Chemistry and Physics of Surfaces and Interfaces
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Lecture - 06
Introduction to Ultra-High Vacuum and Preparation of Clean Surfaces

Hello everyone, welcome back to lecture number 6. In today's lecture what we are going to discuss is about the Type of Interfaces.

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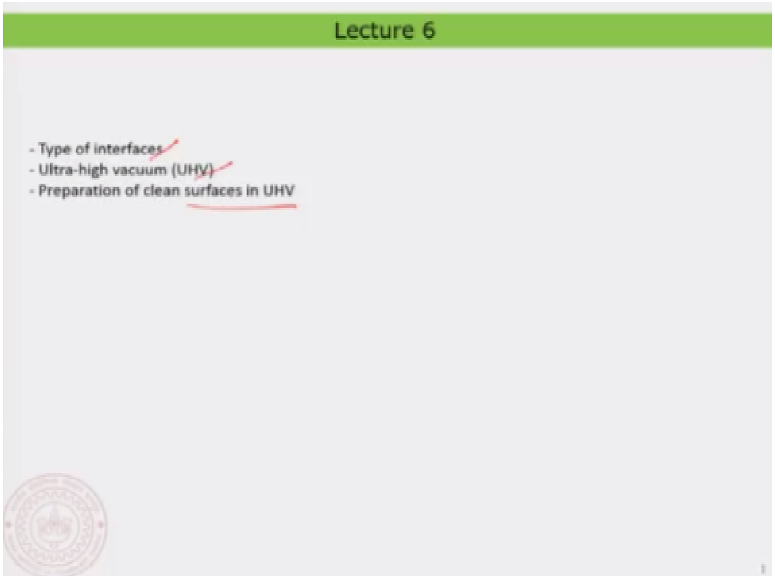

We had a quick look at that in the previous lecture and the most important thing that we are going to learn today is the Ultra-high Vacuum technology itself, so how to obtain the Ultra-high Vacuum technology and a very brief introduction to how to generate basically an Ultra-high Vacuum technology because that is an extremely vital point in our discussion, because we are going to work out on crystalline surfaces and all the surfaces that we are going to study in this lecture will actually be studied inside an Ultra-high Vacuum chamber, where the base pressure is extremely clean or base pressure is extremely low. Therefore, the atmosphere inside the Ultra- high Vacuum chamber is very, very clean. So, then we therefore also would look at the preparation of a generic preparation methodologies for cleaning the surfaces to obtain atomically flat surfaces. Because we are going to work with the atomically flat surfaces further as you would see in the examples. Good we have already seen that the typical type of interfaces. For example, you have solid vacuum interface, solid liquid interface or stronger solid-solid interface for example.

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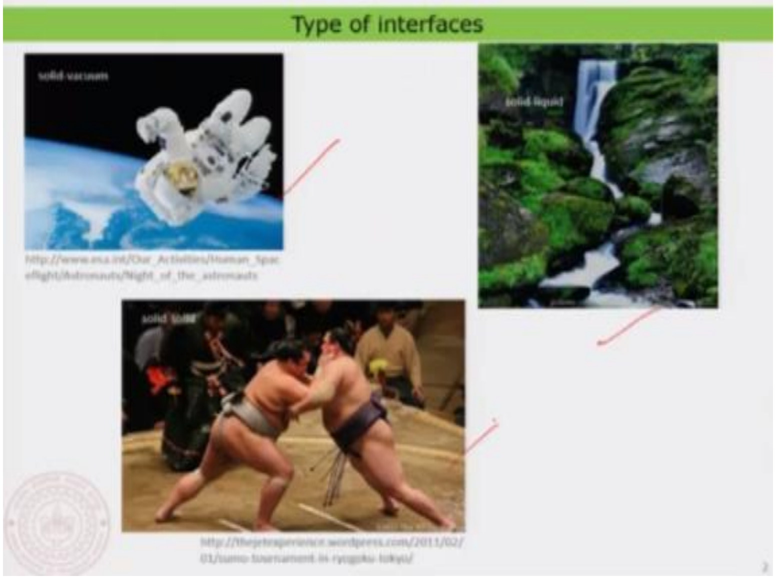

