VLSI Technology
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Lecture - 7
Crystal growth Contd. + Epitaxy I

So, we have discussed about the two main crystal growth techniques which are used to grow any crystal and in particular, silicon and you have seen that even though the Bridgman crystal growth technique is simpler, it will give rise to a lot defects and dislocations in the crystal and therefore almost universally silicon is grown by Czochralski technique. Czochralski technique is more complicated, more expensive equipment is needed, but it will give you much better quality of crystal, much lesser defect density, because the crystal as it is growing, it is not constricted by the confinement of any boat. But, the Czochralski grown crystal is still not the perfect crystal that we would, may like to have for certain applications.

The primary source of problems in a Czochralski grown crystal is because of the oxygen and if this oxygen is beyond a certain limit, then this oxygen will be precipitated inside the crystal and that will give rise to precipitates from where dislocations will emanate and if you are trying to fabricate power devices on such a material, then those devices may have lower break down voltage, premature break down or higher leakage current. So, if you are very particular about the application, very stringent requirements, if you have very stringent requirements, then it may be necessary to further purify the Czochralski grown crystal and that is called zone refining.

Zone refining is a, it is even more expensive. In order to get some, you have to give some, so you pay, in order to get a better quality of crystal. It is more expensive. The idea of zone refining however, is fairly simple.

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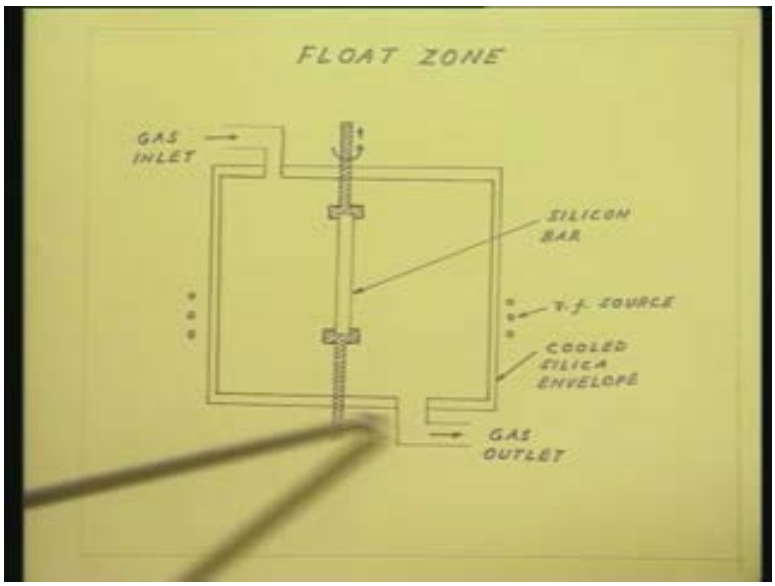

What we have here is, we have a cooled silica envelope. Just like in case of a Czochralski unit you had a water cooled silica envelope, even here we have a cooled silicon envelope and we have gas inlet and gas outlet. Obviously, an inert ambient has to be provided much like in Czochralski. Now, in this cooled silica envelope, you hold a silicon bar vertically like this. Therefore you see, you need a very expensive starting material, you need a silicon bar, a rod of silicon. So, this starting material itself is going to be quite expensive, unlike in Czochralski or Bridgman where you could use powdered or poly crystalline silicon material, so here we have to have a silica, silicon bar.

This silicon bar is held in a ..... and at one end of this silicon bar, a small seed crystal is provided which I have not shown in the figure, but you can assume that at this end a small seed crystal is fixed and the heating is provided by the RF source which is movable. So, the heating starts from the seed end of the silicon bar. The seed end of the silicon bar, a small region about 1.5 centimeter in length that is first molten. Using this RF heater, a small portion of the silicon bar is molten and this molten portion is in contact with the seed crystal, so that the tip of the seed crystal is just molten and now the heater is moved slowly along the length of the rod. What is going to happen? At the retreating end of the heater, as the heater is moving up, let us say, it will leave behind a portion of

silicon that is getting solidified and since that portion is in contact with the seed crystal, it will grow as a single crystal. It will grow as a continuation of the seed crystal itself.

