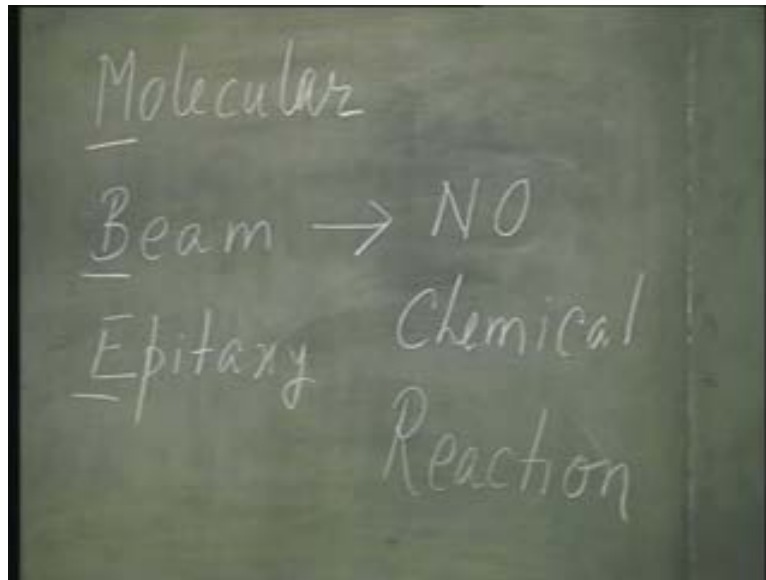


VLSI Technology
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Lecture - 10
Molecular Beam Epitaxy

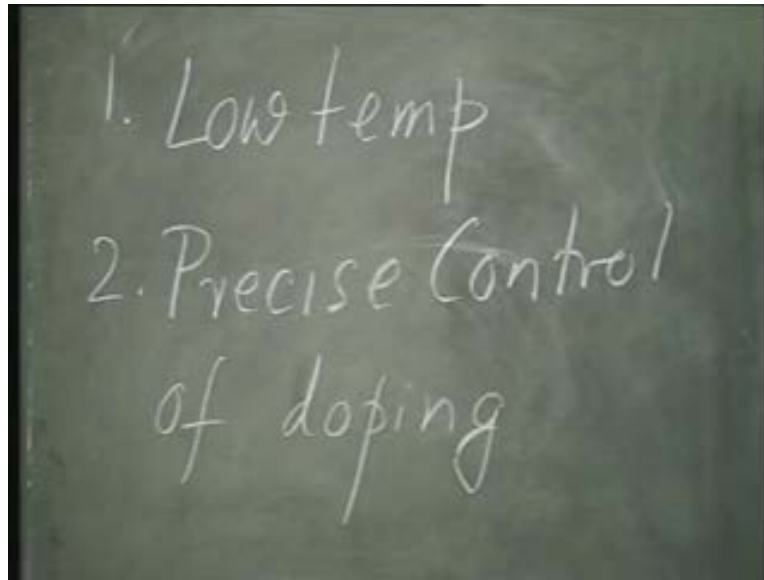
We have classified the epitaxy system or the epitaxy process in three groups, liquid phase epitaxy, vapour phase epitaxy and molecular beam epitaxy and I have already told you that for silicon, it is usually vapour phase epitaxy. That is what we have discussed so far and now I want to discuss the third group of epitaxy system that is the molecular beam epitaxy or in short it is called MBE, MBE or molecular beam epitaxy.

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I may have already mentioned to you that MBE is a physical evaporation process. It is a physical evaporation process with no chemical reaction involved in it. That is the basic difference between MBE and other epitaxy systems. In molecular beam epitaxy there is absolutely no chemical reactions involved. It is simply a physical evaporation process. Now, let us compare the merits and demerits of the molecular beam epitaxy process with respect to the vapour phase epitaxy that we have already seen.

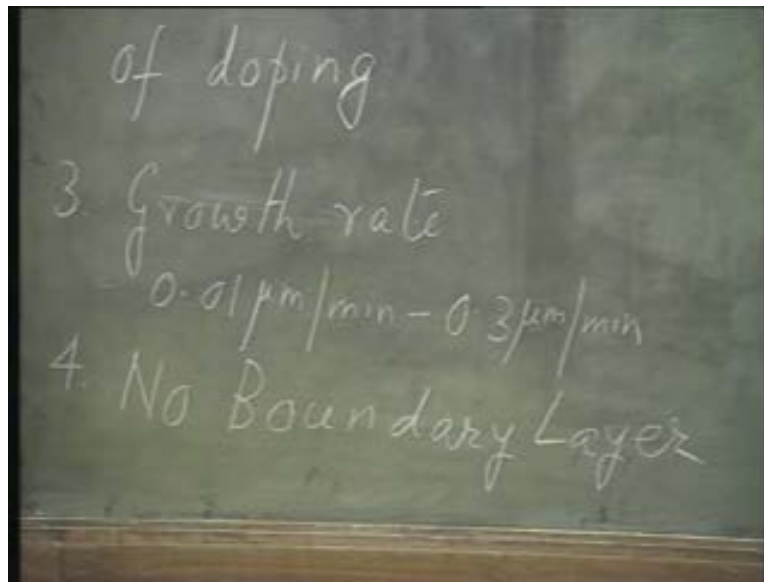
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The first major advantage of the MBE process is it is a comparatively low temperature processing. Compared to vapour phase epitaxy, it is a low temperature processing and that is the advantage that the effect of auto doping is minimized. You know, auto doping is basically because of out diffusion from the substrate into the epitaxial layer and vice versa, as well as its evaporation from the substrate and then reintroduction from the gas stream. Both these processes will be aided at higher temperature. So, if I can have a low temperature process the intermixing will be minimized, I can have less of auto doping problem. Then the second point, second advantage of molecular beam epitaxy is one can have precise control of doping. Again, this is because there is absolutely no chemical reaction taking place.

