So it will go towards the exhaust. And this will be the incoming reactant. And here what we have to have, we must have this hot wall reactor, so this is actually the hot wall reactor, so which will have the heating coil. So here we have the heating coil. So this is actually the hot wall reactor. So this side whatever we have seen at the very beginning, that means this figure.

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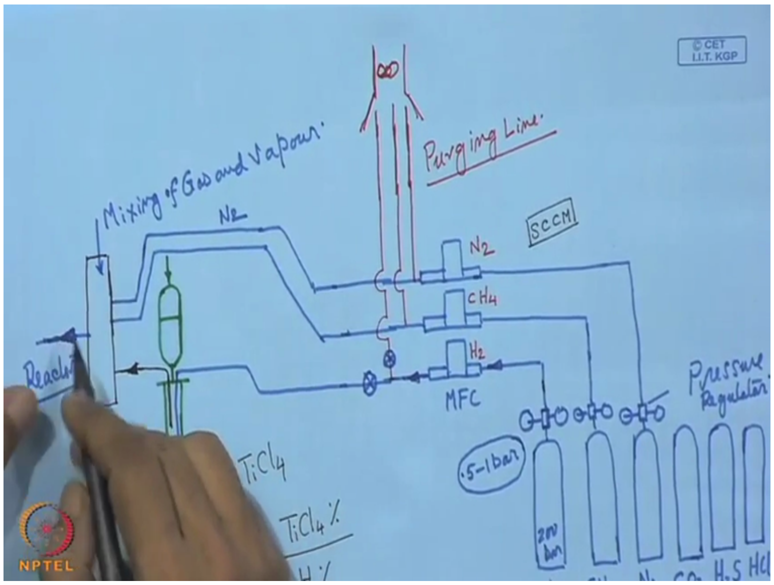

This figure we can show once more, that means this point what we have shown here.

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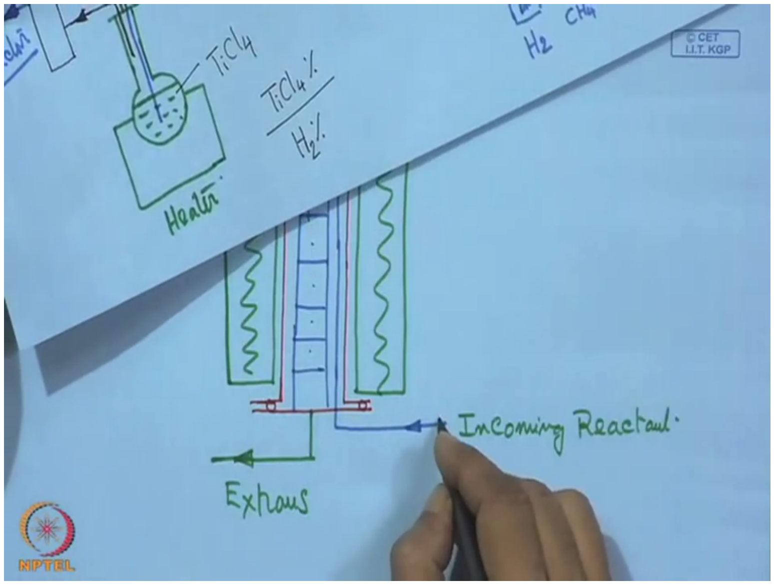

That will be connected at this point. And that is actually the reactor module.