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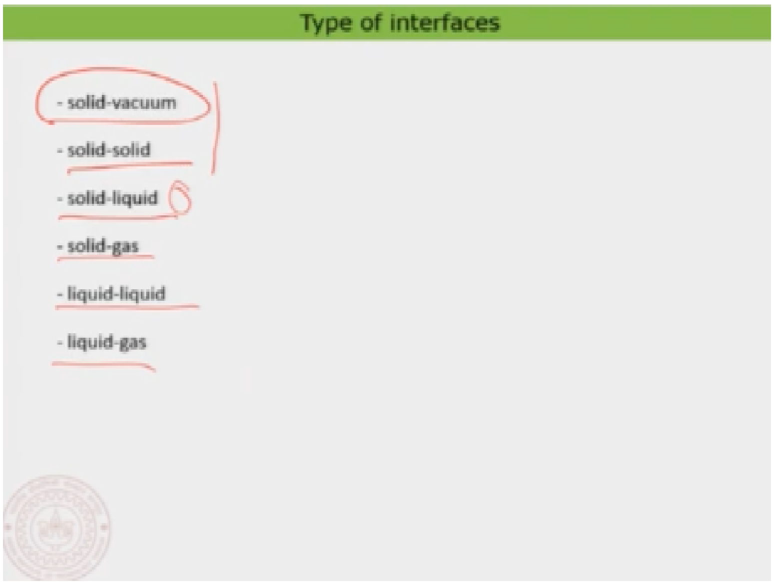

So, if you want to list basically the different type of interfaces the most fundamental interface that you would talk and or you would name is, basically the solid-vacuum interface, this is indeed the most cleanest and the most ideal surface that you can produce, and that is actually the reason why we would also study the Ultra-high Vacuum technology itself because with that you can create the solid vacuum interface and then you can create solid-solid interface, solid- liquid interface, solid-gas interface. Solid-gas interface is also something that we always encounter in our life, because we always are in touch with gas molecules then, liquid-liquid interface, liquid-gas interface you can actually just name enormous possible interfaces that you think about. And in our lecture, we majorly discuss about the solid vacuum and solid-solid and in a very few examples we would also have a look at this solid-liquid interface.

Well, you can see this is extremely large and wider topic that you can cover through these systems. So therefore, we would be like mostly concentrating on the solid-solid interface because that is something that you would require for generating thin films, which is the fundament for the technology that we have already discussed in the previous classes. Good so solid-vacuum interface. So, when you talk about solid-vacuum interfaces either you take your solid to outer space as you have seen in the photograph that I have showed you in the beginning, or else you need to prepare a solid-vacuum interface in laboratory, then you need to have something called an Ultra High Vacuum chamber.

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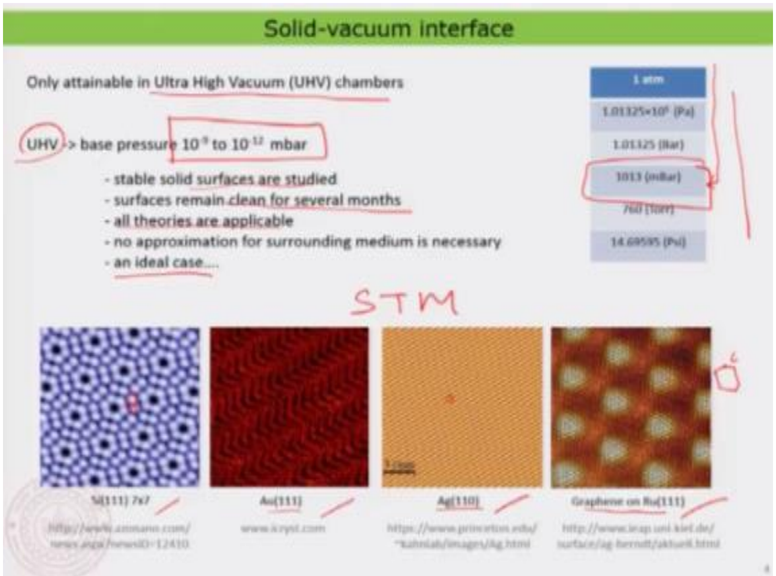

