

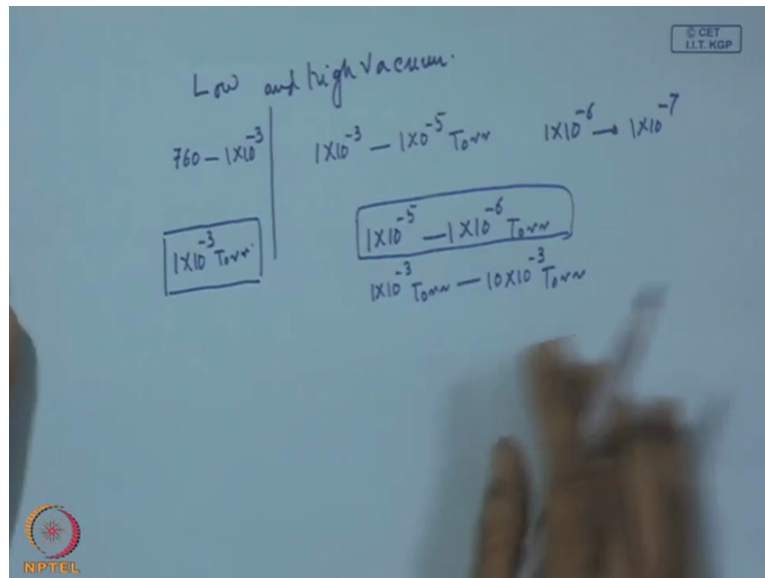
Technology of Surface Coating
Professor A.K Chattopadhyay
Department of Mechanical Engineering
Indian Institute of Technology, Kharagpur
Lecture-31
Production of Low Vacuum

In this topic we like to understand the role of vacuum technology in the deposition of paper coating that means whenever we conduct a process of deposition, it can be CVD, PVD, it can be some spray process, it can be coating by heating, it can be electro deposition. Now in most of the cases we need one control atmosphere or a restricted atmosphere and by this we mean that the pressure inside the reactor or the deposition chamber has to be properly controlled and in most of the cases it is below atmospheric, hardly there are process which can be conducted with above atmospheric or atmospheric within a chamber, but there are process which can be conducted in open atmosphere.

Now here we find the role of vacuum technology, the technology means here that means it is the technique of producing a vacuum and in a particular degree. Now we know that 760 millitorr of mercury or thousand millibar that is the equivalent that is actually we understand as atmospheric pressure. Now depending upon process it can be say 760 millitorr that is atmospheric pressure, it can be conducted in several hundred torr, it can be just one tenth of a torr, it can be some tens of torr, it can be 1 into 10 to the power -2 , -3 or even 10 to the power -4 .

So these are the degree of vacuum as required by a particle process and accordingly there must be some ways and means to achieve that vacuum in a consistent manner throughout the deposition process without any I mean risk of failure of the vacuum system because then the whole purpose will be lost, so this is one important aspect of coating technology or surface coating technique and this vacuum technology is a part and parcel of this though it is a peripheral support but one of the most important support for a particular coating process. Now here we understand that this is a lecture on production of low vacuum that means we can just classify low vacuum and high vacuum.

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The term is very-very relative but from our user's point of view we can make some broader demarcation that means low vacuum means maybe from 760 to 1 into 10 to the power - 3 and this is one range, we can also have from 1 into 10 to the power - 3 to 1 into 10 to the power - 5 that is the torr and something even better than 1 into 10 to the power - 5 means it can be 10 to the power - 6 or it can be 1 into 10 to the power - 7 or - 8 or - 9.

These are the very special demands and requirements but for all routine things, routine things what I mean those processes whatever we have discussed so far for those we need a vacuum at least in certain cases CVD say few 10s of torr or few hundreds of torr that is a CVD process. However, that means during the process we have to maintain a pressure, it may be few tens of torr or few hundreds of torr however there must be a pre-vacuum to remove air and oxygen so in that case the pre-evacuation that should be in the order of at least 10 to the power - 3 torr. So for all routine CVD this is just pre-vacuum pre-evacuation of the reactor before we can really start the process.

Now when it comes to the PVD process where cleanliness is of I mean extreme important and in that case a pressure between 10 to the power - 5 to 10 to the power - 6 that is pre-evacuation that is most demanding and we cannot here compromise with this base pressure because in that case the residual oxygen which is present in air that can also cause some contamination of the target particularly those transitional element targets which are very-very reactive with atmospheric oxygen and polluting the entire chamber which may ultimately