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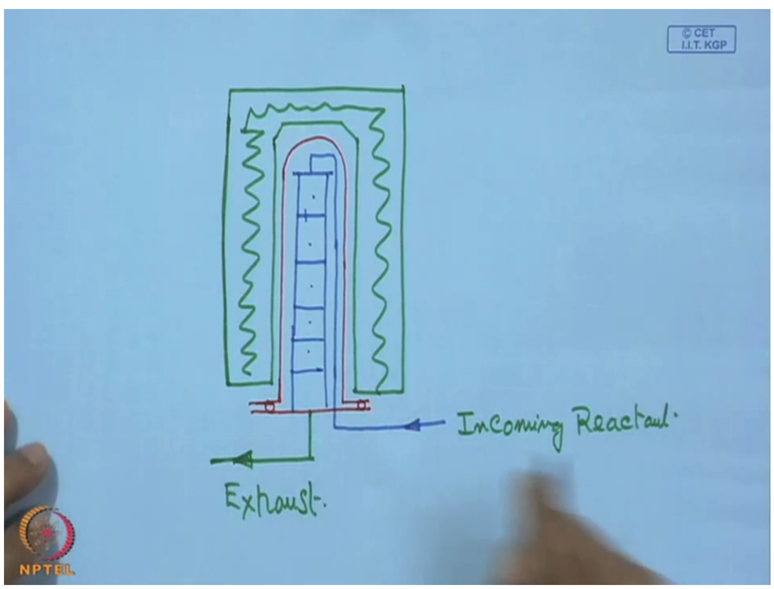

That means this is actually the coating deposition module which consists of the flux, the vessel, the CVD chamber along with a source of heat. It can be hot wall or it can be even radiant, in that case it can be a quartz tube and we can have some tungsten filament lamp for even heating that one. Now we go to this exhaust. Now what we have in this exhaust, we have to see that.

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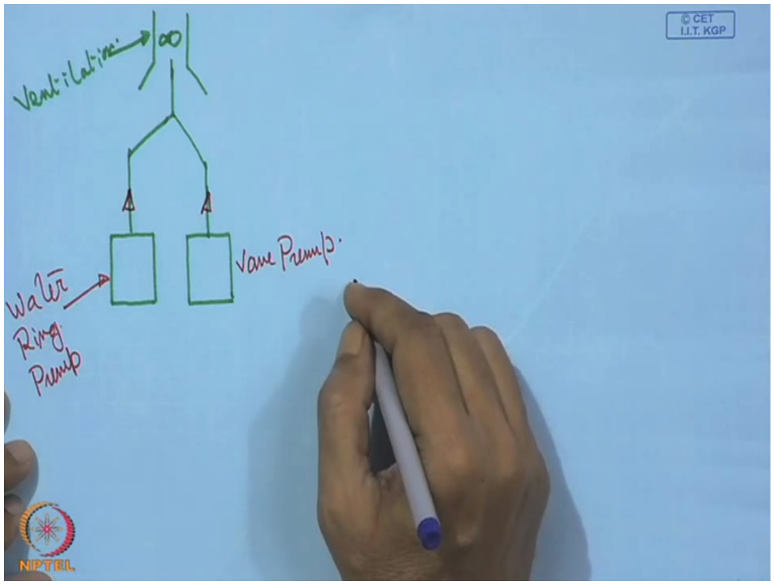

So from this point, what we have, here we have one, we can show symbolically two pumps operating in parallel, so they are connected. So this is actually ventilation. And one of this, that is called a mechanical vane pump, this is a vane pump. And this one is called water ring pump. This one is water ring pump and that is the vane pump. And that is the exhaust of this water ring pump and this is the exhaust of the vane pump. Now we have to have now the input.