You can, you are having basically a physical evaporation system. Along with, along with evaporating silicon, you can evaporate measured quantity of dopants. So, the control of dopant incorporation is much more precise. MBE is also, the growth rate can be more precisely controlled.

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You can achieve a growth rate as small as 0.01 micro meter per minute up to say, 0.3 micro meter per minute. So depending on how thin or thick you want your epitaxial layer to be, you can control the growth rate and particularly I would like to draw your attention to the lower end of the epitaxial growth rate. It is 0.01 micro meter per minute that means 100 Angstrom per minute, very precise control. So, with the advent of VLSI technology, we are going more and more towards reducing all the dimensions, right and if I reduce all the dimensions that automatically means that the thickness of the epitaxial layer is also going to be reduced.

Remember, in vapour phase epitaxy process, first of all the growth rate may be a bit too high. If you remember, we had a growth rate in terms of fractions of micron per minute to say, 1 or 2 microns per minute, much faster growth rate. It becomes that much more difficult to obtain a thin layer. Then, the other problem is of course the problem of auto doping. It becomes, the thinner your layer is, the more difficult it becomes to control the thickness of the transition layer.

MBE eliminates both these problems, by having a smaller growth rate as well as by minimizing the auto doping problem you can grow a very thin layer with precise control

of doping. In other words, you have much better control over your epitaxial layer and not to forget I have another advantage of MBE. That is here there is no boundary layer or stagnant layer problem. Remember all VPE processes, particularly the horizontal and the barrel type of reactor is prone to boundary layer problem. That means the growth rate will not be equal all through the chamber. You have to take special precautions like tilting the substrate holder or increasing the gas flow rate or reducing the pressure in order to minimize the boundary layer problem. In MBE, you do not have this boundary layer problem at all. So, if MBE offers so many advantages, what is the flip side? There has to be a flip side, you see.

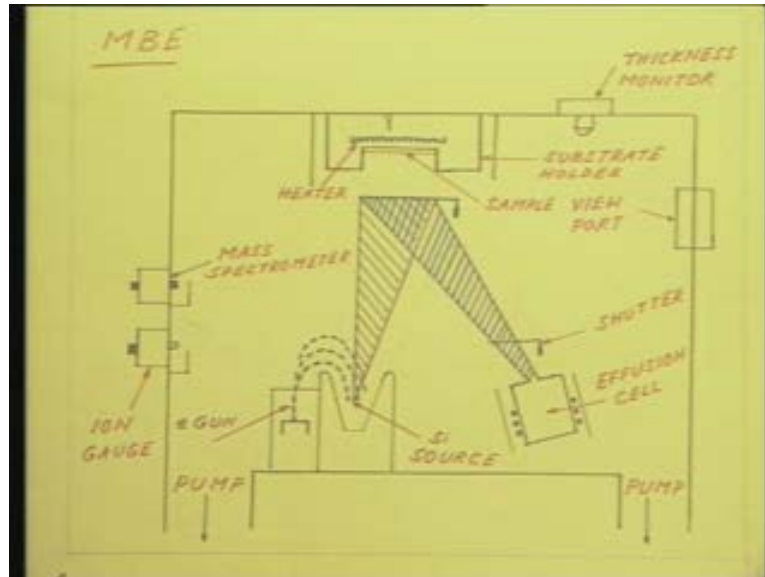
The flip side is simply this. MBE is a very costly and a very sophisticated equipment. In fact, I do not know, may be all over India there are not more than 2 or 3 MBE systems in the institute levels. One of them is housed in IIT Madras, one of them is housed here. It is a very expensive, very sophisticated, very complicated system. So, that is actually the flip side that using MBE is much more difficult than using vapour phase epitaxy. But of course, MBE gives you much, much better control of the epitaxial layer compared to other conventional epitaxy systems.

So, what is this MBE? What is done in this MBE? How is it done? As I have already told you, MBE is a physical evaporation process. That is very simply what is done is the layer that you want to deposit, it can be silicon, it can be any other material, it has to be evaporated and this evaporation is done under very high vacuum condition to minimize the contamination problem, number one and also so that there is no collision. So, MBE is always done under very high vacuum condition. Silicon MBE in particular, it is done in ultra high vacuum that is 10^{-8} to 10^{-10} torr.

The heart of this MBE system is basically this ultra high vacuum pumping, how to achieve this ultra high vacuum pumping and then, once you have achieved this ultra high vacuum condition inside, all you have to do is evaporate the particular species. In case of silicon, since silicon has a very high melting point, you know, silicon's melting point is greater than 1400, so it is very difficult to do so by thermally heating it. So, what is done

is by using an electron gun, an electron beam is focused on the silicon source and silicon is evaporated. First of all, let us have a look at the MBE system.

