

Fundamentals of Materials Processing-2
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Module – 02
Thin Film Deposition
Lecture – 02
Vacuum Technology

Welcome to lecture 2 of Thin Film Deposition module. In this lecture will learn about vacuum technology. In the previous lecture we had discuss why is vacuum required for thin film deposition, to recap that they were 2 reasons that we discussed, one was that the lambda mean free path or the mean free path is higher if the pressure is lower. So, we create vacuum to lower the pressure such that the mean free path of gas items is larger. Second was to avoid of formation of mono layer of the ambient gas because we are dealing with very pure thin films which will be used is semi conductor devices. So, purity is of essence here, so we want very pure material and we do not want any of the ambient gases to get deposit it on our surface or the substrate during our thin film deposition process.

These 2 things are very important. So, remember sometimes in some of the thin film deposition techniques, we will introduce some gases, we want to do some deposition in the presences of some gases, but first we take out any unwanted gas which is air, nitrogen and oxygen and then we introduced in our deposition system very pure gas that we want. So, vacuum is still very important even if we do not want to do our thin film deposition process under vacuum.

Let us move forward and learn about how we create vacuum.

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Vacuum Technology

Throughput (Q): The quantity of gas (volume of gas at known pressure) that passes through a given plane in known time, Throughput is measured in units of pressure-volume/sec.

$$Q = P \, dV/dt$$

Conductance (C): Conductance is defined as the ability of an object (system) to transport gas between two pressure regimes.

$$C = Q / (P_2 - P_1)$$

for pipes connected in parallel

$$C_{sys} = C_1 + C_2 + C_3 + \dots$$

and for the pipes connected in series

$$\frac{1}{C_{sys}} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3} + \dots$$

To create vacuum remember we have this ideal gas law PV is equal to nRT . So, if the volume of our chamber or the vessel is fixed and temperature is fixed to lower the pressure we need to reduce n , n is a number of atom or number of molecules of the gas. So, we need to take out gas from our chamber to reduce pressure before we go and learn about different types of vacuum pumps and how to create vacuum let me introduced to you some of the definition.

First one of this is throughput Q . So, the throughput is defined as a quantity of gas you can say volume of gas at known pressure that passes through a given plane in known time. So, if I am passing some gas through a plane in known time, how much gas has passed through is the quantity throughput. Now this is we have defined as volume of gas at known pressure why is that? Because gas will occupied any volume that you keep it in right, so volume is not the exact measure or of the quantity of gas volume into pressure being to be is the quantity of gas. So, throughput can be defined as Q is equal to change in volume at a fix pressure or you can also write as constant volume dP over dt this is my throughput.

Now, the second definition is conductance, conductance is defined as the ability of an object or system to transport gas between 2 pressure regimes or 2 pressure regions, suppose I have 2 chambers, 2 chambers or 2 vessels connected through a pipe and one of this is at P_1 and another one is at P_2 then it is very intuitive to know to gas if this pipe in

between has larger diameter than more gas can pass between these 2 vessels to equalize the pressure or because the gas will always flow from higher pressure to lower pressure. So, if P_2 is greater than P_1 then my gases flowing in the left direction in this pipe of conductance C . If the diameter is large the conductance would be higher and more gas can pass through, so conductance is defined as throughput divided by change in pressure.

Now, in a vacuum system, you will always find you have to connect your chamber to a vacuum pump through a pipe through some walls and they will be different conductance for each of the units. So, if all these pipes or the systems are in parallel then the overall conduction of this system will of individual units will add up or if there in series it, it will be the inverse addition of the system. So, we want to have higher conductance so that we can easily pass the gas between 2 systems, the 2 systems we can define as our chamber and the vacuum pump. So, we want to remove the gas from the chamber through the vacuum pump. Our goal should be to increase conductance.

So, while designing a thin film deposition system and the vacuum chamber or vacuum system for thin film deposition, it is always go to avoid a long and narrow pipe. So, all unit should be kept close together such that the pipe lengths are minimized and pipes are broader and of larger diameter. Now to create vacuum we are going to use some kind of vacuum pump vacuum pumps main objective is to remove the gas from the vacuum chamber or deposition chamber the pump would be specified as a pumping speed how much amount of gas, it can remove at what rate.