What is the advantage? The advantage is that here I do not need, repeat, I do not need a crucible. In Czochralski system, we needed a crucible to hold the charge. The crucible was made of quartz, silica, so that was the primary source of oxygen in silicon. Here, in the zone refining technique or the float zone technique as it is called, I do not need a crucible. Silicon bar is held without any crucible. Just at the two ends it is held. Therefore, you do not introduce any contamination, do not introduce any contamination there. So, by this technique we can reduce the oxygen content by a factor of 10 or even 100 and no dissolved impurity, since we do not have any crucible.

The crucible may be a source of oxygen and also other impurities. So, the purity of the float zone crystal is going to be much better than the normal Czochralski grown crystal and you can use repeated passes. By pass, I mean one movement of heater along the entire length of the bar. You could use repeated passes in order to further improve the quality. So, it can be shown that the normal Czochralski grown crystal has a certain resistivity. After one pass the resistivity improves, after two passes the resistivity improves further. So, depending on what is the particular substrate resistivity you want, you may decide to use one or multiple passes. But understand, this is going to be a more expensive process, because your starting material itself is going to be very expensive.

This is only justified when your requirements are very stringent. So, particularly for power device applications, when you are looking for a substrate you will look for an fz crystal, not for an ordinary Cz crystal. By Cz, I mean Czochralski grown crystal, fz is the float zone material. A float zone material will necessarily have lesser oxygen content, better resistivity and the devices fabricated on this will have higher break down voltage. So, when you need a break down voltage of 1000 volts or so, then you should always opt for a float zone material. So, zone refining is nowadays quite popular. Many of the venders, they sell this fz crystals and this will give you, your semiconductor single crystal, silicon single crystal.

After we get this single crystal a lot of mechanical and chemical processes, the crystal has to under go a lot of chemical and mechanical process, so that we finally get the wafer. First of all as you grow the crystal, you know, the seed end and the bottom end, these two ends are normally cut off, they are not used. Then mechanically it has to be ground out. Then, it has to be cut into slices, then each slice has to be chemo mechanically polished; first mechanically polished and then chemo mechanically. Using a slurry of aluminum oxide and glycerin usually it is chemo mechanically polished, so that you finally have one surface mirror polished that is the surface on which you are going to fabricate your device. So, the crystal thus grown we call it the bulk material, the bulk material. It is grown by the crystal growth technique and we have the bulk material.

Now, this bulk material can then be subjected to some gettering processes, gettering.

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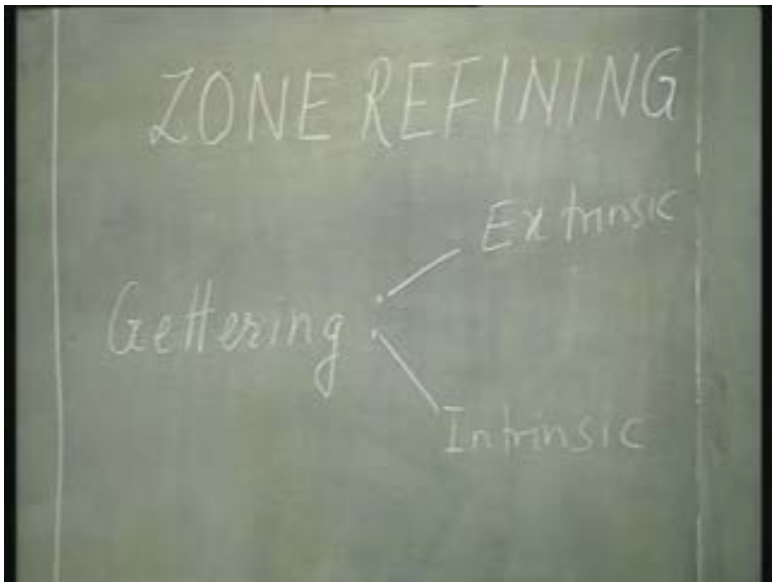

This gettering is done, so that the active region of the device is free of any metallic contamination. Please understand that most of the device is actually contained on the top few microns of the silicon wafer. The actual silicon wafer is, may be 300 to 500 microns thick. That is 0.3 to 0.5 mm, but the actual device is contained may be within the first maximum 5 or 10 microns. Isn't it? Whether it is a bipolar junction transistor or a

MOSFET, it is going to be only on the top surface of the device, rest of the portion, rest of the crystal is only going to give you the mechanical support. So, it is necessary that this active region, this top few microns, it is free of any metallic contamination.