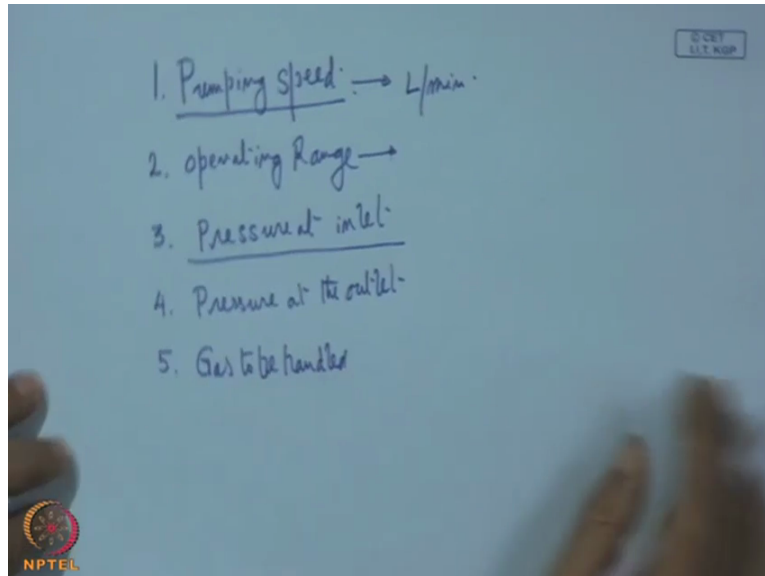
lead to poor quality of the coating, though the process pressure may be say it can be 10 to the power – 3 torr to say for example, 10 into 10 to the power – 3 torr that is the process pressure.

But evacuation to this base pressure that is just one has to arrive and then the process pressure can be set to any of these values by proper throttling and by incorporating gas. So here we understand for most of the CVD process a pre-evacuation of this order is necessary and for a sputtering iron plating then this all sort of evaporation process, arc evaporation process, base pressure of this order is essential. So now what we see the production of low vacuum, the production of low vacuum let us see exactly what is mean by reduction of pressure.

Now here we have a device which is called the pump, so it is well-known for any user the role of the pump, but in this case the role of the pump would be to reduce the pressure inside a chamber and at the beginning of the process it should be when we open the hood or the door, it is totally atmospheric pressure and after loading the sample or specimen to be coated or treated, it has to be evacuated and that can be done by this pump. That means pumping down is the process that means getting the pressure inside chamber reduced.

Now by this what I directly we mean that it is actually reducing the number of gas molecules within the chamber so it is the role of the pump that means at the atmospheric pressure within a given volume of the vessel there will be certain number of molecules and when the pump starts operating connected to that chamber, it will be just reducing the number of molecules inside the chamber and that will be translated into reduction of pressure and which will be measured by some pressure measuring device like parameters or there are certain cases which can be used for measurement and assessment of the vacuum.

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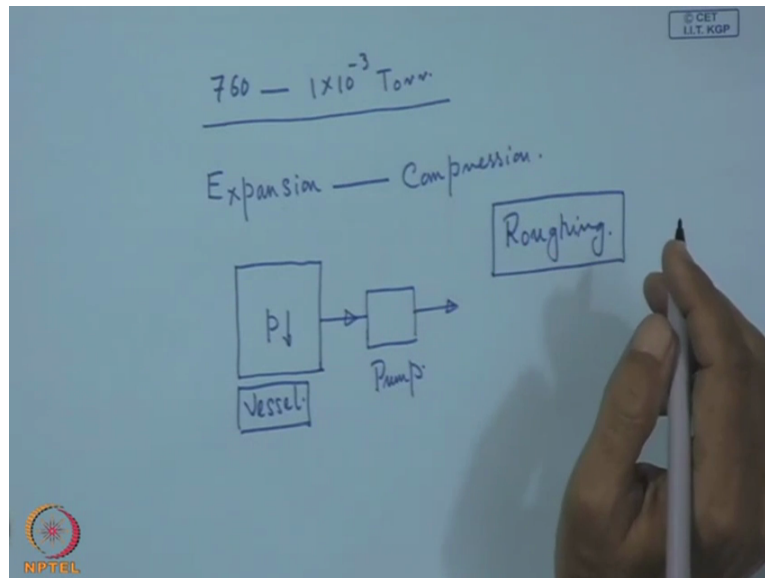
So what we see here, parameters of the pump, now here parameters of the pump it should be guidelines for proper selection, now just like in any device what is very important that is called the pumping speed, pumping speed that means how many litres of this residual gas can be thrown out so that is one of the parameters of the pump, pumping speed litre per minute or litre per second. Then comes another important parameter that is the operational range, operating range that we should be very clear about that means all the pumps we cannot put to the chamber which is say at atmospheric pressure, so there must be a coupling between just not one pump, so one will follow other so one will give some backing about what will be used in series so that is the operating range over which it can work.

Then come very important thing that pressure at the suction side that means to what pressure it can bring down what is the level of reduction of pressure that means pressure at the inlet, pressure at the inlet of the pump that means the port inlet port which is connected to the chamber to be evacuated that is called the inlet port and we have also another called the exit or the exhaust port, so it is the inlet pressure at the inlet. Similarly we have to know, what is the pressure it can deliver at the outlet that means at the exit?