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Pumping Speed:
Pumping is the action of removing gas molecules from the system through the action of pumps. Pumping speed is defined as

$$S = Q/P$$

Ex. A pipe of conductance C connects a chamber at pressure P to a pump with inlet pressure P_p and intrinsic pumping speed S_p at pump inlet ($S_p = Q/P$), therefore $Q = C(P - P_p)$ and effective pumping speed

$$S = \frac{S_p}{1 + S_p/C}$$

However, real pumps always have some outgassing or system leaks, which can be defined as oppositely directed throughput term Q_p such that

$$Q = S_p P - Q_p = S_p P [1 - (Q_p/S_p P)]$$

When $Q = 0$, the ultimate pressure of the pump P_0 is reached and the effective pumping speed is then

$$S = Q/P = S_p (1 - P_0/P)$$

Which falls to zero as the system pressure approaches ultimate pump pressure P_0

Pumping is the action of removing gas molecules from the system through the action of pumps, pumping speed is defined as the throughput divided by pressure. So, this S would be in volume per second that would be the unit for pumping speed, Q is volume in to pressure.

So, this is an example that a pipe of conductance C connects a chamber at pressure P to a pump with inlet pressure P_p . So, we have a pump we have chamber at pressure P and it is connected to a pump, let me draw the pump like this, this is my vacuum pump and its inlet pressure is P of p , the pressure at the inlet of the pump. So, this creates the pressure difference and pumps objective is to keep this P_p low enough so that gas always flows from chamber to pump and if this is conductance C so, for this whole system, let me write this is pump pressure and pumping speed of the pump. So, the effective pumping speed will be given by this expression remember that we need to increase the conductance, if conductance is high then we can neglect this term in favor of one then this whole pumping speed will become the pumping speed of the pump, pumping speed of the system pumping speed of the pump. So, we should try to maximize the conductance always.

This is for an ideal system, sometimes these pumps also out gas some of the gas comes out from these pumps into the system or sometimes your system vacuum system might has some leak that gases coming out leaking through some walls or somewhere some

joints. So, in that case that leak or the leak rate can be visualized as negative throughput positive throughput is gas coming out of the chamber negative throughput is gas coming in to the chamber. So, this is Q_P which is negative throughput.

This is my positive throughput gas going out of the system this is gas coming in to the system. So, the overall throughput would be Q and then you can define this Q_P as this. Now there some leak then what is the ultimate pressure you can reach by using the pump with the pumping speed of S_P , this expression defines what is the ultimate pressure can be reached. So, the pumping speed is S_P and the pressure of the inlet is P then the ultimate pressure there can be reach this P_0 because as your removing more and more gas from the chamber the pressure of the chamber is going to drop and at some point, they will be no difference left between the chamber pressure and the pressure at the pumping let that would be the ultimate pressure that you can achieve by that pumping system.

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Pumping Time:

The time taken to pump down a system from initial pressure P_i can be calculated noting that throughput may be defined as the time derivative of the product of volume and pressure, i.e.

$$Q = -d(VP)/dt = -VdP/dt,$$

$$\checkmark V \frac{dP(t)}{Dt} = \underline{S_P P} - \underline{Q_P} \checkmark$$