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This is the schematic diagram of a MBE system, very simple schematic diagram. The actual MBE system is going to be much, much more complicated. What I have put here is just the essential parts. First of all I have shown here two pumps. That is because this ultra high vacuum is very, is almost impossible to achieve by using any single pumping system. Commonly, when we are not talking about ultra high vacuum, the type of pumps we use, you know, for a simple thermal evaporation system for example, that will be rotary pump and diffusion pump.

Now, you know, both these pumps use oil, particularly diffusion pump use oil. For MBE, therefore one should not use the diffusion pump, because of its oil vapour. So, usually it is a combination of various pumps like say, turbo molecular pump, then cryo pump or ion sublimation pump, something like that. More sophisticated pumping system is required to achieve this very high vacuum, usually as I said, a combination of pumping system. Then, inside you see this is the silicon source, the schematic representation of the silicon source. That is I have a holder, crucible or something like that and in that silicon is

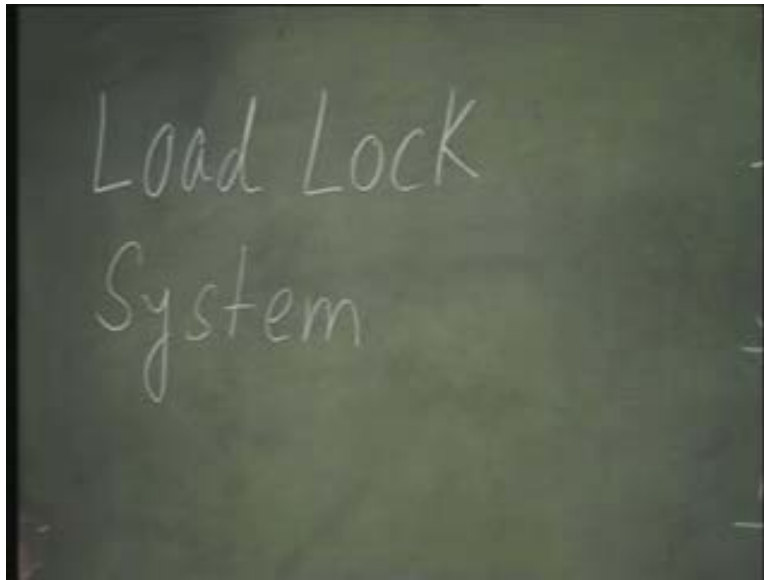
placed. Next to this the electron gun is kept. E beam is focused on to the silicon source and from there silicon is getting evaporated in this conical shape. In order to introduce dopants what is usually done is one uses an effusion cell. An effusion cell is again a dopant holder which is heated and there is a small opening at the mouth of this effusion cell through which a jet of evaporated dopants come out.

You have one or more than one shutters. These shutters are usually controlled by microprocessors that is to control the layer thickness as well as the amount of dopants incorporated in the epitaxial layer that is grown. The sample itself is placed here. There is a substrate holder, there is a small heater, there is a thermocouple to measure the temperature of the substrate. The substrate temperature is usually much lower around 400 to 800 degree centigrade. That is the substrate temperature.

Essentially what is done is this. You pump down the system to the required vacuum level and then you switch on the electron gun. The evaporated cone of silicon comes here. If you want to incorporate dopant, you hit the effusion cell, open the mechanical shutter, the dopants are also incorporated. So, this hatched region is actually where the silicon mixed with dopants are available and that goes and gets deposited, condenses on the relatively lower temperature substrate. Very simply said, but like I said, it is very simply said, it is more difficult to achieve.

The first important problem is how to maintain this ultra high vacuum. Please understand, we are talking about a vacuum level of very high degree, 10^{-8} to 10^{-10} . Now, every time I introduce the substrate, the chamber has to be open to the atmosphere and from there to take it down to 10^{-8} or 10^{-10} is almost impossible. That is you cannot do it every time. Then, in between two runs you will have to spend a lot of time. I am not talking about hours, I am talking about days or even weeks of pumping down, in order to get down to the requisite vacuum level from the atmospheric level. So, what do you do? What is done is called a load lock system.

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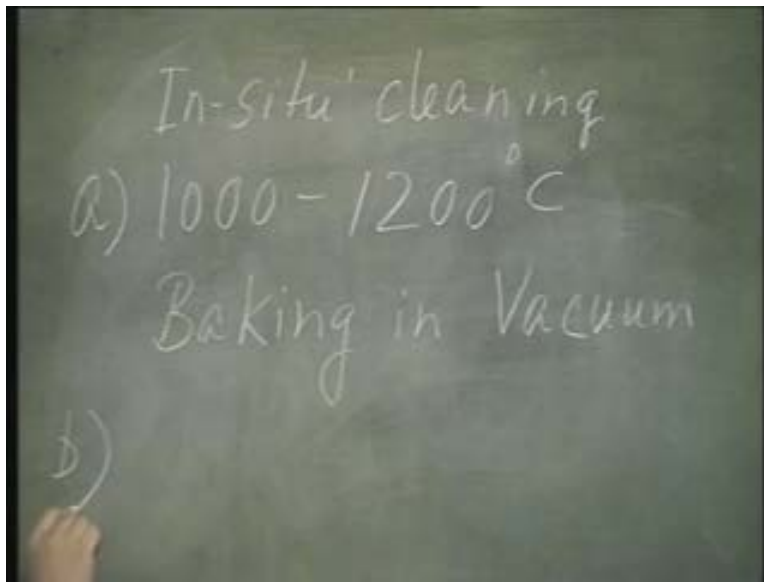


In a load lock system, the sample can be introduced into the chamber without having to expose it to the atmosphere. What is done is something like this. You have a door or a hatch with two O-rings. You place the sample on this side of the hatch. So, this side O-ring is opened. You place the sample on this hatch and then you turn this. When you are turning this, vacuum is not broken. So, the sample goes on to the other side. Now, this other side O-ring is loosened and the sample is introduced in the chamber. You may also have a multi chambered system.