This point is important in that there are many pumps which cannot deliver the pressure at the exit point above atmosphere so in that case we need another pump which will be a backing so that it can be coupled in series in the downstream side and that can raise the pressure of the residual gas above atmosphere and then that can be thrown outside so this is also another important parameter so what we can find, pumping speed operating range, pressure at inlet and pressure at outlet, so these are the 4 parameters at least one has to look in, then also another thing that is also important that whether what type of gas it is handling, whether it is

inert, whether it is reactive or whether it is corrosive and accordingly the material or construction of pump has to be chosen so this is also the gas to be handled that has to be also enlisted, Gas to be handled so these are the parameters for the pump.

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Now principle of pumping for low vacuum, now in this case what is the principle? Now we have different types of terms say one case it is just the displacement that means few parts in the pump those are moving, either it can be rotary or it can be reciprocating and there is one casing or a stator and there is one moving piston or a rotor so that is one form of pump and these are we call mechanical pump. So these are mostly recommended for achieving a vacuum not a very high vacuum so maybe well within 1 into 10 to the power - 3 torr, so from 760 to 1 into 10 to the power - 3 torr it is mostly this is engaged that is the mechanical pump which are engaged in this range.

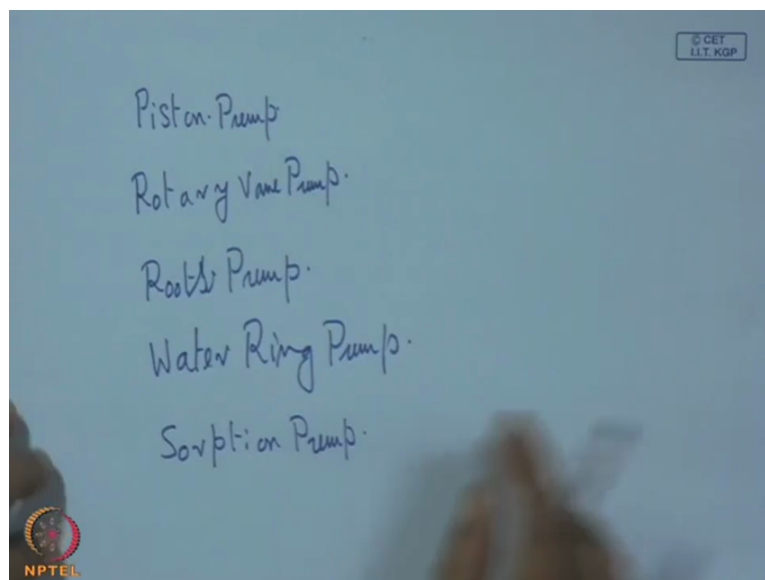
Now principle of pumping for low vacuum that what we mean, it is actually expansion followed by compression, expansion followed by compression say for example, we have one chamber which is filled with atmospheric air and then we have to evacuate this thing then definitely to draw to draw this thing with the help of a pump that means in this downstream side the pressure has to be below then pressure what is prevailing here otherwise there will be no movement.

And then this pump will handle just by expansion of this so that pressure inside that will fall because of the expansion that means we have to create a volume which is more than this volume and thereby we can have the expansion and later on it will follow it will follow this compression will follow his expansion so this expansion will be followed by compression and during the compression what is going to happen we have to increase the pressure above

atmosphere so that it can be thrown outside so this is expansion reduction of pressure and then by compression increase of pressure above atmosphere.

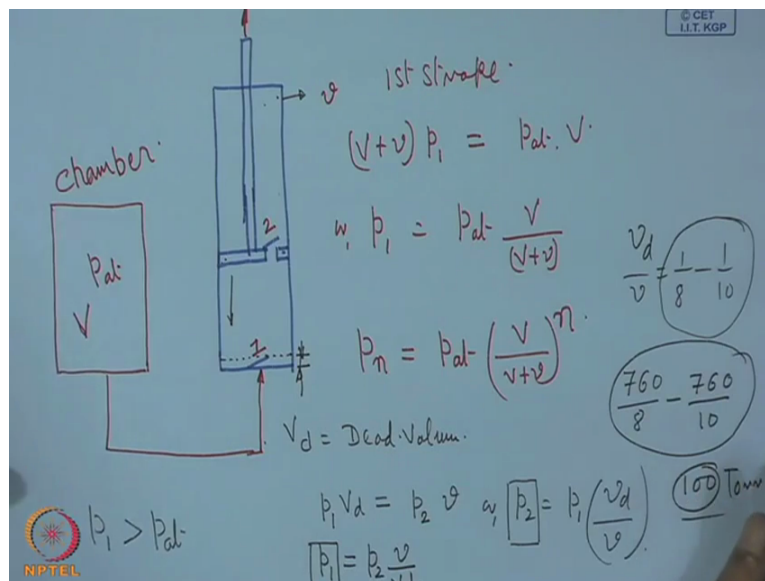
So if it is atmospheric pressure, pump will reduce in its chamber this pressure below atmosphere at that it will be increased above atmosphere and then it will be thrown out, so this is the pump and that is vessel or a chamber which is to be evacuated, so that is called principle of pumping for low vacuum, but there are exceptions we can also mention that that without any rotary part or a moving part the pump also can be used for this low vacuum operation and in the language of this vacuum technology, this low vacuum evacuation that is also called Roughing, that means rough pumping or simply roughing, so roughing means actually getting the pressure down within this range and not below that. So this roughing operation we have mostly it is rotating pump but we do have also non-rotating pump.