So, we will have a look at the Ultra High Vacuum chamber, but before that there are some important parameters that we need to get used to understanding the Ultra High Vacuum chamber itself. So, what is an Ultra High Vacuum chamber? Vacuum you have already heard about it is just the amount of gas molecules in the chamber or in the space when it getting reduced is actually something you call it as vacuum. So, for example the atmospheric pressure or here we have about atmospheric pressure of gas molecule present, so this you would call it as a standard normal situation. And now if I start to reduce the amount of molecules or atoms gas molecules from this room, then I can basically create vacuum. But in the Ultra High Vacuum so the pressure in the chamber that we are talking about is extremely low. It is actually in the order of 10 raised to minus 9 to 10 raised to minus 12 millibar. Let us have a look at a comparison to kind of understand what is a relation between atmospheric pressure units and also the millibar unit. So, you can see here in this table one atmosphere actually corresponds to something like 1013 millibar. So now you can imagine 10 raise to minus 12 millibar, means how low it is, it is actually quite close to the pressure of the outer space. So that is also why we have special situation in vacuum so we need actually those kind of surfaces to situation to clearly understand the surfaces. So, this is the typical unit and most of the time you might actually encounter with this unit and this is the unit that I am going to use most of the time which is actually the millibar. But you can express it in any a different unit and that actually is given by this table here. So, what is the UHV go to offer you? It is very clean that is the most important thing, so now just for an analogy if you would take basically a clean surface. Let us imagine that you clean a copper surface or a gold surface or whatsoever, within a few millisecond the entire surface is going to covered with gas molecules that is present in this atmosphere. So that means no matter what you cannot prepare or you cannot have a clean surface in these ambient conditions or in this kind of room situation. That is the reason why you need to reduce the pressure and if you reduce the pressure to something like, an Ultra High Vacuum chamber, ultra-high volume condition then you can basically keep the surfaces clean for several months. That is the interesting thing, but of course reactivity also matters. Metals like silver, platinum, gold can actually be kept for a long time even without any cleaning, so they will remain clean. But materials like more reactive surfaces like tungsten or rhodium, ruthenium these kinds of surfaces are much more reactive or iron for example more reactive surfaces and those surfaces are actually still be maintained, for a few days clean in the Ultra High Vacuum chambers. But now having it very clean the interesting thing is that, you can now study the surface in an ideal situation. Now, I have the surface which is not making any interface with other material, so that means I can study an absolutely clean surface and that is the importance here. So, it is a stable solid surface can be studied and therefore all the theoretical calculations can be applied in that case because you do not have any strange material that is interfering with your surface or you do not have anything else interfering with your surface. So that is why it is actually called an ideal situation which is actually the solid surface a solid vacuum interface and that is the first interface that we are going to basically just look at. And then we use that solid vacuum interface to deposit at so big molecules that means atoms or molecules and even bigger molecules, we will try to absorb them on the surface and then we will basically make thin films and also study their properties, so that is the aim. Now this is the good thing about working in Ultra High Vacuum condition, you can see like I can now nicely image clean surfaces. So, you have here this go silicon 111, 7 by 7 reconstruction gold 111 surface, silver 110 surface.

Graphene on ruthenium this is also some interesting surface we will come back to that in in after some lectures. You can actually prepare all of them and you can now see that each of these dots are nothing but silicon atom or this line what you are seeing as you remember in the previous case, it is nothing but a chain of gold atoms. So here I have basically a silver atom sitting or here in these hexagons you have basically here actually like the fundamental building block is a hexagon and each corner of the hexagon is actually a carbon atom for example. So now you can image the surface as clean, as you see here in these images and these are all scanning tunnelling micrographs which is actually a micrographic technique which can actually give you atomic resolution. So that is now the interesting thing. If you have clean vacuum chamber you can basically image surfaces very clean and therefore you have an ideal situation to study the surfaces. So, that is the point.

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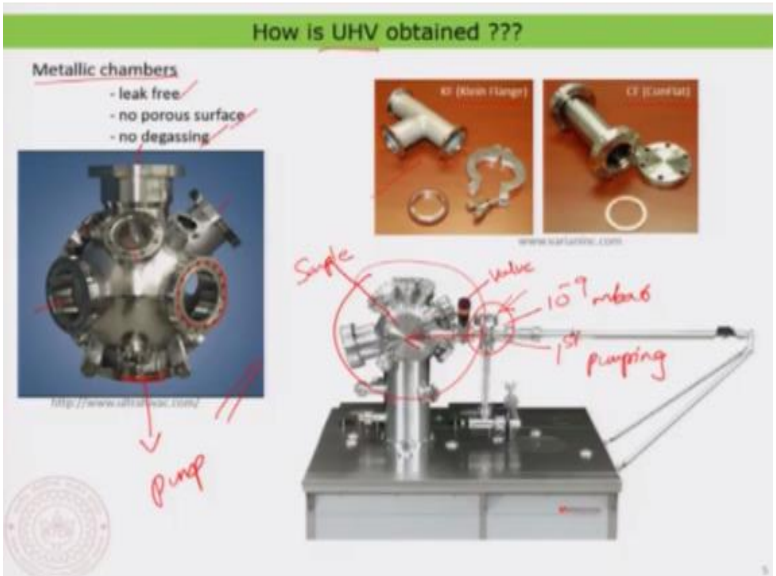