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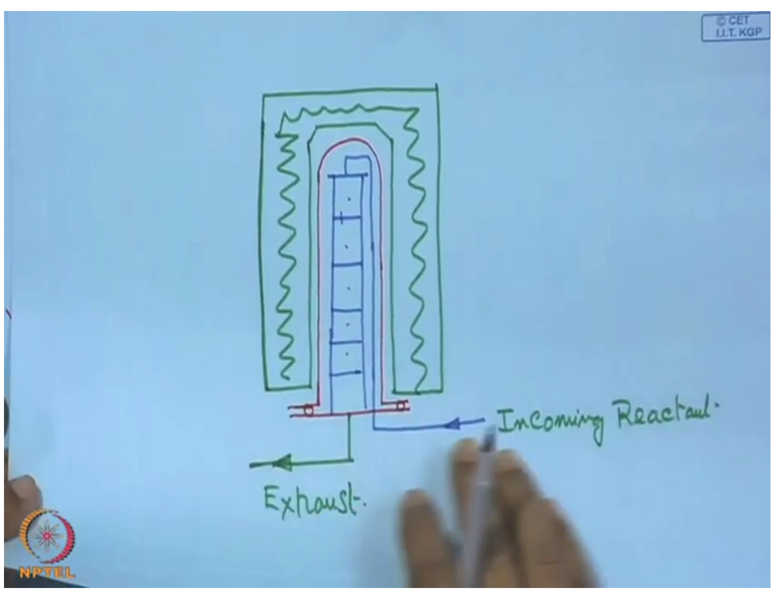

So from this exhaust, what we have shown here, this exhaust.

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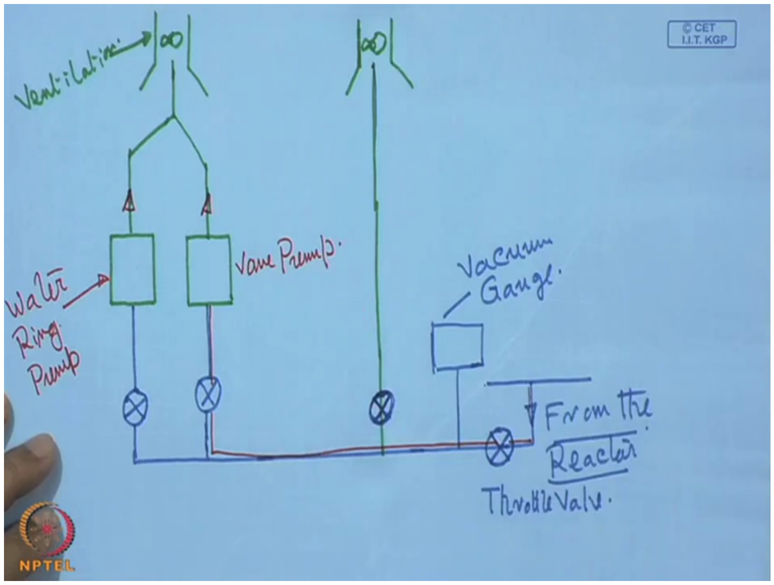

So there will be some plumbing work, so what we have here, in fact we have here one isolation valve. Here too, we have one isolation valve and then it goes like this. Plus, here too we have, this is actually the ventilation also. So here also we have one connection. This is from this flux, so we can have this thing. So here what you have to, what we have to have, here we have to have one vacuum gauge. So that means this is actually the vacuum gauge and this is actually from the reactor, this is from the reactor.

So what we see that, so what we see in this case that if we like to evacuate this system, then we should not, we should block this line. And here what we have, we have also one throttle valve here which can be 100 percent open or which can be 100 percent closed. So this is actually a throttle valve.

So if we like to evacuate the system after loading the chamber, then the upstream side that will be blocked, so there will not be any gas flow. So what we have to do, just by opening this throttle valve, 100 percent open and then there will be a valve. These are the isolation valve in every (connect), in every line we have this isolation valve which we have shown by this symbol. So this will be blocked, only this line will be open. Only this line will be open up to this. So the system will be evacuated. And after this evacuation, what is going to happen?