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Pumps for low vacuum, so pumps for low vacuum that we can see here one is piston pump, then we have Rotary vane pump. We have here Roots pump and then what we have here Water ring pump and there is another Sorption pump, so this sorption pump that is used for absorbing the gas molecules on the surface of a specially formulated material and that has very high surface area compared to this volume and thereby this material will be absorbed on the surface either by physical absorption called by chemisorptions. So either it is physisorption or chemisorptions, by these 2 processes this sorption pump can also work and here no part is rotating, so these are in the domain of pump for low vacuum.

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Now let us look into the piston pump, so piston pump is just like a barrel and here what we have? We have here one valve and then we have this piston and this piston also can have another valve and this can reciprocate. Now here what we have, we have the vessel to be evacuated and we can have connection with this tubing with this plumbing to this point so this is valve 1 and this is valve 2, so this is the volume to be evacuated. Now say this is capital V, so this volume means volume from this chamber up to this point so this is a tubing for connecting to this pump.

Now what happens, this piston can move up to this point so that is the topped point end thereby what happens that is the actually the volume of this barrel when this piston go to the topped end and if that volume is small v, what we find that as we move this piston in this direction then this volume v is gradually increasing and it attends the maximum value when this piston is here and in that case we can equate suppose now this volume v that is at atmospheric pressure say for example, now what we can do here just by equating we can write that for the first stroke as we take it there so there will be a fall of pressure within the system because of the change in volume so what was originally V that is now capital V + small v enlargement of the volume and that is why this atmospheric pressure has to fall.

So if we have the fall of pressure, if we write it just like P 1 so that should be equated by P atmosphere into V or we can get $P_1 = P_{at} \cdot \frac{V}{V+v}$. Now this is one stroke and we can have nth stroke, after nth stroke we can have P atmosphere by this into to the power n, now this way we can go however one thing we have to also look in so to

what extent should we can get down with this pressure. Now here we have to look in very important thing that this piston during its downward movement what happens, then this is coming so the whole gas which is admitted within this volume V when it was here, then as it the piston comes down, this port will be closed so connection will be cut off and in that case what is going to have now this is at P_1 pressure and here this pressure P_1 will keep on keep on increasing because of the change in volume and it will squeezed.

Now this piston cannot come to the surface so there is some clearance left so it will come up to with point, so this is called actually the dead volume so V_d that is called the dead volume and so so it can increase the pressure, it can compress the pressure up to a volume which is corresponding to V_d , so this way it will the pressure after this squeezing here that will exceed the atmospheric pressure and in that case this valve 2 will open and there will be some space for evacuation of this thing because now the pressure is more than atmosphere so this will escape through this valve. However the point what just we have mentioned here that we can also have another equation that means this P into V_d that will be given by this say if it is P_1 and if it is P_2 then what we can see that this P_1 and P_2 that into V .

That means what we can get this P_2 will be equal to P_1 into V_d by V and this is called the compression ratio that means if we have a situation where this pressure after squeezing if we cannot increase this pressure P_2 say this is the pressure so what we have here P_1 that means here what we see this is P_2 but we can also write $P_1 = P_2$ into V by V_d so that means this is the actually reduction of pressure but because of the squeezing P_1 will be more than P_2 . However after this squeezing what has to be done that this P_1 it has to be greater than P atmospheric pressure in order that this Valve 2 will open. But if we have a pressure which corresponds to this P_2 that means this P_2 will be coming from this equation that means a particular point the residual pressure within this that is actually P_n .

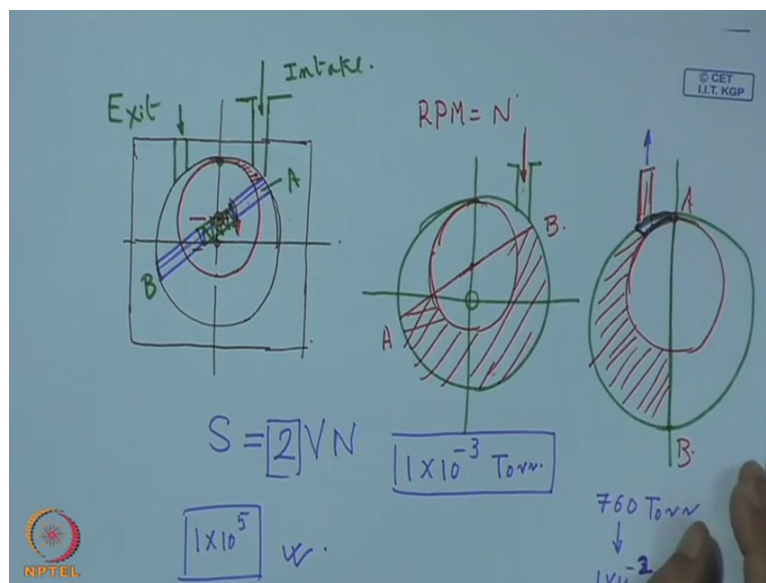
But this P_n after squeezing to this volume dead volume V_d if it cannot attain the volume, note down P atmosphere, in that case there will not be any further evacuation. So what is going to happen at this point? At this point it will be simply expansion and contraction of that volume of here so it will during the movement of the Piston in the upward direction there will be expansion of the gas and during the downward movement there will be compression of the gas but that compressed gas does not have the required pressure for opening of the valve.