Where Q_P includes pump as well as chamber outgassing. Upon integration we get

$$\checkmark \frac{P(t) - P_0}{P_i - P_0} = \exp\left(-\frac{S_P t}{V}\right)$$

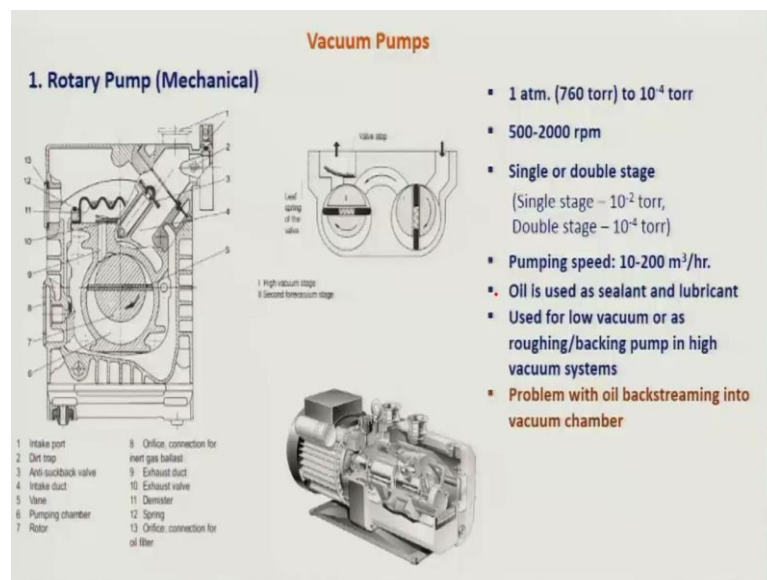
Where $P(t)$ is the pressure of system at time t , V is the volume of system and P_0 is the ultimate pump pressure. Note that the system pressure can never reach below the ultimate pump pressure.

Now, suppose you have chamber which you have connected through some pipes to a vacuum pump and you have some leakage also how much time, it will take to pump it down to a certain level. So, as we had seen, this is change in pressure, pressure is going down that is why we have used negative sign volume is constant DP by DT is the throughput of the pump and this is the throughput of vacuum pump minus leakage if you

solve this, this expression integrate this and so you will get this expression which is given by this.

Where P_0 is the ultimate pressure, p_i is the initial pressure of the vacuum chamber and P is the pressure at time T , you can see this is an exponential function. So, the pressure decreases exponentially. So, very fast in the beginning, but very slow towards the end. So, this is these are the some of the definitions which are used in vacuum technology, it is good to know these because if you are working on thin film deposition you would have to deal with the vacuum pumps once in a while.

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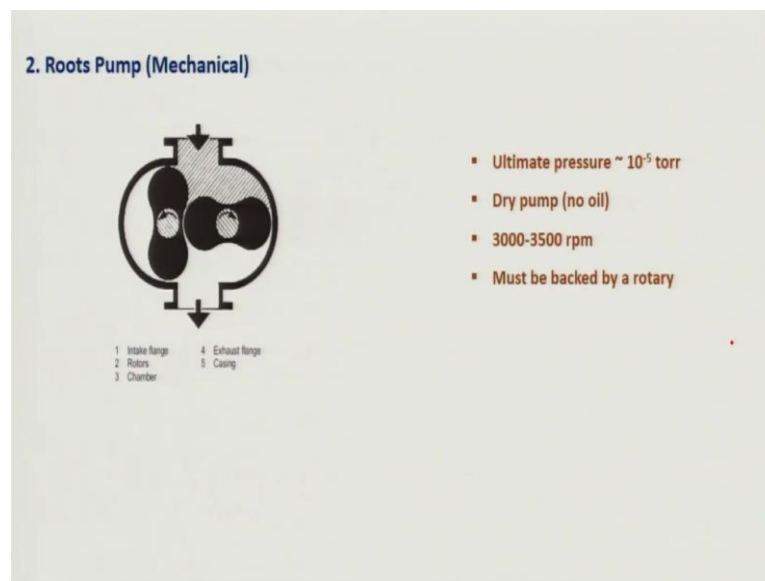
Let us now go and look at various kinds of vacuum pumps. First one is a rotary pump, it is a mechanical pump, the working principle is shown here in this figure, let me briefly explain this, there is a vane, if you see this, this is spring mounted. So, it can contract and enlarge and this is mounted on a drum which is not at the same center as this volume around it. So, when this is moving say in clock wise direction like this, this area will increase because this part is always in touch with this. So, this will increase and this inlet for gas from the vacuum chamber this is my vacuum chamber somewhere here in which I want to create vacuum. So, it draws air or gas from that vacuum chamber it fills this.