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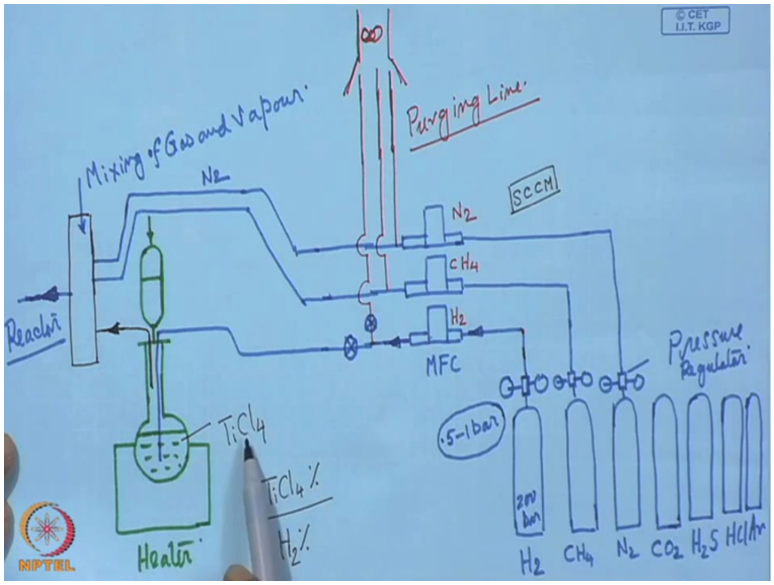

Then the necessary gas, what we can see immediately from this diagram, so all the gas supply including hydrogen, methane in some sequence following the principle, there we have to follow the principle of this CVD, the theory of this CVD. In what sequence, the gas has to be passed and to be admitted? It can be even argon also. We must have another bottle of argon here too. This is argon, so we have to have argon here. So (depend) it depends upon the particular sequence of operation and accordingly the gas will be passed. But this will be done after evacuation. And after this evacuation, the gas will pass.

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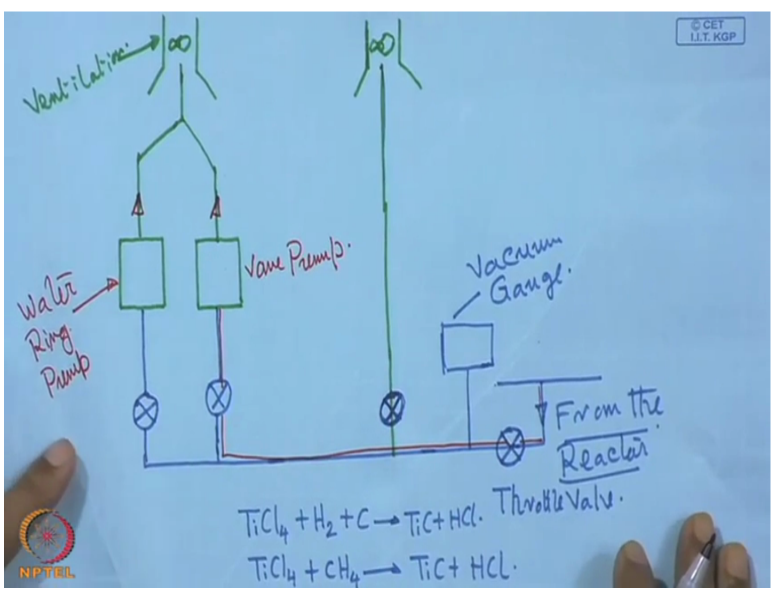

And when exactly this TiCl4 and H2, CH4, those are starts participating, in that case what we have, we have actually then TiCl4 plus H2 at the very beginning plus carbon from the substrate, we get TiC plus HCL. That is the first stage of this deposition. And it will follow this stage, TiCl4 plus CH4 and some hydrogen, so it will give us TiC plus HCL. Of course, we have to balance this thing. So this will be the reaction.

Now when this operation starts, that means actual CVD starts, in that case we should not use this vane pump, rather we should block this line. This vane pump will not be operational. We should block this line. And in that case, we should open this line. And with this opening of the line, this water ring pump, that will be operational. Actually in this water ring pump what we have, it is actually a ring formed by water because of the churning by the impeller, by churning by the impeller. We have a wall formed by this water which will be thrown by the centrifugal action. That will be thrown towards the wall of this pump, that means the stator.