So this way we can see this limitation and this ratio ratio comes about this dead volume to the volume of the barrel this V by V_d that means this V_d by V that is about $1/8$ to $1/10$ and as a

result of this the pressure what can be attained in this case, it will be around it is 760 y 8 so or to 760 by 10 for... As a general rule we can say that pressure attainable by this piston pump will be around 100 torr so it cannot go beyond that. So this is though it is very handy, very useful pump but we can attain a pressure about 100 torr in this case.

Now come the Rotary vane pump, the rotary vane pump is one of the industrial pumps which used with any commercial vacuum system and that is coupled to CVD or a PVD coating machine or equipment so that the necessary evacuation is possible. In fact it is a rotor and this rotor is kept within housing that is called the stator and let us have a quick look in this case.

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So we have this is the casing of the pump that is called a rotor and here we have this casing something like this and inside what happens, inside eccentrically a rotor is placed. So this has eccentricity so it is the axis of the stator and it is the axis of the rotor so this is the eccentricity and this is moving in this direction, so that is the $RPM = N$. Now here what we have Now here what we have? We have 2 vane rotary vane so through this we can have the axis and here what we have, we can put a slot , this is one vane and this is it is matching with the circular arc of the stator so that means it is in bodily contact.

Here also we do not have much clearance so it is also actually in contact so we have another vane on this side so what we see here, a slot is cut diametrically along one diameter and in that slot would be put 2 plates like Vane, so this is the thickness of the Vane and it has certain width normal to this plane of the paper. And in between what we have, we have here a spring, so this is the spring so with this what we see that these vanes, now here what we have, we

have one intake port which is connected to the pump and here also we have one exit port so this is intake and this is exit, so this is vane A and this is vane B.

Now, what we can see here that from this point so this whole thing rotor is rotating continuously and during this process what happens, this A and B they are changing their position with respect to this intake port. Now as this chain will rotate what we can see, we can have a quick look here without going into details of this drawing what we can see that this is the axis of the stator and what we can see here, you changed their position so what we can see that now it is A is here and B is on this side so as this B is gradually moving on this direction A is moving and B is also moving upwards in that case what we can show here by this hatch mark this volume will keep on increasing.

So that is actually the volume which is directly connected with the vacuum chamber and this volume will have the maximum capacity when these 2 are almost here so that will be the volume maximum volume which will be swept by these 2 vanes and this will be the swept volume so that will be the total volume that means when we have such thing so in that case as it comes here so then the volume will increase and that will be the volume that means now the gas which has been evacuated and inducted by this expansion process, so because of this expansion of this volume the pressure will fall and there will be a stream of gas which is flowing inside and now it is interrupt here so it is isolated.

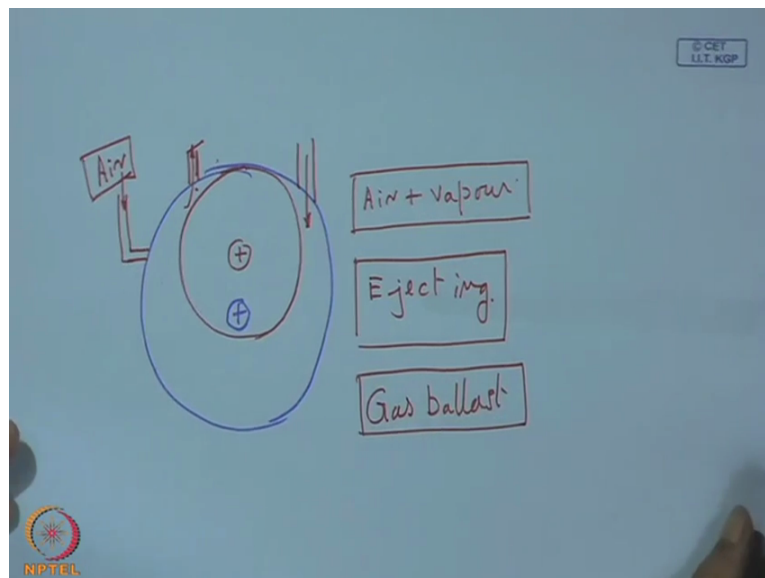
Now for the moment this chamber is isolated from these 2 vanes and A and B have changed their positions. Now what is going to happen further to this, what we can see further we can have another drawing here so this will be the position of this in (())(34:55) and then this A will be on this side that means when A is here, now A is here and B is on this side so this interrupt volume has changed its position now it is on this side.