Now, as it goes through this, it compresses it and pushes out this way and there is a deiform and it is oil field so that gas can only be pushed out, it cannot come back in. So, this way when this is rotating, it is drawing gas from this vacuum chamber which is

somewhere at the top and it is pushing it out. So, this is the single stage rotary pump and a similar arrangement for dual stage rotary pump. Now this vacuum pumps can reduce pressure up to 10^4 Torr, the speed of rotation for this rotors and the wind is the around 500 to 2000 rpm, single stage can go up to 10^2 Torr, double stage 10^4 Torr pumping speed. Now this is the volume per unit time, it can be 10 to 200 meter cube per hour.

Now, oil is used as sealant and also as lubricant. So, if the oil is used sometimes this oil vapors can back stream inside my system, oil will not allow air to enter, but oil vapor may enter into the system so that is the problem with these kind of pumps and also this can be used as low vacuum pumps, but these are mostly used as roughing or backing pump in high vacuum systems what does this mean will see in later slides.

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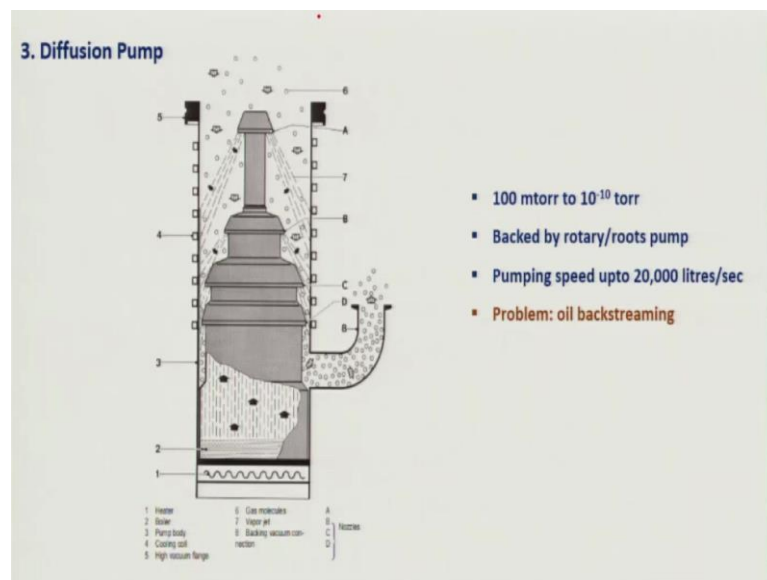


This is my primary pump or rotary pump, an improved version of a mechanical rotary pump is the roots pump. Now roots pump, it uses no oil. So, this no problem of oil back streaming ultimate pressure can be reached 10^5 Torr, it operates as around 3000 to 3500 rpm rotation per minute and some time it is backed by a rotary. Why it is backed by a rotary? Backed means that there is the rotary pump which creates say 10^2 Torr initial low vacuum then this roots pumps takes over why is that? Suppose if you have some dirt particles and in your system which are coming out

with the gas. So, they are handled by rotary pump vary efficiently, but roots pump the way it works it will those dust particles or some larger particles make log roots pump.

So, it is go to have a rotary pump as a backup pump, in roots pump you see they are 2 dumbbell shape rotors which rotate in opposite directions. So, as it is in this position, my vacuum chamber is somewhere here, vacuum chamber it draws air or gas from that and as it is rotating it passes into this side and it pushes out. That is how it creates vacuum in the vacuum chamber. A similar principle, but these are very finely designed or match to pieces such that we have to avoid any dust or particle coming in and getting larger in between, this is about roots pump.