This can be done by gettering and the gettering can be extrinsic or intrinsic. By extrinsic gettering, what I mean is the back side of the wafer, the not polished side of the wafer is subjected to lesser aberration or electron irradiation. What will happen? It will give rise to dislocations near the back surface and these dislocations will act as sinks for metallic contamination. So, when the wafer is subjected to processing, if there are metallic contaminants, they will travel through the active region of the device and get sunk at the bottom, so that the active region of the device is free of such contaminants.

The other type of gettering which is called the intrinsic gettering, it uses the oxygen that is in the crystal. We told you that if the oxygen is above a certain range, then it will give rise to precipitates and the precipitates are usually undesirable, because dislocations will emanate from that. But, you can use it to your advantage provided you make sure that such precipitates are not there in the active region.

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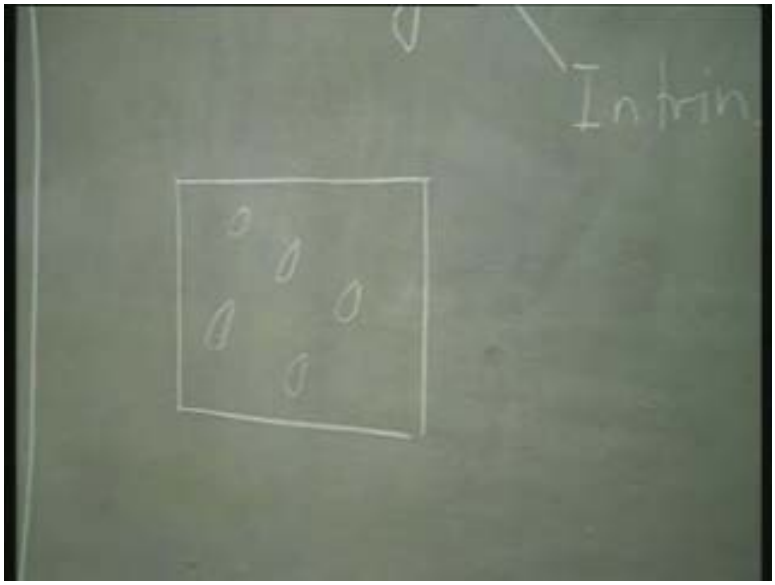

That is what I am trying to say is suppose this is your wafer cross section, this is the thickness of the wafer, full 500 micron. So, I have this oxygen in this locations or oxygen precipitates all over the place. Now, I heat this wafer at 1000 degree centigrade or above in an inert ambient. What will happen? The oxygen from near the surface, it will be evaporated. They will evaporate out. So, the active region is free of this oxygen precipitates, but I still have oxygen precipitates deeper in the bulk, which can be used as gettering centers, which can be used as sinks for metallic impurities. So, you can use the oxygen precipitates also which are already present in the crystal. That is why it is called the intrinsic gettering. You are using what is inherently present in the crystal itself. That can also be used to your advantage provided you make sure that these precipitates are not threatening the active region of the device.

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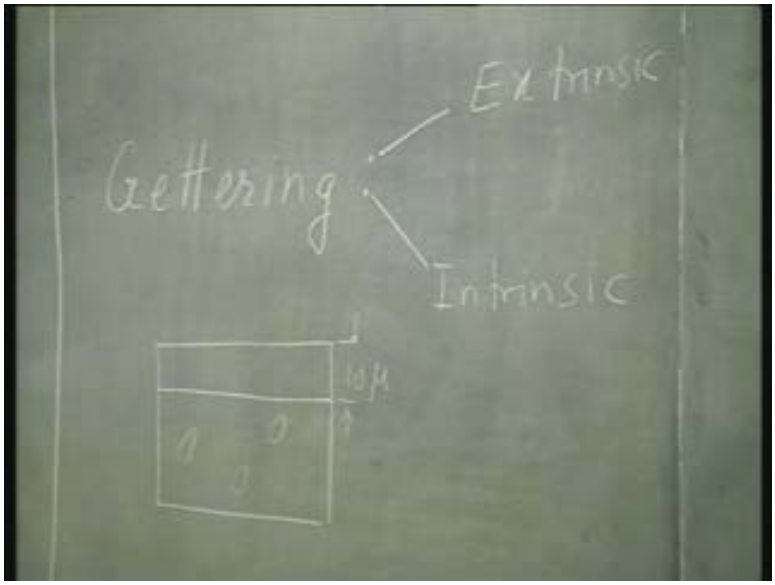

So, your device is contained in the top, let us say, about 10 micron and the rest of the portion, it is only used for mechanical support. You are not going to house your device there, therefore you do not care. There can be oxygen precipitates there and that can be used to your advantage. So, this is what a bulk semiconductor crystal is like. This is how it is grown, this is how their properties are, these are the factors by which their properties are controlled and how you can further improve the properties.