In fact, most modern MBE systems have more than one chamber. One chamber is for introducing the substrate. So, that chamber with the help of load lock is used to introduce the substrate. Once the substrate is inside that chamber, that chamber is first pumped down and then from there the sample is taken to the inner chamber. So, you may have a multi chamber, in addition to the load lock system. So, without breaking the vacuum, the substrate must be introduced into the chamber that is without exposing the chamber to the atmospheric pressure it must be introduced and that is done by the help of the load lock system. So, your sample is introduced into the MBE system.

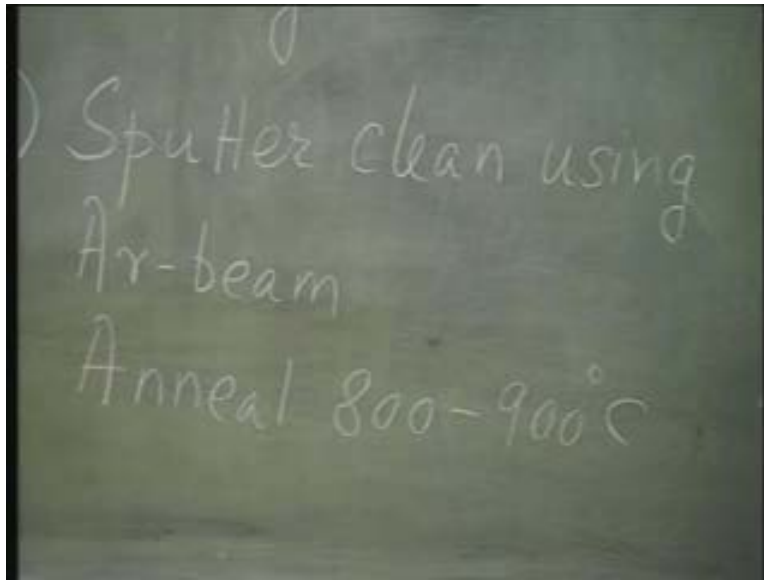
Now, the next most important thing is, you know, before the epitaxial growth, there must be a provision for in situ cleaning. Otherwise, you will have a lot of defects on the epitaxial layer. Whatever is present on the surface, if you have patches of oxide or if you have some damages, it will just get translated into the epitaxial layer, causing stacking faults, dislocations and things like that. So, this in situ cleaning has to be first done. Please understand in the MBE system, I have no provision for a chemical etching, I usually have no provision for a chemical etching.

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So, what is done is you can either bake it at high temperature, high temperature that is 1000 to 1200 degree centigrade, bake it in vacuum in order to clean it in situ. The other possibility is you can sputter clean the surface. In order to sputter clean the surface, you use a low energy beam of an inert gas like argon. You focus that low energy beam of the inert gas on to the substrate surface and you are sputtering the surface clean. That is you are physically removing a thin layer of material from the substrate surface. This will of course cause some damage to the surface. It will sputter off some material from the surface alright, but this will also cause some damage to the surface. That damage can be annealed out by heating it to a temperature of 800 or 900 degree centigrade. So, there are two provisions.

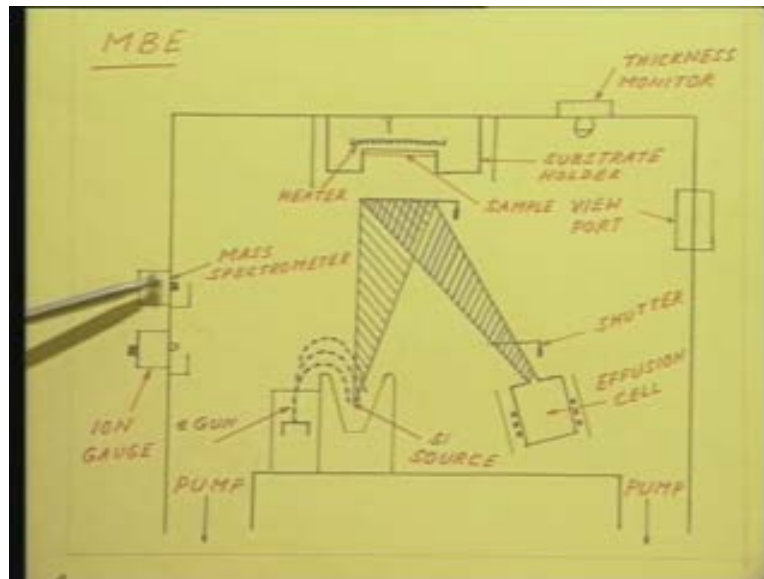
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Normally, argon beam is used. Argon is an inert gas, everyone know. This has to be annealed at 800 to 900 degree centigrade, in order to **clear** clean the surface of the damages in addition to the sputter cleaning. In fact, this is preferred nowadays, because obviously you can reduce the temperature. Remember, when we talked about the advantages of MBE, we said it is a comparatively low temperature process. If we have to bake it at 1000 to 1200 degree centigrade for a few minutes, may be 10 minutes or so, then you know, you are surrendering some of that advantage, you know, because you have to still heat it at that high temperature. So, this is preferred nowadays - an argon beam sputter cleaning followed by a short anneal at comparatively lower temperature, 800 to 900 degree centigrade.