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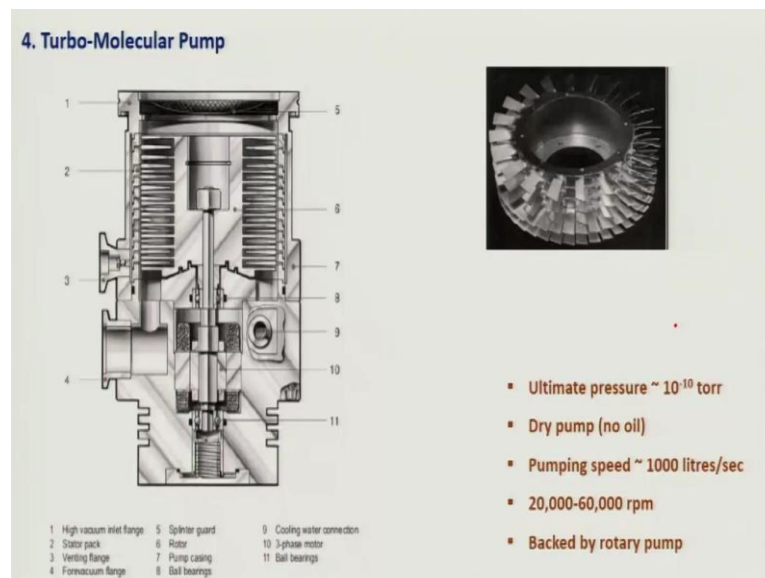


The third type of pump is diffusion pump and the diffusion pump can go up to 10^{-10} Torr, it is backed by rotary or roots pump for the similar reasons that we do not want the initial our good pumps to handle the bulk of the gases. So, first we want to remove the bulk of the gases or which may have some particle metal dust etcetera by roots or rotary pump than we pump switch on the diffusion pump so that the load on this diffusion pump is less.

Pumping speeds can be very high up to 20000 liters per second and again so, these have a problem of oil back streaming, by the way the diffusion pumps work is that this oil which you heat and you pass upward through a specially design nozzles and these oil vapors come out as jet streams, when the oil vapors are coming out this atoms are

molecules of oil will impact the gas items and by momentum transfer because they are have a direction downward direction they will give the momentum to these in the downward direction. So, by just momentum transfer these jets are very high speed jets of oil they will push the gas items down which are collected here and then driven out by a rotary or roots pump, oil is cool down and recollected and heat it up again. So, this is how a diffusion pump works, but because of oil back streaming these are not use in semiconductor industry they are still some use and some of the lab equipment you will find working with the diffusion pumps, but most of the semiconductor industry has switched on to what is known as turbo molecular pump.

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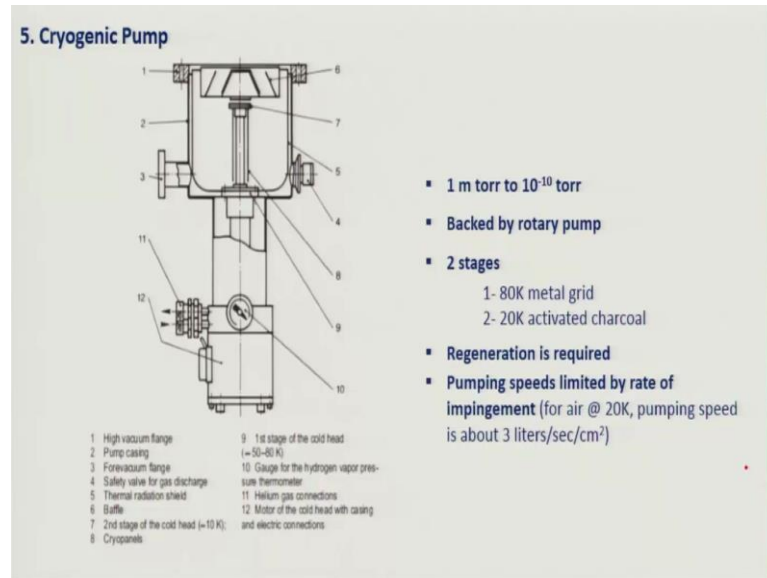


Turbo molecular pump have a similar principle of momentum transfer as diffusion pump, but rather than using oil or oil jets for momentum transfer these turbo molecular pump use a specially design rotors the design of the rotors is shown in this figure here and you can see that this rotor has many blades which are inclined in a certain direction.