Now how do we obtain ultra-high vacuum? So, that is the question, of course you would say like we need to pump. But before pumping can I create just ultra high Vacuum in this room? That is a question. Well, the answer is no absolutely no because if I try to evacuate or pump this room there is extremely many number of leaks at the interfaces of this room, and that allows me never to obtain a high vacuum or even normal vacuums in this room. Therefore, what is very very important is actually to have a metallic chamber of this type as you see here this is a type of metallic chamber it looks like, but the most important thing is that the chamber should be leak free. So, that means it should be a single unit with whatever features that you want to have it on, and then the surface the internal surface from where you create the vacuum should be absolutely non porous. If you have porous materials that are used, then of course you can actually have gas molecules getting trapped and then you are never going to attain ultra high vacuum. And then the materials that you use should not degass, degass actually means that it should not evaporate anything to the vacuum itself. So, that means not all material can be used but in interesting thing is you can use it stainless steel. Stainless steel extremely polished surface can actually be used for making the Ultra -High vacuum chamber. And once you have this kind of chamber you can basically close these chambers using this different type of seals that is actually what you see here is actually something called a flange. And the flange can actually be sealed using a KF type of flange, which is a Klein Flange or a CF Type of flange, that's actually a cone flat type of flange. So, these are the different type of flange that you can use, actually to close the chamber and once you close the chamber then you can ideally pump the chamber and then you can obtain the High Vacuum inside the chamber. So, when you work you basically just close all these valves with the CF flanges and then you ideally pump through the bottom of the chamber. So, this actually this part goes to the pump and you can basically obtain the vacuum. So, now this is a very simple example of a simple Ultra High Vacuum chamber which is having a very big chamber here which is actually then connected to something called an entry lock, through which you actually put insert your samples to the Ultra High Vacuum chamber. So, you insert your samples here first, and then after inserting the sample you pump this region first. First pumping and then after you obtain the pressure here in the order of 10 raised to minus 9 millibar, you would move your sample to the main chamber by opening this valve. So, this is basically a valve so you can now move the sample and finally your sample will actually be inside this chamber here. So, sample will come here and then once you have the sample there you can actually do all the studies. That you want for example you can do microscopy, you can do spectroscopy, you can do desorption analysis, you can do many things and everything will be done in kind of a similar chamber. So, that is the key element in our in our course, for example is the vacuum chamber, it is an Ultra High Vacuum chamber.

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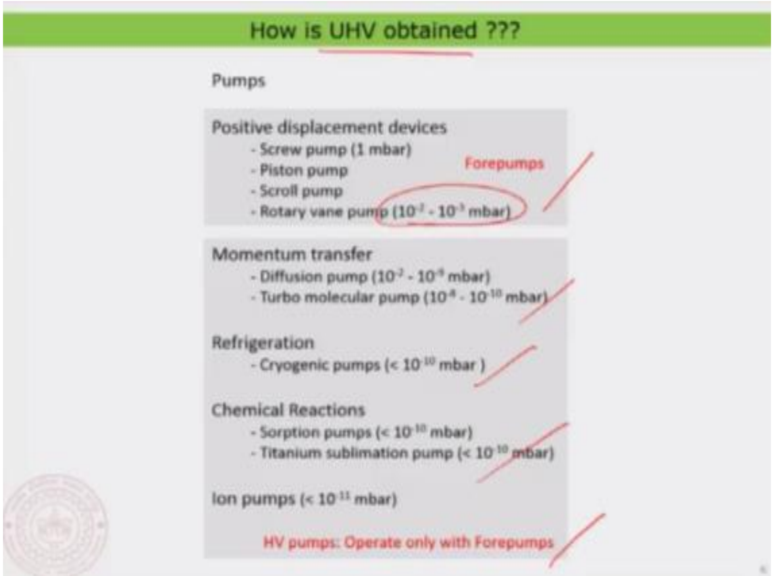

Now how to obtain pump, the Ultra High Vacuum is very very simple, you have to pump as I told you so. Now, we know how the chamber should look like in the type of the chamber. Once you have the chamber you can basically pump the system. But pumping there are two stages one is actually called as a Forepump, and then we actually have got the High Vacuum pump. So, the Forepumps are very simple. So, they are actually just displacement devices, it is like kind of a Screw pump or a Piston pump, Scroll Pump or Rotary vane pump. This is something you basically come across in kind of every day's life, but once you actually just pump the chamber with this pre vacuum and then you basically generate a small Vacuum inside about 10 raised to minus 2 to minus 3 millibar and once you obtain this so called pre vacuum. Then, you would basically go to the real high vacuum pumps which are actually different type something which is using the Momentum transfer or like refrigeration or even Chemical Reactions are used for pumping the chemicals inside the chamber. So mainly we are going to look into the High Vacuum Pumps only, the pre vacuum pumps you might have already seen in your daily life.