Now as I said, the heart of this molecular beam epitaxy is the ultra high vacuum system. It is very difficult to maintain and all that, but this ultra high vacuum system gives us an advantage that many of the sophisticated characterization techniques they also need high vacuum to operate. So, since in MBE you already have a very high vacuum, you can incorporate a lot of sophisticated characterization techniques inside the system itself and therefore, monitor the quality of your epitaxial layer much more closely.

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For example, like here I have shown a mass spectrometer, a quadruple mass spectrometer. This is used to monitor the gas composition and if you can monitor the composition inside the system, of course you can monitor the dopant levels as well as the layer quality. There are also various other things like for example, here I have shown that the doping is done from this effusion cell. Dopant is taken inside the effusion cell and heated and through that small opening it gets evaporated. There is another provision.

I have mentioned that doping can be done by ion implantation. An ion implantation system can be incorporated inside the MBE itself. So, that is actually shown here. You can have an ion implantation system inside the MBE, so that in addition or instead of using the effusion cell, you could use ion implantation. As I have already mentioned, ion implantation will give you a better control over the doping profile of the epitaxial layer.

So you know, for example there is a device called a varactor diode where the capacitance of the diode, reverse biased capacitance of the diode, varies. You know, it varies with increasing the reverse voltage. But in normal diodes, this variation will not be linear. In a varactor diode we would prefer that to be linear. So, for that it will be good if the diode doping profile could be linearly graded. So, this is something which can be easily achieved using MBE system. You know, you could have the substrate doping linearly

graded by using MBE, which is one use. So, you can have the ion implantation system incorporated inside the MBE.

There are other characterization techniques. For example, one is called RHEED.

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This stands for reflection high energy electron diffraction, reflection high energy electron diffraction. This is a characterization technique used normally for assessing the crystal quality of the epitaxial layer, as the layer is growing. So, what you do is you focus an electron beam at a grazing angle, very small angle about 1 degree or so; focus an electron beam 5 to 40 kilo electron volt energy, focus it in a grazing angle like this. If this is your substrate, focus it like this at a very small grazing angle, so that the penetration depth will be very small. So, what you are measuring is actually the surface quality. As the epitaxial layer is growing, you are measuring the surface quality in situ and looking at the RHEED pattern, the diffraction pattern you can find, you can assess how the crystal quality is going to be, whether it confirms to your requirement or not.

Various other characterization techniques can be incorporated in the MBE system. For example, X-ray photoelectron spectroscopy, XPS. XPS you know, it is used in order to

find the composition of the layer. That is in fact more important when you do not have silicon, but may be a compound semiconductor molecular beam epitaxy. You want to make sure whether it is stoichiometric or whether for example, if you are having say, aluminum gallium arsenide, what should be the relative composition? What should be the relative percentage of aluminum with respect to gallium that can be measured in situ if you have a XPS system. But like I said, these systems can be incorporated. That does not mean that all MBE systems will come equipped with these systems. Most MBE systems however will have the quadruple mass spectrometer to monitor the gas composition and the RHEED to assess the crystal quality in situ. In addition, it may also have the ion implantation, in order to facilitate doping. So, this is basically a molecular beam epitaxy system.

Now that we have grown our epitaxial layer, by whatever process - vapour phase epitaxy or molecular beam epitaxy, we have created a substrate. Now is the time to follow it up with other device processing steps, other processing steps. The two important steps in connection with crystal growth we have already covered. That is the bulk crystal growth and the epitaxy. So, now our substrate is ready. We follow it up with some processing step. Invariably the first processing step you do, you know in the lab we first take out a substrate material wafer. Usually the growth of this wafer is outside the purview of most fabrication labs, it is a highly specialized technique. So, we have specialized vendors all over the world, who specialize in giving you this substrate material whether it is bulk substrate, whether it is epitaxial substrate, what kind of epitaxy they use, what kind of crystal growth they use, you know it is a highly specialized job.

So, what we get actually in any fab lab that is you have the wafer, the 4 inch or 6 inch diameter discs of shining grey silicon material, one side mirror polished. So, once you have that substrate material, first of all of course, you will have to clean the sample, before you do any processing. Cleaning the sample is very important and in semiconductor cleaning, you know, cleaning in water is quite important. Now, when I say water do not think tap water, because tap water contains a lot of impurity and mostly a lot of ionic impurities, which is going to be extremely detrimental of, detrimental to the