But for various applications, just having this bulk single crystal is not enough. For example, think about your bipolar junction transistor. We started out in order to make an npn transistor. We started out with a p-type substrate and then, after the buried layer diffusion we had to carry out an epitaxy, in order to have the n region grown on top of this p type crystal. An n crystal grown on top of the p-type bulk crystal and the actual device was housed in this epitaxial portion, not in the bulk crystal, remember. So, just having this bulk crystal may not be sufficient for your needs. For that you have to use epitaxy which brings me to the second processing step after crystal growth. That is epitaxy.

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Actually epitaxy is a combination of two words - epi means upon and taxis means ordered. So, the meaning of epitaxy is simply juxtaposed these two words, ordered upon. What is ordered upon what? That is the epitaxial layer is arranged or ordered upon the bulk crystal. So, please try to understand the difference between the bulk crystal growth and epitaxy. By bulk crystal growth, we grow, as the name itself suggests, so, you grow big crystals. We cut the entire wafer, discs from this crystal. We are talking about thicknesses of 500 micron or so. Once I have such bulk material, I can grow a thin layer, much thinner than this bulk crystal thickness, much thinner, a few microns, from fraction

of a micron to a few micron. That thin a crystalline layer will be arranged on top of this already existing bulk single crystal. This is epitaxy, ok. So, an epitaxial layer will always be much thinner compared to the thickness of your bulk crystal and this is a very interesting point.

What can be arranged upon what? That is in other words, epitaxy, in order to grow this thin crystalline layer on top of a bulk crystalline layer, what are the restrictions? Can I grow anything on anything? Well, which brings me to the question of homo and hetero epitaxy. As is self evident, when I say homo epitaxy, I mean that the same material is grown over the bulk crystal. That is I already have a silicon bulk crystal, I want to grow a silicon layer on top of that, may be of a different doping concentration, of a different resistivity. But, the material is the same which you have encountered when we talked about the bipolar junction transistor. We had a p-type silicon substrate, on top of which we grew an n-type epitaxial layer, but both of them are made of silicon. So, this is an example of homo epitaxy. Homo epitaxy is fairly simple.

In contrast, if I have a certain crystal as the substrate, as the bulk material and I want to grow something else on top of that. That is if I have two dissimilar materials - the epitaxial layer, the material of the epitaxial layer is dissimilar from the bulk material, then it will fall under hetero epitaxy. A very common example of hetero epitaxy is silicon on sapphire. You might have heard of it, SOS, silicon on sapphire.

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For example, silicon on sapphire. Sapphire is actually nothing but aluminium oxide, Al2O3. It has a hexagonal lattice pattern and silicon as you know, it has a diamond lattice structure. So, two dissimilar material, silicon and sapphire, two dissimilar material we want to grow, one on top of other. So, hetero epitaxy is necessarily, it is much more complicated. The three cardinal rules that are observed during hetero epitaxy that is hetero epitaxy is possible if there are no chemical reaction between the substrate and the epitaxial layer.

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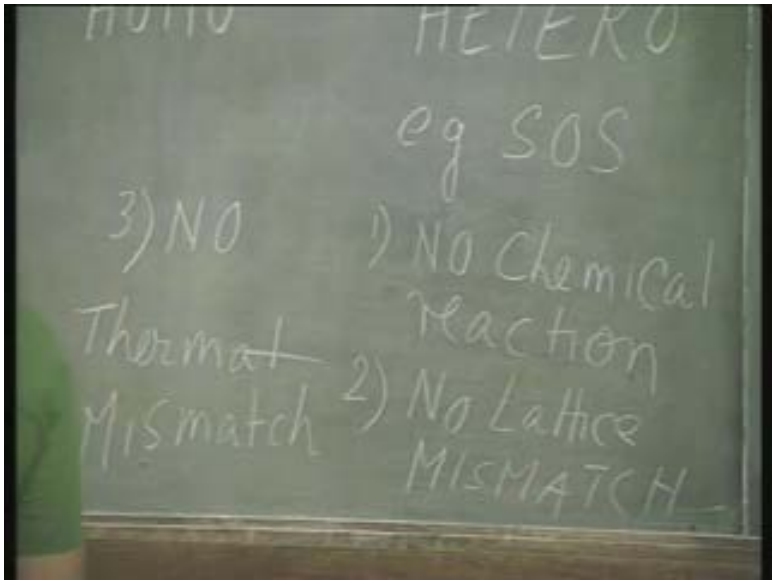