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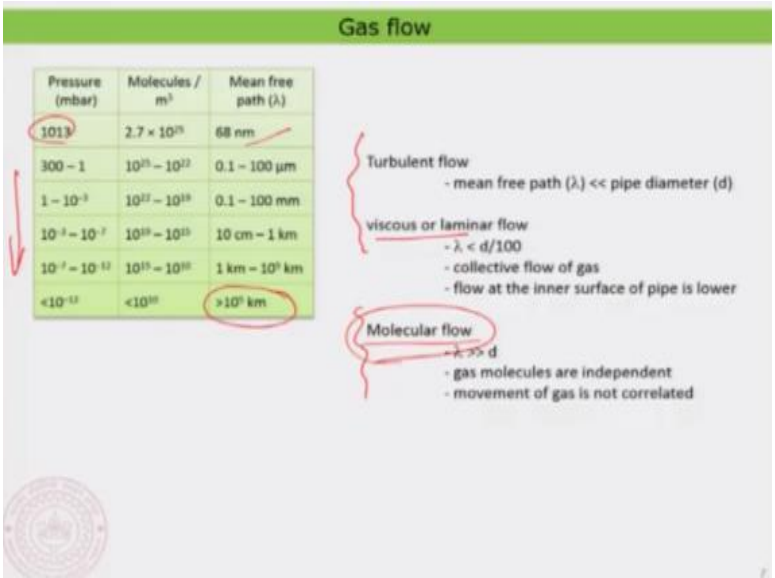

So have a look. Before that I just want to tell one important thing is that, at different pressure the point is, the molecules that are present in the chamber are going to have a different type of flow. So, for example if in the atmospheric pressure the main free paths between adjacent molecules are actually in the order of 70 nanometre but as you decrease the pressure, then what happens actually that at very very large or very high vacuum you see the main free path with them extremely long in the order of thousands of kilometre for example. Now the problem is actually while you do the pumping you are not going to see more molecules coming together. So, you have to efficiently pump either increase the efficiency of the pumping or you basically wait long until you capture a molecule. And now the interesting thing to note here is actually in the room temperature regime you typically have the turbulent flow, or a viscous type of flow known as actually a laminar flow of the gas. Therefore, you can basically create vacuum by pumping molecules continuously. But when it come to something like very, very low vacuum, so the problem is actually the molecules are going to do something called a molecular flow where every molecule is actually independent of the other molecule, because molecules are far away from each other and therefore they do not communicate to each other. So, if you pump one molecule the other molecule does not care. But in the laminar flow or in the viscous region if you pump one molecule the other molecules start to flow along with that so it is easy to pump. So, therefore this need, to be considered while you make the pump in the High Vacuum regime.

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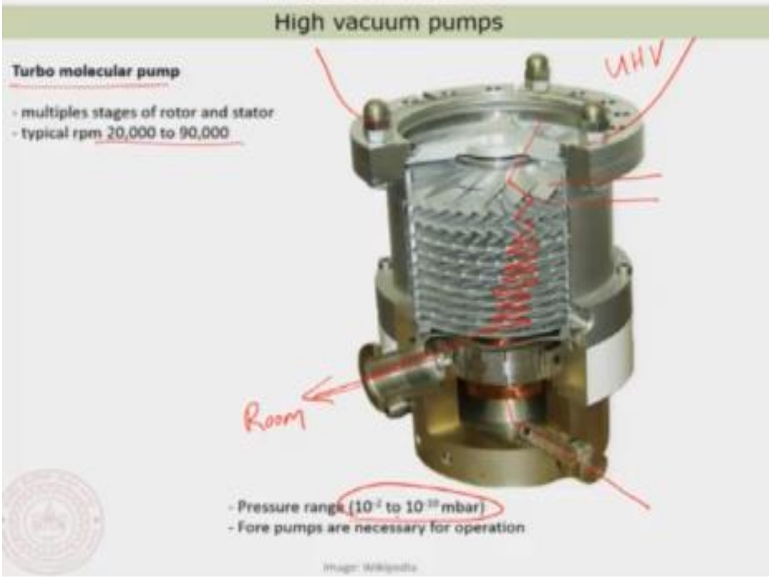

Now this is the most important and the crucial pumping the High Vacuum pumping which is actually called as a Turbo molecular pump. As the name suggests it is actually a Turbo pump that actually means it is something which is working at a very very high rpm, you can see it is about 90 kilohertz. That is a speed that it can reach and it is also a molecular pump that means you are pumping molecule by molecule away from the chamber and so this is basically the part where the chamber is connected, so this is the UHV part and then the pump is actually finally connected to room along this part here. So, ideally what you are doing is you have kind of different stages of rotors and stators connected together and they actually move at a very large speed and the molecule that actually get into this one get deflected through the pump and then, they finally just come down to the bottom where the pressure is already high and then finally it is actually thrown out of the chamber that is the way you do the pumping. Well, you can actually attain a vacuum of about 10 raise to minus 10 millibar with this Turbo molecular pump.