So now what is going to happen, we have this exit port and here this volume will be squeezed and compressed gradually and because of this we have here one valve and this valve will open because of the squeezing action that means the volume which was expanded up to this which is shown here by this hatch mark that will be again squeezed over the surface and then the pressure will build up in the zone and this will be squeezed up to this point that means the squeezing will be done between this point of this port B, B will move this side up to this point B will move and then we have this confinement through this surface so that will be the volume and that will corresponding to a pressure which will be more than the atmospheric pressure and there will be exit of this gas.

So this will be actually the dead volume in this case so for the removal of this material is not possible and this way it will keep on working and here the pumping speed we can write the pumping speed will be actually $2 V \text{ into } N$, now what is V ? V is the volume maximum volume of that created by this impaler and through this volume this material will be swept and this is equal to V and it happens twice in one rotation that means one once A will be actively participating in pushing the material and in another case when B will be doing the same job. So it will be twice in one rotation then that is why we write 2 and that will be $2 \text{ into } V N$ so that will be the pumping speed.

Now here what happens, this is actually dead volume so if we are interested to get down to a pressure of $1 \text{ into } 10 \text{ to the power } - 3 \text{ torr}$ then we must have a compression ratio of at least $1 \text{ into } 10 \text{ to the power } 5$ because of the simple reason, if we have $1 \text{ into } 10 \text{ to the power } - 2$ that means from 760 torr we have to get output to $1 \text{ into } 10 \text{ to the power } - 3 \text{ torr}$ and rounding of this 1000 we can get here that this is $1 \text{ into } 10 \text{ to the power } 2$ if we like to have a pressure of $1 \text{ into } 10 \text{ to the power } - 2$ then rounding of this to 1000 we must have a compression ratio $1 \text{ into } 10 \text{ to the power } 5$ that means this volume which is available for inducting the gas and that is the maximum volume which is available and that is squeezed to this dead volume.

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So this is eventually the dead volume, so this volume to this volume that should be actually $1 \text{ into } 10 \text{ to the power } - 5$. Now here one thing we have to also mention that in this case we have also gas ballast, gas ballast means in this compression side in this side we have to have we can have a quick look here that means on this side so these are the rotor and stator that is

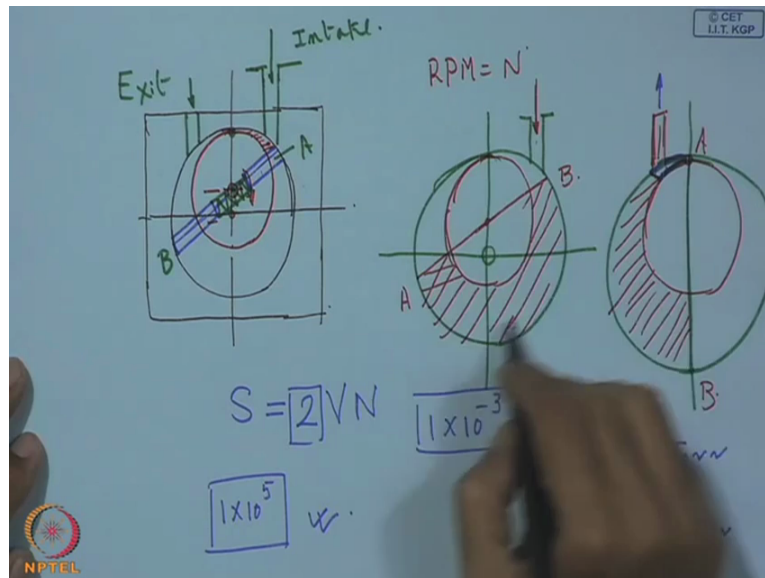
shown so on this side what we have to have, here we must have one entry for air and the whole idea here is to that if this pump admits some moisture or some condensable gas from this vacuum chamber then that will be also squeezed and that can undergo some kind of condensation and during this condensation what will happen in this case?

There is oil so this will be actually contaminated with the oil this will contaminate the oil because on this side we have oil reservoir and that is used this oil is used not to allow any air leakage in the in this barrel so this is actually the barrel and this barrel extends normal to this plane of the paper so not to allow any leakage of air in the barrel we have to have this oil and this contamination of this oil is prevented by just looking by just putting air, so now what we have instead of this condense instead of this condensing liquid, on this side we have a mixture of air and this vapour so the whole idea here is that here we must have a pressure ejecting pressure, it is actually ejecting pressure so this ejecting pressure must be greater than this vapour pressure of this one.

That means ejection should be done without his condensation so what we do? In this case we can raise the vapour pressure for condensation of this mixture of if had it been just only the water vapour then condensation would have been earlier. So by putting this mixture of air and the vapour we elevate this vapour pressure so ejection of this mixture can be done without having any condensation of this vapour and it is why this is air venting and that is called gas ballast, so that is allowed at the very beginning not to have that kind of contamination problem within this pump.