There should be no chemical reaction and then there should be no lattice mismatch and finally the coefficient of thermal expansions should match. That is there should be no thermal mismatch either. These are the three cardinal rules that there should be no chemical reaction between the two materials, there should be no lattice mismatch between the two materials and there should be no thermal mismatch between the two materials. But, only the first rule is really truly very stringent. If there is a chemical reaction between the two materials, you cannot have an epitaxy.

Lattice mismatch, we can tolerate up to a certain level. I told you already that sapphire and silicon there is no lattice matching, right. Sapphire is a hexagonal crystal and silicon is a diamond like crystal. There is no lattice matching, but it can be grown. The thickness of the layer that is the problem. That is you see, when there is a lattice mismatch, what will happen, there will be a lot of strain in the epitaxial layer. So, up to a certain thickness, if you are growing very thin layers, up to a certain thickness this strain can be taken, strain can be tolerated. Beyond that it is not allowed. So, strained layer epitaxy is a very important phenomena today, which is however outside the scope of our present discussion. But, let me just digress a little bit and tell you.

You see, actually this hetero epitaxy is particularly useful when we talked about compound semiconductor technology, because there you may have to, you want to have so many things like aluminum gallium arsenide on gallium arsenide. In this case of course, you have perfect lattice matching. Aluminum gallium arsenide is perfectly lattice matched with gallium arsenide. But, suppose you want some other material like indium gallium arsenide on gallium arsenide, there is no lattice matching. So, there has to be a strained layer. So, strained layers have a lot of interesting properties and that is why a strained layer epitaxy is a very, very major hot topic today.

In fact, I was just going through some technical journals and it said that the year 1997 was the year of grow anything on anything. People have tried so much on this epitaxy, hetero epitaxy. They tried to grow anything on anything and with a lot of success, with a very high degree of success. Like I said this is, right now this is beyond the scope of our present discussion, but let me tell you that this hetero epitaxy is a very, very hot area, a lot of wonderful research is being carried out all over the world and most of the barriers are going down. That is previously it was felt that you cannot grow gallium arsenide on silicon, but even that is possible today.

Even, I mean, see, one major problem with gallium arsenide technology is cost. So, if you can use the silicon bulk crystal and grow a thin layer of gallium arsenide on top of that, you have circumvented the cost problem. So even that is possible to a certain extent today. So, hetero epitaxy is again a very interesting point and the question of thermal mismatch, again that is also not a such a very stringent rule. Again, this will introduce some strain in the layer, but like I said within a certain limit, the strain can be tolerated. However, the first rule is still a rule. That is there must be no chemical reaction between the two material. Otherwise, you will not, in that case you cannot have a proper epitaxy.

Now, as far as our present discussion is concerned, we shall talk mostly about homo epitaxy that is growth of silicon on silicon. We want to grow for the VLSI technology however, we want to have mostly silicon on silicon with different doping concentration, yes, but the material remains the same. Before I start the discussion of silicon epitaxy in

particular, let me talk in general about epitaxy. You know, depending on what kind of reactions are being used or whether any reaction is being used at all, epitaxy system can be classified as liquid phase epitaxy, vapour phase epitaxy and molecular beam epitaxy.

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Liquid phase epitaxy, vapour phase epitaxy and molecular beam epitaxy or MBE. Now, this vapour phase epitaxy and the liquid phase epitaxy there are some chemical reactions involved in this. In contrast, in MBE there is no chemical reactions involved. This is a physical evaporation process. It is a very complicated, very sophisticated system, but basically the essence of a MBE is simple thermal evaporation. But, it is like comparing the, you know, super computer with your pocket calculator. The super computer is the MBE and your pocket calculator is the basic thermal evaporation system. The degree of complexity between an MBE and a simple thermal evaporation system is that big, but the principle is still the same. It is still a thermal evaporation system, a physical evaporation system with no chemical reactions involved. We will discuss about MBE little later.