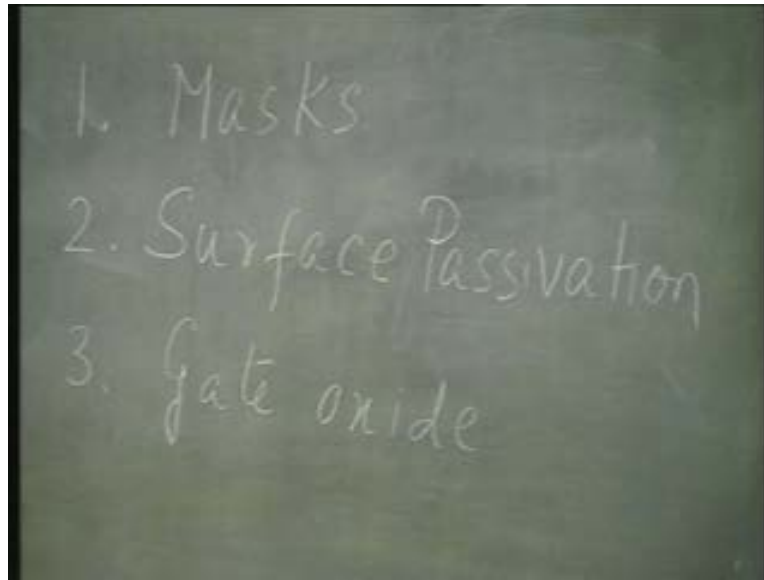
performance of the device. In fact, no device will perform. If you just wash a wafer in tap water and try to use it no device will perform. In addition, you would have contaminated all the systems in the lab.

So, in semiconductor processing, you have to get used to a term called de ionized water. Normal tap water has a pretty low resistivity. If you ever try to measure the resistivity of tap water, you will find it is fairly low - hundreds of ohms to kilo ohms. That is because, there are a lot of dissolved salts in it, sodium ions, potassium ions; so, that lowers the resistivity of the tap water. What is done is that water has to be passed through an ion exchanger, where all the cations are replaced by hydrogen, all the anions by OH and then they are further purified using a lot of filters and all, so that finally for semiconductor device processing we need water of more than 10 mega ohm resistivity, 10 mega ohms resistivity. If you are working with mass devices, your requirements may be even more stringent. You may want more than 16 or 20 mega ohm resistivity, because the mass device is a surface device and therefore more prone to contamination.

So, after you have taken the substrate, you have to subject it to various chemical cleaning, degreasing, you know. However careful you are there are some grease, some dust on the surface. So, use a number of organic solutions like trichloro ethylene, acetone, etc, then finally clean it in de ionized water and then before any processing, the usual practice is to boil this wafer in an acid, usually nitric acid or a mixture of sulphuric acid and hydrogen per oxide. This is in order to grow a thin layer of oxide on the surface which will then be sacrificed, be etched off in a diluted hydrofluoric acid solution.

So, what you are getting finally is a fresh surface. You form an oxide on the top of silicon surface and then etch it off, in order to expose a clean fresh surface on which you do your further processing. Now, whenever I start my processing, anybody starts the device processing, one of the very first steps is oxidation, you know. You need this oxide and then, you have to follow it up with photolithography to open windows in the oxide in order to selectively dope the semiconductor material, so that your devices can be realized. So, that brings me and you to a very important processing step called oxidation.

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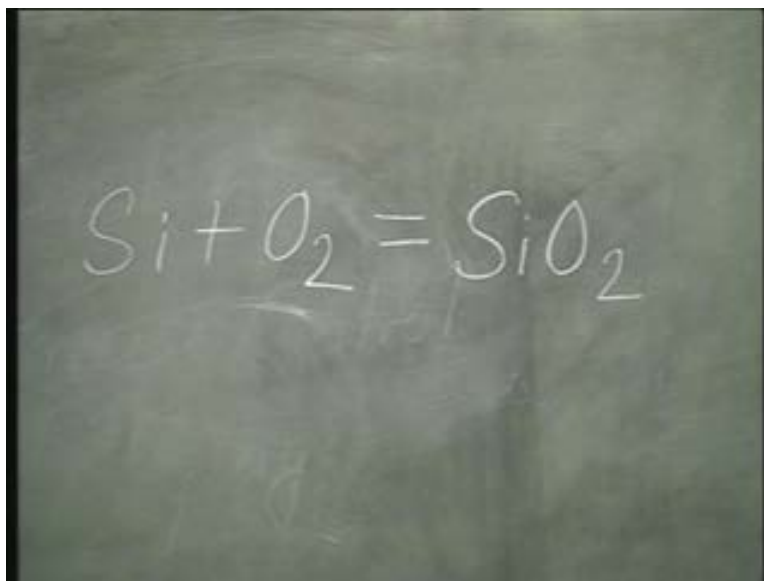
First of all what is the use of this oxidation? The most general use of oxide is for masks. This oxide is going to protect certain regions through which I do not want any doping to take place and they will open some windows in this oxide, through this doping will take place. So first, most important is, as masks. This is also used for surface passivation. That is after your device is fabricated, you do not want all the junctions to be exposed to the ambient condition. So, what is done usually is that any exposed junction, it is passivated by silicon dioxide. Only those portions are exposed through which you are going to take your metal contacts. That portions are covered with metal, the rest of the surface is covered with silicon dioxide and that acts as surface passivation and of course you know, these are all passive use.

Passive use in the sense is that the oxide, all though it is helping either in device fabrication or in preserving the device characteristics, it is not playing an active role in the device performance. But, the third use of oxide is as an active element in the device and that is as gate oxide. So, my requirements are different. You see, when I am talking about gate oxide in a MOS field effect transistor, I am more worried about the quality of this oxide.