When this rotor is rotating a very high speed these speeds could be 20 to 60000 rotations per minute, they impact the gas items and because of this downward slope in the design they give a negative or downward momentum transfer to gas items and they each subsequent rotors keep pushing this gas items or molecules downwards which is again taken out by a rotary or roots pump. These are dry pump so no oil back streaming, so your thin film remains very pure ultimate pressure can be reach which is 10 to power

minus 10 Torr, pumping speeds are straightly lower compare to diffusion pump, but still very good 1000 liters per second. So, these are used very extensively this turbo molecular pumps.

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The next category of pumps which is also used very extensively is called cryogenic pumps.

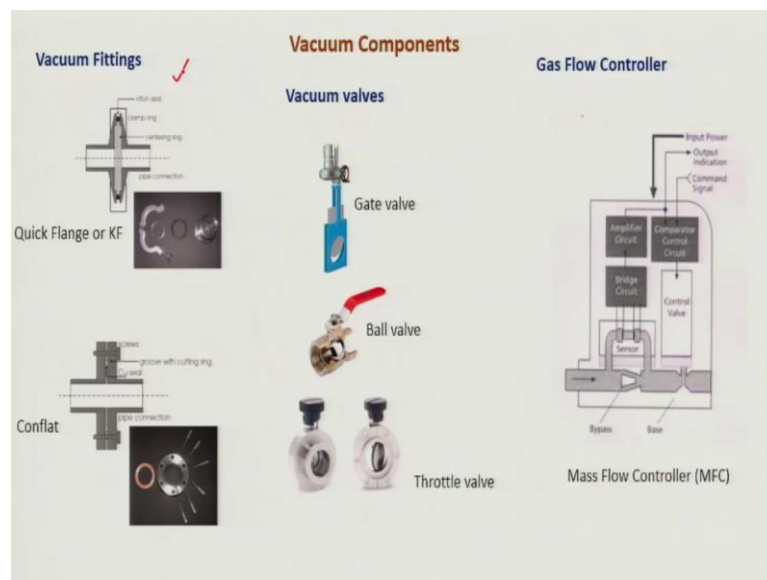
Now, the cryogenic pumps are different principles you know that gas tends to get condense on any cold surface. So, if you introduce a cold surface in your vacuum chamber all the gas atoms and molecules will go and get attach to it once they get items or molecules get attach to that are cold surface, less atoms of gas are available in the chamber so reduce pressure. So, curve pumps work on the similar principle that they have first of all there also root back by rotary pump ultimate pressure can be 10 to power minus 10 Torr, most of the curve pumps have 2 stages, one is the metal grid which is capped 80 Kelvin and this a second level which is activated charcoal which is capped at 20 Kelvin. These are very low temperatures and these are inside this cryo pump. So, as gas items and molecules come they get trapped there at these low temperatures and they cannot go back in the system.

So, you can keep trapping more and more gas atoms and molecule and create vacuum in your vacuum chamber; however, from time to time you have to take out these pump heat them up so that all the gas which is absorbed on metal grid and activated charcoal is

dissolved and goes to atmosphere so that you can reuse this pump again for vacuum creation.

Regeneration is required and pumping speeds is limited by rate of impingement, remembered the rate of impingement we had discuss if you introduce surface in gaseous environment what would be the rate of impingement. So, it is limited by only that. There are several mode pumps, but these cover most of the pumps which are used labs as well as in production level industries. Our part from pumps, there are several other components which are part of the vacuum system like vacuum fittings, they should be leak proof.

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They are different type of vacuum fittings, quick flange or conflate vacuum valves where you can control the pressure by other nomadically or computer controlled or manually or you can close a valve or open a valve or also sometimes when you have to flow a gas into the system a pure gas then you can use of mass flow controller to introduce the gas at a control trite. These are all part of the vacuum system.