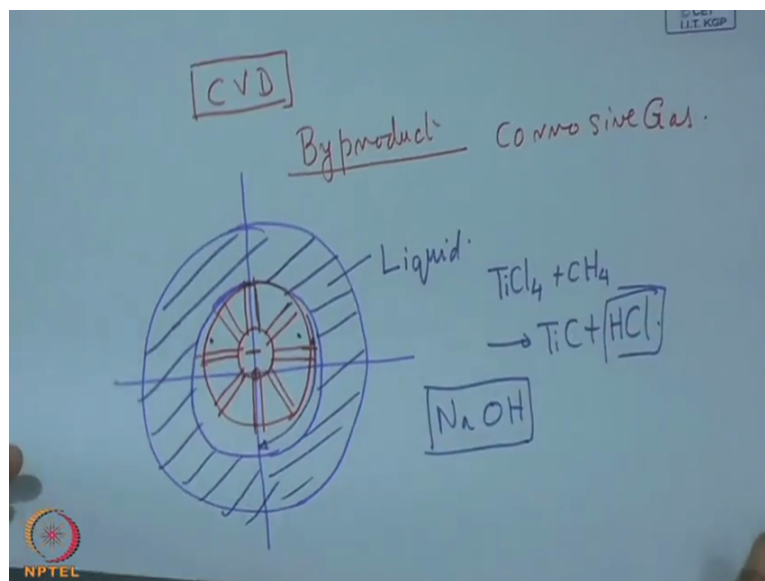
Now what we have here a Liquid ring pump, now this liquid ring pump that is used for handling the corrosive gas in the CVD set mostly, in the CVD we use this liquid ring pump because in the by product of this CVD it can be a corrosive gas and this corrosive gas cannot be just we cannot set it free or it can also create problems for the various parts of the pump so here what is used, we use a water ring pump. This is also the principle is same that means if we have a look re-look in this figure that means what we can see?

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The basic principle is that here because of this eccentricity we have expansion of the volume and then we have contraction of the volume, this principle is also used in water ring pump.

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So here we have the casing and then what we have, so here what we have one impaler and this impaler has this kind of plates and what happens that during the movement of this impaler which is eccentric to the stator so we can see this thing that in this case this impaler what is shown by this stator that is eccentric to the stator and here we have this water ring so that means this is actually the water ring, it can be any liquid ring and that is the border so what happens during this movement of this rotation, this impaler will throw this liquid and by

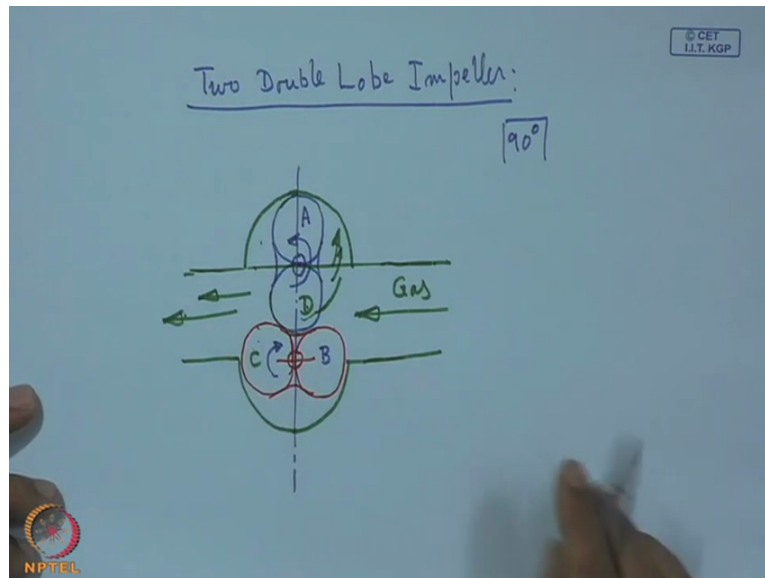
the centrifugal force that will be thrown towards the wall and this will assume form a circle which is parallel to this axis of the state.

Now what is what is going to happen? In this case there will be a between this rotor and this water ring so if we have some kind of admission point from this side and an exit point in this direction what we can see here that the volume is gradually changing and it is actually expanding in this side so there will be a fall of pressure in the CVD chamber is followed by that what is going to happen this material will be squeezed in this side and then because of this squeezing action this material will be compressed and its pressure will exceed the pressure just outside and it will be thrown outside.

So it is in principle having the same practice which is used in vane pumps, only the thing is that instead of metal casing we have one water ring and that wall is found by this ring which is being churned and which is under circulation continuously and because of this eccentricity we have expansion of this space and then there is a contraction of the space so material will be admitted from this side and then it will be delivered on this side.