On the other hand, when I am talking about masks I am more worried about the thickness of this oxide, because I need a requisite thickness. I know that I will follow it up with a certain doping step. So, during that doping the oxide thickness must be such, as to withstand, as to mask during that dopant incorporation. So, my requirements are different. You see, for gate oxide I am talking about thin oxide, but with very good interface quality. For the masks I am not that bothered about the interface quality, I am bothered more about the thickness. So, depending on these requirements, I will suitably use different oxidation techniques, in order to grow thin oxides or to grow thick oxides. But, whatever I want to grow, it has to be a chemical reaction between silicon and oxygen.

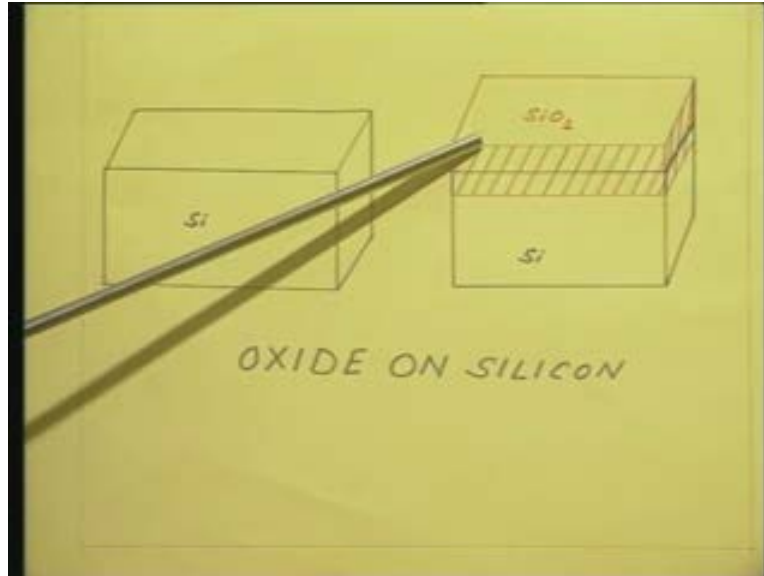
Of course, we can have chemical vapour deposition of silicon dioxide also. Even in that case you know, you have the chemical reaction, so that silicon dioxide is formed and then it is deposited on the substrate. Right now, we are not going to discuss the chemical vapour deposition of silicon dioxide. We are only going to discuss the oxidation of silicon that is where the substrate itself is reacting with the gas that is oxygen or steam or some oxygen containing species and forming silicon dioxide. So essentially, I have the reaction like this.

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Silicon reacts with oxygen to form silicon dioxide.

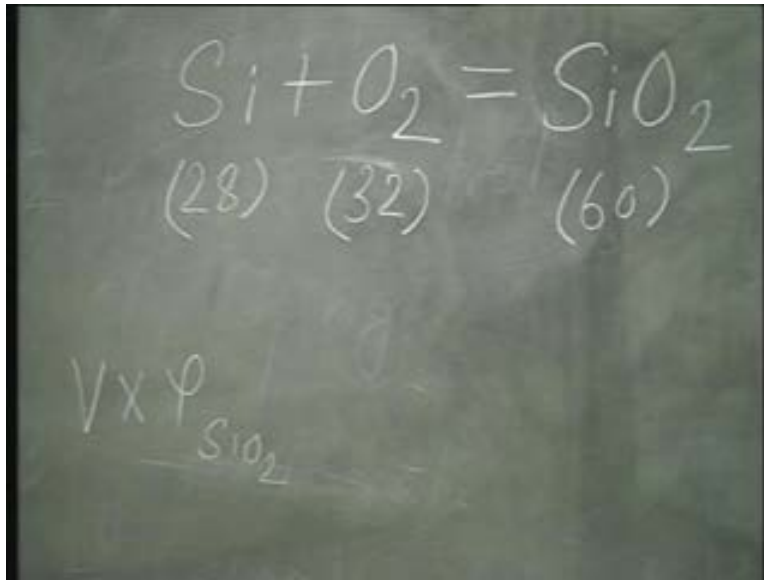
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So this is my bare silicon substrate and I subject this to oxidation. So, what happens? The entire surface is covered with silicon dioxide. Now, if you notice in these two figures, this was my original silicon surface. That original silicon surface you can see here and deliberately I have drawn the oxide like this. Some part of it is rising above the original silicon surface and some part of it is going below the silicon surface. How much of it is above and how much of it is below and what is the logic for drawing it like this? You know, if you look at this reaction, silicon plus oxygen is forming silicon dioxide. So, the logic is quite clear. Silicon is being consumed in order to form the silicon dioxide. So, obviously the oxide is not, you know, not staying atop the surface, it is consumed; it is consuming the silicon, so that is why some part of it is going below and some part of it is going above. By how much? How much is below the surface, the original surface and how much is above the surface?

We will do some simple calculations and see what is this? We know, the molecular weight of silicon is 28.