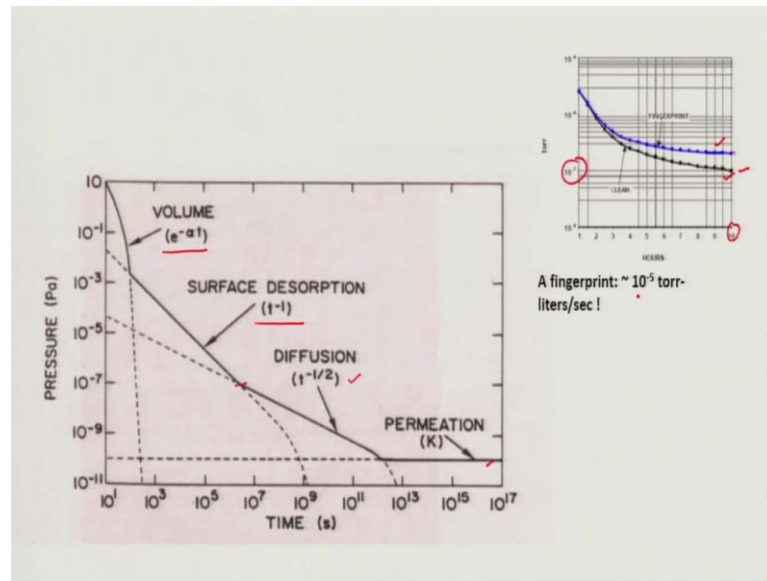
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Vacuum (Pressure) Measurement	
▪ Thermocouple Gauge	10 torr \rightarrow 10^{-3} torr
▪ Heat conduction gauge ("Pirani")	1 torr \rightarrow 10^{-4} torr
▪ Baratron-gauge	10^4 torr \rightarrow 10^{-5} torr *
▪ Gas discharge manometer ("Penning")	10^{-3} torr \rightarrow 10^{-13} torr
▪ Ionization manometer	10^{-1} torr \rightarrow 10^{-11} torr
Residual Gas Analysis (RGA) for partial pressure of different gases	

Once you create vacuum, you also need to have some kind of measurement, right. So, they are different pressure gauges which you can use in different pressure regimes like you can use thermocouple gauge or heat conduction gauge in the range of 10 to power minus 4, 10 to power minus 3, 10 to power minus 4 Torr. These work on the principle of that if you have a heated element and more gas in a higher pressure more gas will impinge on it and it take away more heat from this heated element.

Either you can measure the temperature or you can measure the current to keep the temperature same to adjust to know how much pressure is reached. Baratron gauges are capacitance manometer, it works on the principle of capacitance gas discharge and ionization manometer they work on the principle of how much gases being ionized. So, you take a small sample of gas small volume and see how much of gas can be ionized and you count the number of electrons generated if less gas, less gas is ionized, less electrons and you can use in this in low in high vacuum regions. So, now, you have almost all the components of a vacuum system.

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How does vacuum process works with time? Remember when we have looked at the pumping time, we have said this has exponential relation with time, some kind of exponential relation this is called volume pumping. So, in this stage you are taking out the volume of the gas.

Now, second slope is a surface desorption, remember there is some gas which is always absorbed on the surface of your vacuum chamber. So, once you reach the certain pressure, this gas will start to desorb then your pressure keeps going down at certain point the diffusion process will take over. So, this diffusion is gas diffusion through some joints somewhere in any curved surfaces. So, any type gases will diffuse out and the limitation here is permeation, permeation means gas coming inside the vacuum chamber from outside through permeation, through the chamber wall, it could happened and that rates is around 10 to power minus 10 Pascal.

To go below this you need to have very special design material for the chamber to stop permeation at this level and takes very long time. One more point before we close our topic of vacuum technology see this figure here it shows that is the clean and there is a fingerprint curve this is the pumping curve and the pressure in Torr. If you see that with cleaned you in 10 hours, you get to 10 to power minus 2 Torr, I am not sure, but at this level. So, this is not visible here nor cleared, but you reach this level, but if you have a fingerprint inside the chamber wall then you are not able to read this level this vacuum

level which means that a finger print has to be avoided, why? We always have some oil or some kind of moisture or something in our hands. So, if you are handling our vacuum system vacuum chamber specially, the inside of vacuum chamber by bear hands, we leave a fingerprint which will slowly out gas and create this leak and we will not be able to reach the final pressure that we want or the vacuum level that we want. So, it is always good to use gloves while handling vacuum systems and vacuum chambers.

With this, I will stop our discussion on vacuum technology. In the next lecture, we will start discussing about thin film deposition methods.

Thank you.