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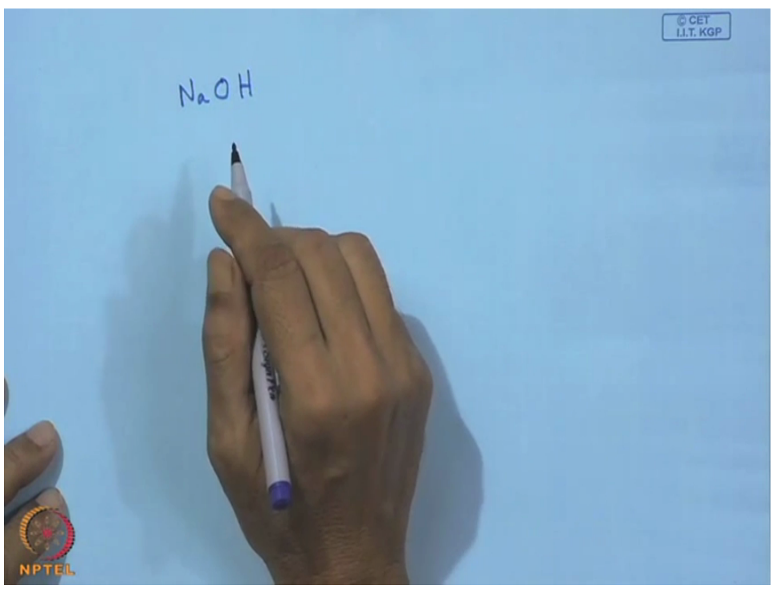

But this is just not water, it is actually in many of those cases, it is actually aqua solution of sodium hydroxide and this sodium hydroxide.

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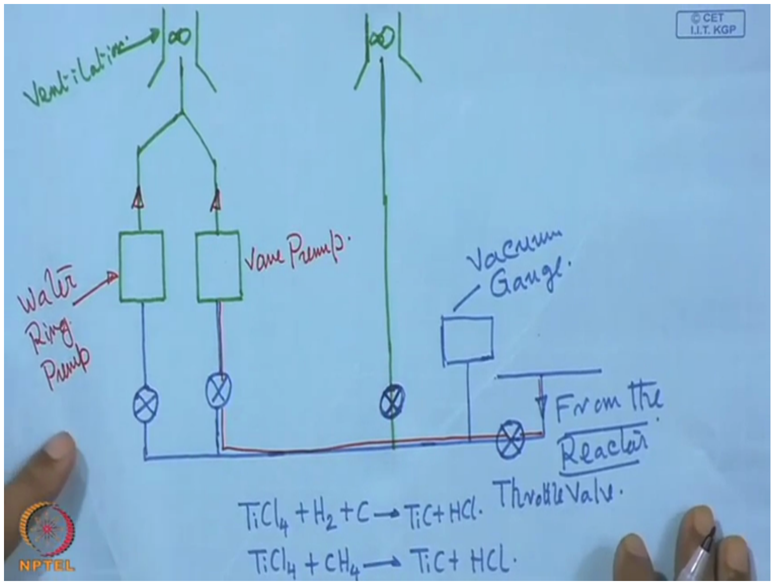

So when we have HCL, that means if we allow this HCL to pass through this vane pump, then this pump will be corroded and it will be damaged.