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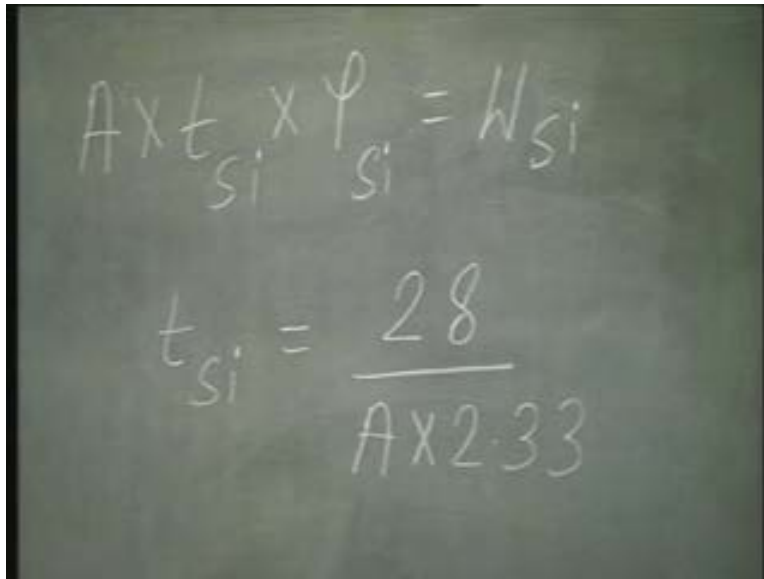
This is 28 and this is 16 plus 16 that is 32. Therefore, the molecular weight of silicon dioxide is 60. So, going by this reaction I can say that 60 grams of silicon dioxide will be formed by consuming 28 grams of silicon. Isn't it? Now, what is 60 grams of silicon dioxide? 60 grams of silicon dioxide actually means the volume into density. What is the volume?

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$$A \times \rho_{\text{SiO}_2} = W_{\text{SiO}_2}$$
$$t_{\rho} = \frac{60}{A \times 2.27}$$

Area into thickness, isn't it. So, this is going to be the weight of silicon dioxide. In other words, I could say t_{ox} is 60 grams divided by area, whatever the area is, that is the cross sectional area of the original wafer essentially, area into the density of the silicon dioxide and the density of silicon dioxide is 2.27.

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$$A \times t_{si} \times \rho_{si} = W_{si}$$
$$t_{si} = \frac{28}{A \times 2.33}$$

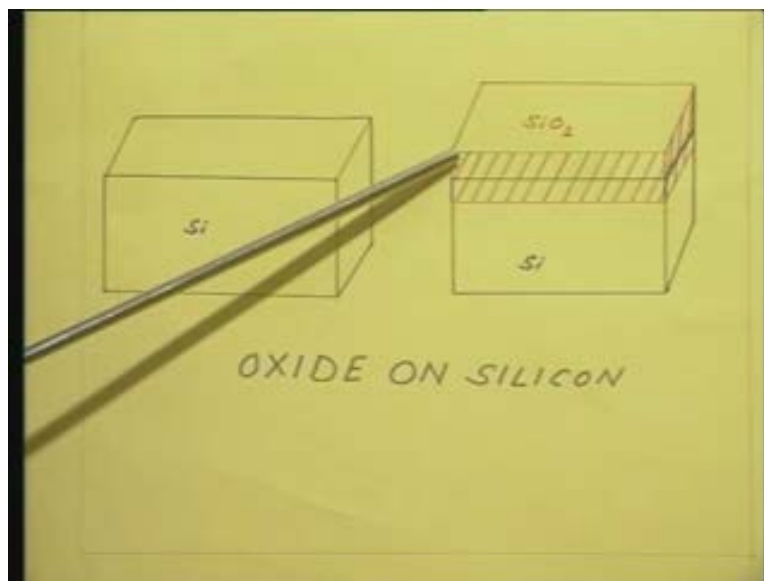
On the other hand, similarly I can write, for the silicon the equation is like this - t_{si} is the thickness of silicon that is consumed. So, I know that in order to form 60 grams of silicon, we needed to consume 28 grams of silicon. 60 grams of silicon dioxide is formed by consuming 28 grams of silicon. So, I could say this t_{si} is therefore equal to 28 by A into the density of silicon and that is 2.33. So, now it becomes very simple. I have an expression for the silicon thickness that is consumed and I have an expression for the oxide thickness that is grown.

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$$t_{Si} = \frac{28}{Ax2.33}$$
$$\frac{t_{Si}}{t_{O_2}} = \frac{28}{Ax2.33} \times \frac{Ax2.27}{60}$$
$$\approx 0.45$$

So you see, since they have the same cross sectional area and this works out to be about 0.45. What is the significance of this number? The significance is, if 1000 Angstrom of silicon dioxide is grown, 0.1 micron or 1000 Angstrom of silicon dioxide is grown, it has consumed 450 Angstroms of silicon.

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That is looking at this figure once again, if the total thickness, total width of the red hatched area is 1000, 450 is below the black line and 550 is above the black line. It has consumed, below the black line is the region of silicon that is consumed. So, 450 Angstroms of silicon have been consumed and 550 is above the original surface. That is why if you form an oxide and then selectively etch it, you will get an indentation on the surface itself. That is because some part of silicon was consumed wherever the oxide is formed.

We will stop here today and we will discuss about the actual oxidation of silicon in the next class. How the oxidation of silicon goes on, what are the parameters governing the oxide growth rate, how does it depend on various parameters like temperature, pressure and so on and from that we will also deduce how to determine the quality of the oxide, how to decide which temperature, pressure or what kind of oxide species to be used, when I want a particular thickness or a particular quality of oxide? So with this, we will begin our third processing step that is oxidation of silicon.