First of all let us talk about LPE and VPE, liquid phase epitaxy and vapour phase epitaxy. Again, for silicon, liquid phase epitaxy does not have much use. So, silicon epitaxy is either VPE, vapour phase epitaxy or MBE. Still, let me tell you what these names signify.

Liquid phase epitaxy means it is done in liquid phase. What is usually done is this. You have some solution; in liquid phase epitaxy, you have a solution and you cool that solution. Suppose you have a, in case of gallium arsenide for example, suppose I have a solution of gallium arsenide in gallium. Gallium is liquid, at slightly above room temperature it will be liquid. So, you can dissolve gallium arsenide in gallium. Now, if you cool it, then you know when the temperature is reduced, the solubility will be reduced and there will be a precipitate. So, there will be a precipitate of gallium arsenide.

Now, if I bring this bulk crystal of gallium arsenide or wafer of gallium arsenide in contact with the solution as it is being cooled, then this precipitate will be deposited on the bulk crystal and if I can control the deposition very carefully, then it will take the orientation of the bulk crystal and I will get a single crystal layer, single crystal epitaxial layer on the bulk crystal itself. So, in essence this is liquid phase epitaxy. However, it is not particularly useful for silicon, because for silicon it is very difficult to dissolve. So, for silicon we almost always use vapour phase epitaxy. So, we will concentrate first our discussion on vapour phase epitaxy.

Now, in vapour phase epitaxy, as the name itself suggests the material transport must be in form of vapour. So, you have to use gas reactants. There is some chemical reactions involved, so you have to use some gas or a liquid with a very high vapour pressure, which can be vapourized very easily. So, essentially this is something like you have, can I have the next chart please.

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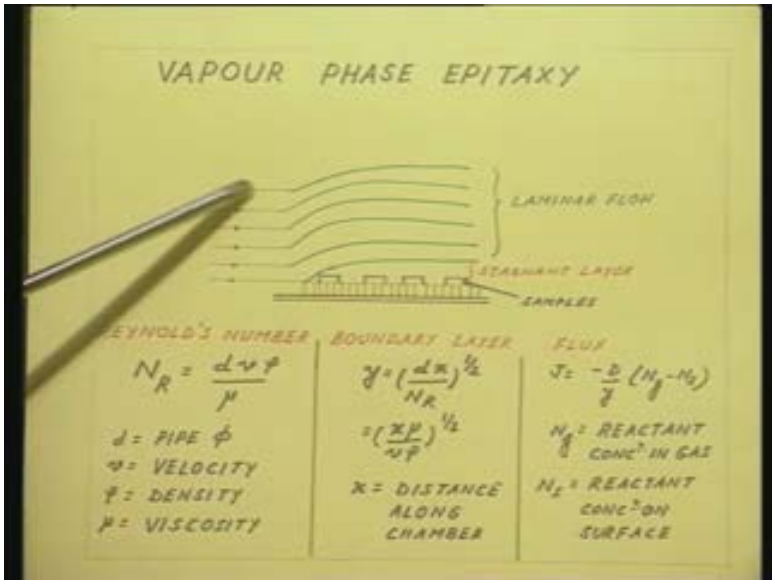

Essentially, the system is very simple you know. You have a sample holder here and you have a few samples placed on top of that and you have a flow of gas. The gas is actually containing the reactants. The gas flow is controlled by mass flow controllers and the reactants, as they flow over the sample, they react and there will be a deposition on top of the samples. This is essentially vapour phase epitaxy. I have some gas flow, that gas is containing the reactants. That gas when they are in the vicinity of the samples, they react. They dissociate or decompose or do something and the material which I want to be deposited is formed and is deposited on the samples which are already present inside the system.

I am talking in general. In general, this is the principle of a vapour phase epitaxy. When you talk about silicon vapour phase epitaxy in particular, we will tell you what is the particular gas reactant is used and all these things. But essentially, this is vapour phase epitaxy. Now here, the vapour phase epitaxy has one, you have to understand, one particular problem associated with this whole thing. See, the gas is flowing inside with a finite velocity. At the substrate, just at the point of contact here, the velocity is zero. So, in the stream, the gases have finite velocity and at the surface boundary, the velocity is zero. So, in between there must be a region where the velocity is changing from this finite

velocity to zero velocity and very close to the substrate there must be a region where this velocity is very low.