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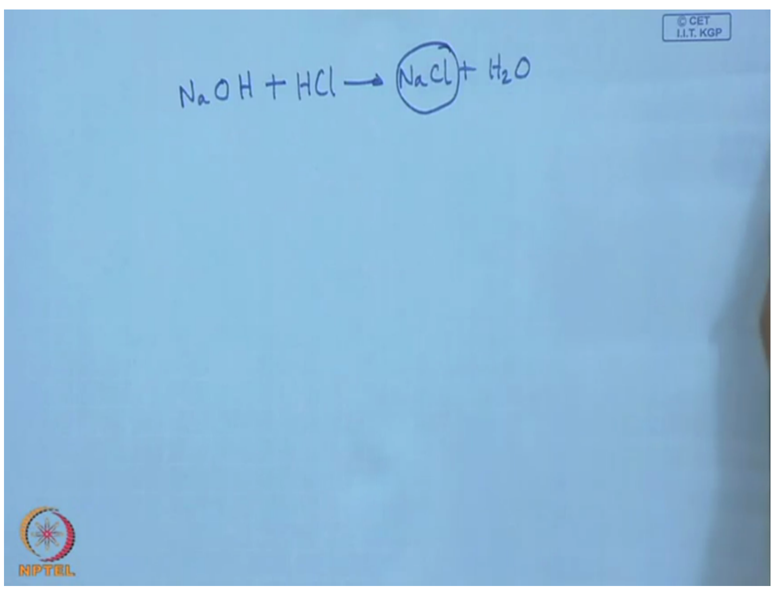

But in this case, what is going to happen? This will be thrown and churned and that will be absorbed. So in this case, it is quite likely this will give us NaCl and H2O. That means okay, so it will be NaCl dissolved in H2O. And with passage of time, this reaction will proceed in this direction. So concentration of NaCl will increase and this concentration of NaOH will decrease so rate of reaction or absorption that will fall.

So this pH value of this solution, that can be examined and accordingly fresh NaOH can be replaced and replenished. So this way this water ring pump, so we do not allow this HCL disposal in the environment, just it cannot be released, so it is actually absorbed by this one. So this water ring pump will be useful throughout the whole operation.

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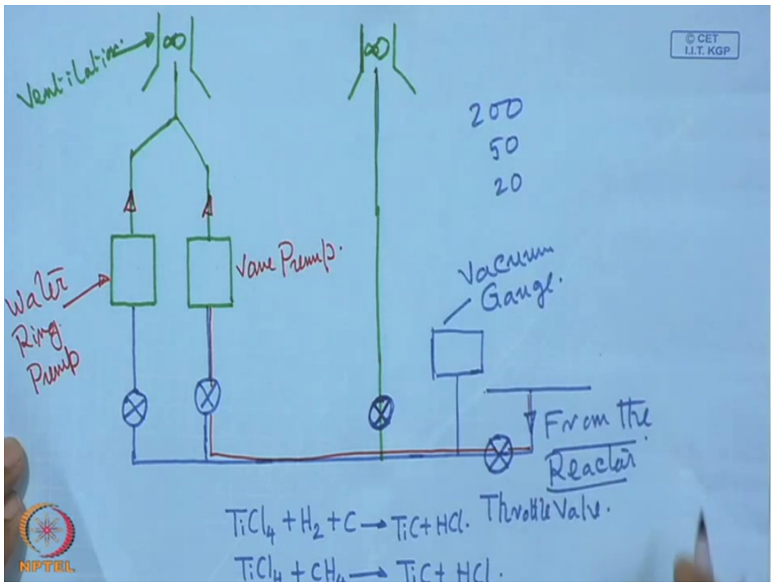

So what we find here that this side is actually what we call exhaust module. So exhaust module consists of a motorized throttle valve, a vacuum gauge. So vacuum gauge will maintain the pressure and accordingly the volume will be, this opening will be adjusted. Depending upon the process pressure if it is 50 torque or 20 torque, or 200 torque whatever may be the set value, accordingly that will be set and this motorized valve will be, will adjust itself keeping the same opening corresponding to a particular torque. And there we have this vane pump and water ring pump along with one line available for ventilation. So this is actually the exhaust module or the module of the downstream side.