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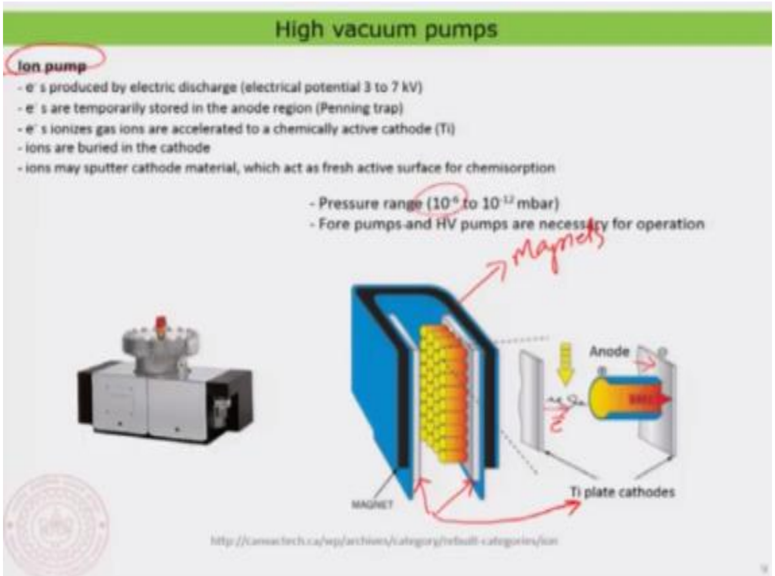

Now then the next one is actually pumps that are based on chemical reaction that is very interesting, what you do so this is actually called as an Ion pump. In the Ion pump as the name suggests you have to create ions of the molecule. So, what you do is let's say this are actually two cathodes which is actually nothing but titanium plates that means very reactive material and what you ideally do is you create an electron flow from this, electrons are flowing and these electrons are actually flowing in a magnetic field as you can see magnets are actually used here magnets. And the magnets would actually just make the electron to flow like this along the other direction. And then when it flows on its flow it actually start to ionize the gas molecule present in the chamber. Once the gas molecules are ionized those gas molecules will be trapped into again this cathode. Because the cathode is actually again having a negative charge that means the positively ionized molecules would be easily accelerated to the cathode and the molecules are actually finally getting trapped onto the cathode. So, you ideally just accumulate the molecule that is present in the chamber. But the important thing is that this kind of pumping cannot be applied if the pressure is actually about 10 raised to minus 6. So, that means these pumps are strictly only working when the pressure is very very low. So that is the most important thing.

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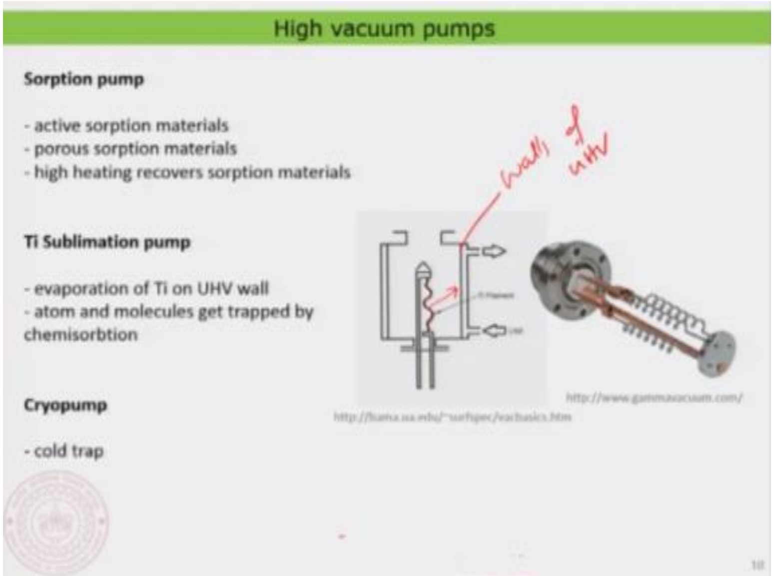

Then, you can use actually different type of pumping simple you can actually use Sorption materials like porous materials which can actually trap molecules or you can actually use something called a Titanium sublimation pump where again you use some chemical reactions. Ideally what you do is this is a titanium filament and you evaporate this titanium filament onto the UHV chamber. So, this is basically the walls of UHV chamber and you evaporate them. And once you get a fresh layer of titanium, the gas molecules that are flowing inside the chamber would actually stick to the chamber. So that is how you basically let them react or you can even use like cold fingers where molecules would also get trapped due to the temperature difference. Then well these pumps are all can actually be used at the very very low vacuum systems.