What does all this mean? That means right next to the sample, the velocity of gas is very small. In other words, the reactants are not moving very fast. In other words, I have a stagnant layer. I have a stagnant layer of reactants. This stagnant layer is also called the boundary layer, as it is shown in the chart. So, in any vapour phase epitaxy for that matter, the width of the stagnant layer or the boundary layer is a very important criteria. First of all the flow of the gas inside the tube is characterized by Reynolds number. Reynolds number is given by N R is equal to d times v times rho divided by mu, where d is the diameter of the tube through which the gas flow is taking place, v is the velocity of gas flow, rho is the density of the gas and mu is the viscosity of this gas and this Reynolds number has to be within a certain limit. If it is less than 2000, we call the flow a laminar flow and if it is higher than that, we call it a turbulent flow.

So, in most practical VPE system, the Reynolds number is much less than 2000. It is somewhere around 100 or 200 or maximum 500, let us say. So, we can consider that this flow is a laminar flow. Now, the width of the boundary layer or the stagnant layer is related to the Reynolds number by this formula, y, that is the width of the stagnant layer is given by d times x divided by N R whole to the power half, which by substituting the value of N R from the first equation, we can write as x times mu divided by v times rho to the power half, where x is the distance along the chamber.

So, you can straight away see that the width of the stagnant layer is going to increase as we move along the pipe, which I think should be obvious from the figure also here. Now, you see I cannot do much about mu and rho, these are material properties, the viscosity and the density of the gas. x is the distance along the chamber and v is the velocity of the gas flow. These are the things which may be in my control. So, essentially what this equation tells me is this. That as I move along the epitaxy chamber, the width of the stagnant layer is going to increase. Does that matter? Yes, it matters.

Why does it matter, because the stagnant layer means gas is not, gas flow is not there. Therefore the reactants in the gas stream will get exhausted. So, I will not get epitaxial layer growth, further epitaxial layer growth. If the stagnant layer width is varying from point to point, then the growth rate is also going to vary from point to point. Along the chamber, on different samples I will have different thicknesses. So, it is very important for us to control the velocity of the gas flow in order to make sure that the stagnant layer thickness is within acceptable limits.

Finally, we have the equation governing the flux. The flux is simply given by, you know, there will be a movement, there will be directional movement when there is a concentration gradient. So, this is the concentration gradient N g minus N s divided by y. N g is the concentration of the reactants in the gas, N s is the concentration of reactants on the surface. So, you see, across this boundary layer, on one side I have N g that is in the gas stream and on the surface, I have the concentration as N s. Therefore, the concentration gradient is given by N g minus N s divided by the thickness of the boundary layer. So, the flux is going to be proportional to this and this is the proportionality constant, d or the diffusion constant, also as sometimes it is called and the negative sign signifies that the flow is from the higher concentration side to the lower concentration side.

So you see, if the deposition has to take place, then this flux must be high. There must be a movement of the reactants on to the surface. So, again the boundary layer thickness must be made small and you have seen from the second equation that the boundary layer thickness can be made small if I play with this velocity of gas flow, but remember within a certain limit. I cannot allow the Reynolds number to go beyond 2000 into the turbulent flow region. But, then again as I said in any practical VPE system, it will be way below that restriction. So, when you are designing an epitaxial system, these are the things we must keep in mind, the problem of the boundary layer. Apart from that, we must ensure that there is a very high level of cleanliness. That is ensured by again taking proper care of the chamber material.

The chamber is made of very high purity quartz and the gas inlets and outlets are with seamless stainless steel pipes. The reactants may need a carrier gas and 6N purity hydrogen is usually used as a carrier gas and the heating system, it can be again either RF heated or resistance heated. But, one prefers RF heating, because then you can have cold wall reactors. That is preferred to resistance heating which will give rise to hot wall reactors.

Hot walls means there will be contamination, very simply. If the reactors are cold, reactor wall is cold, then the contamination deposition tends to be on the reactor wall. If the reactor wall is hot, then contamination deposition tends to be on the sample itself. That is why cold wall reactors are preferred to hot wall reactors and this is a very important point. That is when you are growing an epitaxial layer, the epitaxial layer is being grown on the bulk crystal. So you see, you have cut the crystal into thin slices, wafer. The top surface is exposed to the ambient conditions. So, the top surface is the one which is most prone to contamination. If you grow an epitaxial layer on this contaminated surface, then this contamination will come into your epitaxial layer also and it will deteriorate your epitaxial layer quality and finally the device performance will be degraded.