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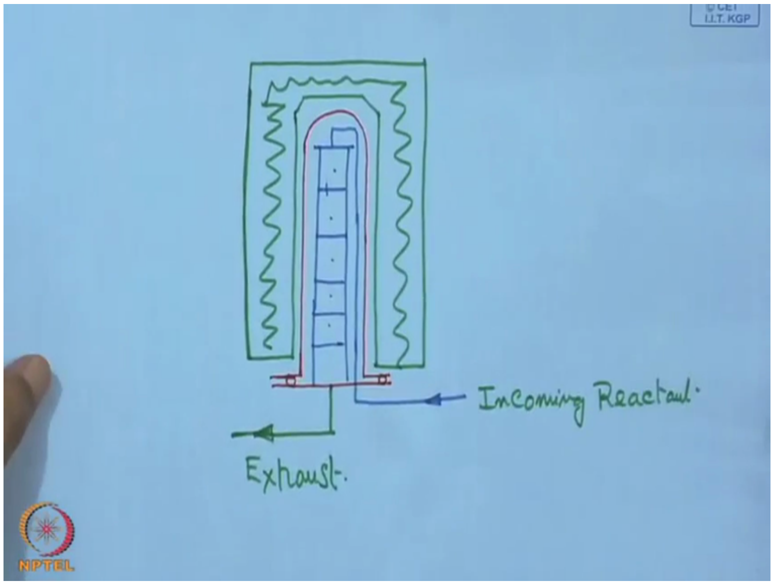

So this downstream module plus the module, this is the reaction module or the CVD reactor.

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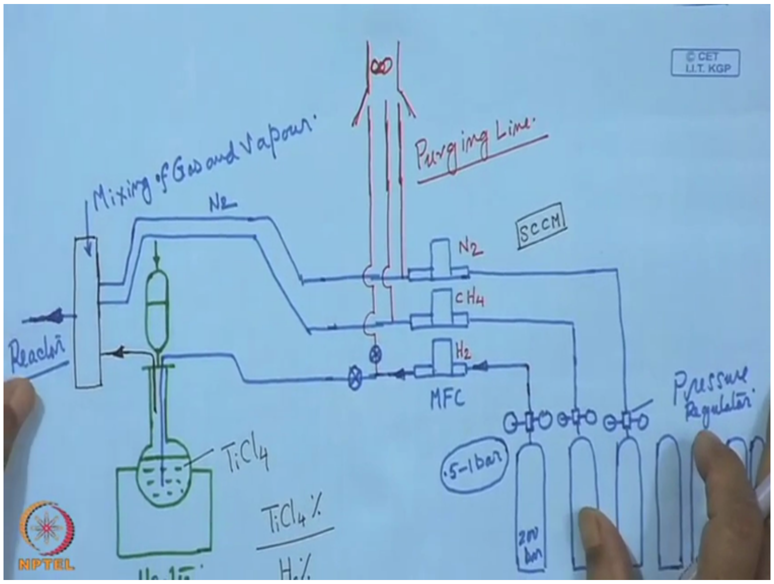

Plus this one what is the gas dispensing module, taken together that makes a complete CVD system.

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So these are the thing what we have discussed and we can summarize that a basic CVD system which is suitable for industrial practice, that mainly consists of gas dispensing system and in the gas dispensing system we have precise metering of the gas. We have, and also the precise regulation of the working pressure of the gas, plus one sublimator, evaporator or one generator for supplying the metal donor in the form of a vapor which will be saturated with hydrogen. So this is actually the upstream module.

Then comes the reactor module and this reactor can be hot wall reactor or cold wall reactor depending upon the reactor geometry and the substrate geometry and the property. Each has its merits and demerits. We can also have reactor in the horizontal mode or in the vertical mode. A tube can be itself a substrate, can becomes one reactor. And for grain coating, powder deposition, deposition on powder, we need to have one fluidized bed reactor.

And on the downstream side what we have, we have a vacuum gauge, a motorized throttle valve which keeps the CVD chamber pressure at a set value. Plus, one vain pump, that is for evacuation before the start of the CVD operation and one water ring pump which will allow this byproduct of this to be absorbed in the pump because it contains an alkaline solution and this HCL or similar material is not just directly released in the atmosphere so all these things taken together, that makes a complete CVD system suitable for industrial and commercial practicing of this CVD technology.