So the advantage in this case is that this water ring actually protects this wall and at the same time this liquid which is present here that can absorb this corrosive gas say for example, $\text{TiCl}_4 + \text{CH}_4$ that gives us $\text{TiC} + \text{HCl}$ and this HCl will be absorbed by this water and it is having this NaOH solution so with it is possible to have neutralisation of HCl so that is the way this water ring pump that is working. Now comes the Roots pump, this is also the pump which is used for handling very high quantity of liquid or high quantity of gas and it is coupled with one of the vane pump and it can handle high volume of gas from the system. So it is actually Two double lobe impalerthat is used in the system double lobe impaler, now how is the construction let us have a quick look here.

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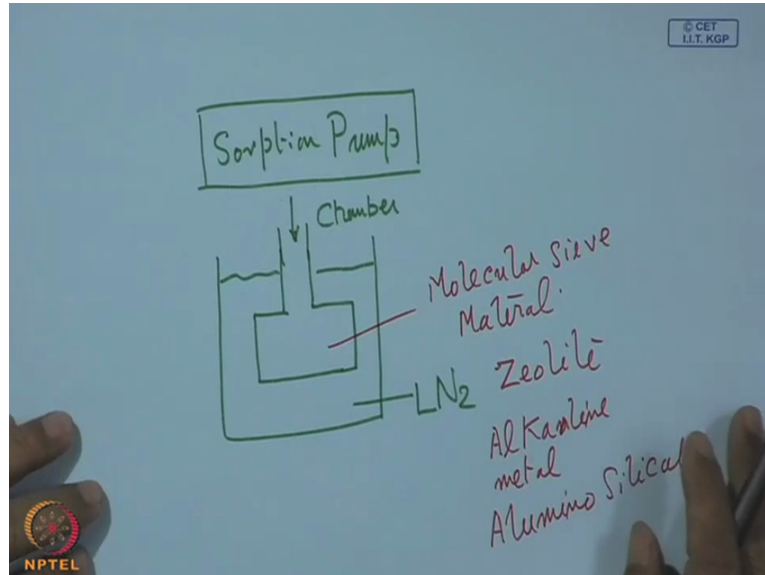
And we have another one so it is a lobe impaler so this is one impaler so that is the axis of rotation and the top one it looks like this so this is the axis of rotation. But they are not in contact and they are having 90 degree phase difference means 90 degree phase angle and this is rotating in this direction say this top impaler A and B is rotating in this direction in the opposite direction, so these are the 2 impalers. Now we can add a few more features here similarly, we can also add this thing so this is incoming and this is outgoing so that is the root pump and they are driven by identical gears so that they are in moving with the same velocity in opposite direction and with a phase angle of 90 degree.

Now what happens, this A now this B that this lobe that it is here and say this is C and here we have A and D. Now what is going to happen at this moment, A and B they can change their position and with this what may happen that this B will come here and then D will take up this position. Now when this D is moving on this side then the air this gas from this side that will be pushed in this direction and that will be dragged down this side and then B is also moving in this direction and finally it will be that B and D this D lobe D and this lobe B they are going to again approach each other because of this rotation so when D will be here, at that point B will assume this position.

So finally when it moves over the surface here from this then B will be here, so finally what it amounts that between this lobe and the lobe from the side that will be some squeezing and the gas which is admitted on this side that will be squeezed between these 2 lobe when they are approaching each other and as a result it will be squeezed and compressed on this side. So

this will be used in series with one vane pump so it will be just before the vane pump and that is a part of the system to have high flow rate of the gas and that can be evacuated at a faster rate from the vacuum system.

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Now we we have seen? We have seen Sorption pump that means so it is actually the body of the pump that will be filled with this molecular sieve material and here what we have this outer casing that is filled with liquid nitrogen and this will be connected to the chamber, so air will be dragged and this is filled with liquid nitrogen and this one this space is filled with molecular sieve material molecular sieve material some Zeolite like material Alkaline, so it is actually some alkaline, metal, Alumino silicate that is used and that can absorb this all those vapours which will be condensed on those surfaces and as a result we can have low value of pressure inside the chamber.

So this sorption pump can be used as one of the substitute for vane pump where we can have some limitation with this contamination with the oil. So these are the pumps, what we can see now in the summary that in the vacuum system we have 2 options; low vacuum or high vacuum but mostly CVD processes it is low vacuum and that can be well handled by this mechanical type pump like root pump, vane pump and water ring pump. So at the very beginning this vane pump is used for the pre-evacuation of the system and when the CVD is ON, it is the water ring pump that is operational and in the process we can also use a Roots pump for increasing the throughput of the system.

And along with that what we have seen that in this case air vent is also used or gas ballast that is used not to allow the condensation unwanted condensation of the vapour which is present in the evacuation chamber and by admitting this air this gas mixture it raises this point the pressure under at which this condensation can take place. But at the same time as an alternative we can have sorption pump, it can be physic option or it can be chemis option and by this process also it is possible to have this Rough evacuation or rough pumping and this can be also used as a substitute for all rotary or moving mechanical pump.