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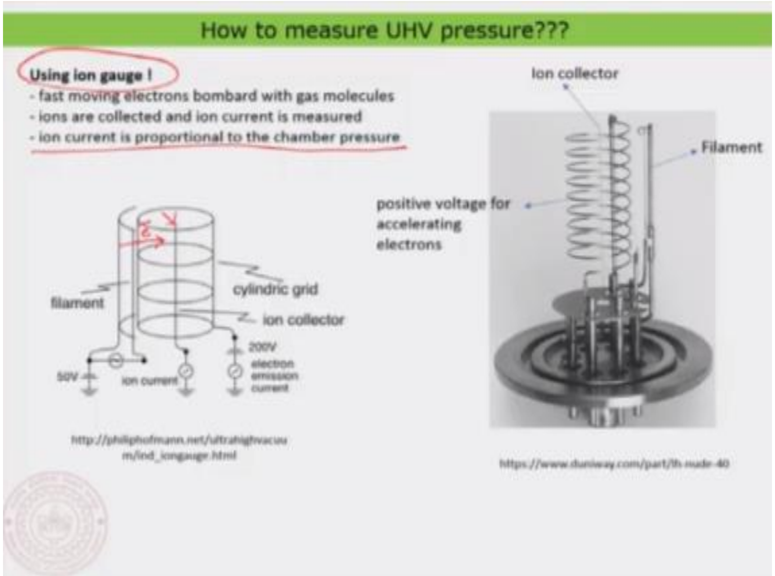

Now, how do we measure the Ultra High Vacuum pressure? So, this is actually measured using again an ion gauge, as I have just told you in the case of Ion pump you create basically ions inside the chamber. So, similarly you have a unit here where you have an electron which is where you have you generate electrons and these electrons that bombard with the ions inside the chamber and those ions will be actually collected on to this particular grid which is an ion collector and the ion current is directly proportional to the pressure in the chamber. So, the ion current is basically proportional to the pressure in the chamber. And by measuring the ion current you can ideally create or you can ideally measure the pressure in the chamber. So, this is typically the major components of an ultra-high vacuum chamber. So, we are not going to revisit the technical part of it but now onwards you believe that we have an Ultra High vacuum chamber with us and we are doing everything in this Ultra High vacuum chamber.

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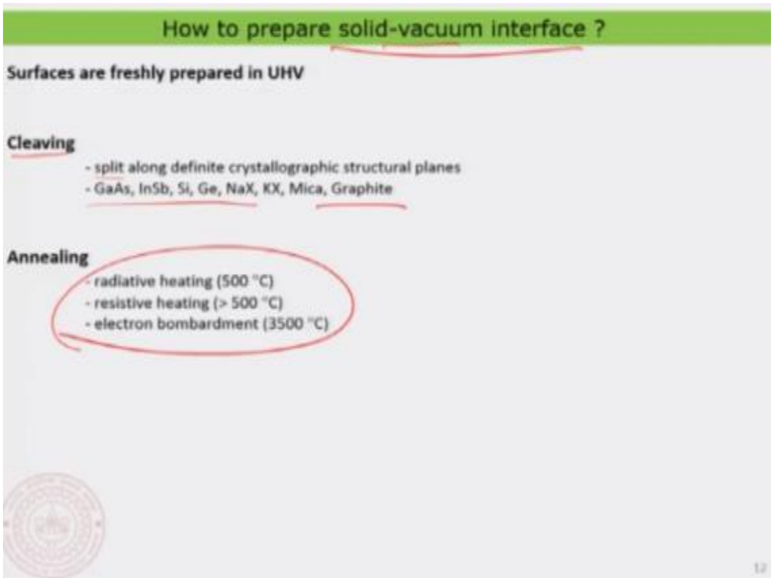

Now that is the point where we can actually now think about creating clean surfaces inside the solid vacuum interface or inside an ultra high vacuum chamber. So, ideally that means it is a solid vacuum interface that you are going to create. So, what are the typical ways of preparing materials? The materials can actually be prepared by different methods. So, simple methods are actually called as Cleaving. What you do is you have your material and then use some adhering material and stick on that and you can basically cleave material. For example, graphite, crystalline graphite can actually be cleaved even using scotch tape it is very simple. So, you take a piece of graphite and along the basal plane you stick the cello tape and then you actually remove the cello tape and you actually just make a fresh surface. So, this is very very simple and of course it is actually becoming difficult for different materials but you can basically do this cleaving method which is basically cracking the materials into two pieces using along a certain crystallographic direction. Then, you can also use simple Annealing technique. You just simply heat the material and once you heat the surface layer would basically evaporate and once you evaporate the surface layer the surface atoms would also diffuse and then make a clean atomically flat surface.

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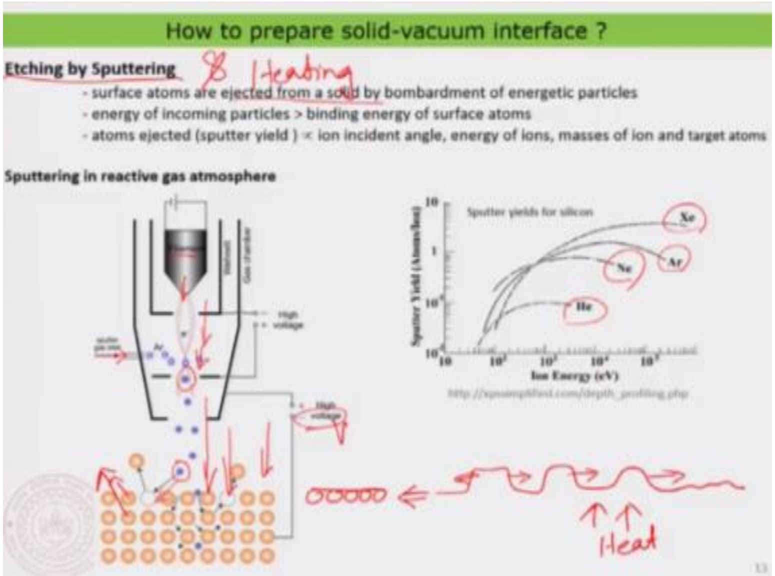

Now, the most important method that people also use is actually something called as an Etching by Sputtering and heating, a combination of both Sputtering and heating. What do you do by sputtering? Sputtering is very simple as the name suggest you have basically a molecule beam of ionized atoms, that are actually coming towards the material and then you basically just bombard the material with the fast moving ions and then using that impact energy you remove the atoms from the surface, so that is the basic key of it. So how does it work? We will understand it using a schematic diagram of a sputtering gun it is known as. So, what it has is actually having a filament, so that is the most important part. So, you have a filament and this filament will basically just create electron just by thermionic emission. So, electrons are actually just coming and once the electrons are actually moving along this path now what you do is that you basically just inject a gas which is usually some kind of a noble gas that you use like argon, helium or xenon and things like that. You inject gas molecules which are inert, and then once this gas molecule meets the electron as you can see here the electron ionize, the gas molecule. Once the gas molecules are ionized then the ionized atoms are basically now being accelerated towards the surface using a negative voltage. Now this negative voltage is very important, so you can basically increase the negative voltage and also decrease the negative voltage in order to basically increase the acceleration of the fast moving ions and once you move the ions they would basically come and impact the surface in with a certain energy, and that energy can be controlled by this voltage. And now you can see when the atoms are actually, the ionized atoms are impacting the surface atom you can basically eject or you can remove the surface atoms from the surface itself or surface or even from the deep. It is not necessary that you always remove the surface atom you can also remove actually the bulk atoms. But not typically very deep in the bulk typically in the around the surface that you remove the atoms. And once you remove this atom you would basically get surfaces that are kind of atomically corrugated like this. And now these surfaces what you do is you heat the surface at very high temperature and once you heat the surface then what happens is basically that these little corrugations that you see which are again atomic corrugation they start to flow on the surface and finally this is leading to a flat atomically flat surface where you can actually have the atoms arranged in this way. So, this is of course an ideal situation, that I have showed you but if you would take for example copper this is always contaminated with oxide, for example. So, the layer of oxide can actually be removed and after removing the oxide you can clean the surface like this. And then Annealing together would give me some kind of a; so now you can basically just create this and then now what I can do is I can also just plot the Sputtering yield as a function of the ion energy. And what you basically see is that the I the energy and the yield is basically related to the ionic energy, as well as the energy is dependent on the type of the type of gas that you use for the sputtering. So, it depends on what you want to clean basically if you have an extremely dirty surface you would surely want to clean the surface using a heavier noble gas and if the surface is moderately dirty then you would use basically healing. This is the typical method that you use for cleaning the surface and with this you can actually just now create an atomically flat surface as I have just shown you here in this case. Well, with that I am concluding this lecture and in the next lecture we are going to look about absorption of atoms onto the different clean surfaces, thank you very much.