So, you must make sure that before this epitaxial layer is grown, there is provision for an in situ cleaning. In situ cleaning means, once I have taken the sample inside the chamber, just before the epitaxy is carried out, there must be a provision for cleaning the sample, cleaning the sample inside the chamber. I do not have to take out the sample, clean it and put it back, because while I am, this interval between cleaning and putting it back, again the surface can get contaminated. So, after I have introduced the sample inside the chamber there must be a provision for cleaning it, in situ. So, this in situ cleaning is a very important aspect. So, all epitaxial reactors should have the provision not merely for deposition of the epitaxial layer, but also for in situ cleaning before the epitaxy, epitaxial deposition has actually taken place and finally how do we sort out the boundary layer problem?

As I said, you can of course increase the velocity of the gas, but then your gas consumption will be more.

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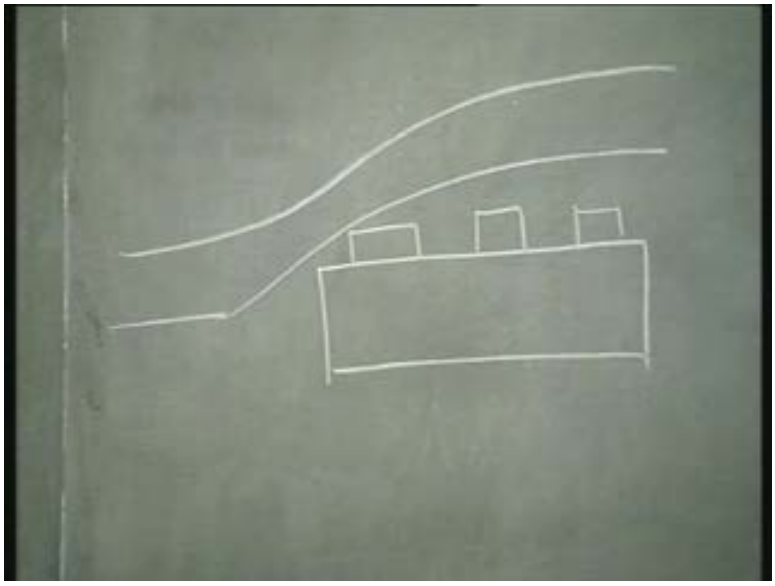

So, the other possibility is, of course, see in the figure we showed it like this. This is my sample holder. On top of that this is how the samples are provided and this is how the stagnant layer is, right. So, as I go down the chamber, the width of the stagnant layer is increasing.

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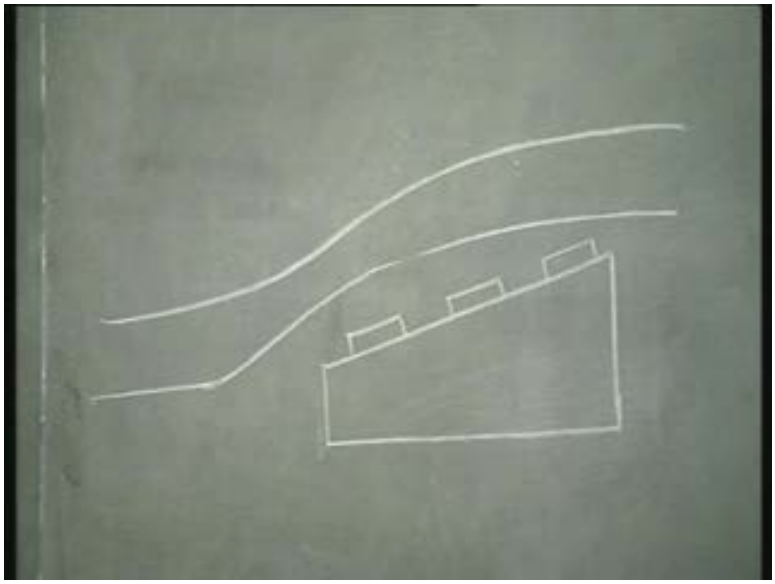

Now, let me change the sample holder design like this. I have a tilted sample holder. The top surface is an inclined plane and the samples are kept like this, so that as you go down the length of the tube the samples are placed higher, so that the effective width of the stagnant layer remains more or less the same, so as to ensure equal growth rate for all the samples. Particularly when the gas flow is parallel to the sample surface that is in this reactors which are called the horizontal reactors, this is almost universally the sample holder design that is accepted in order to reduce the boundary layer problem. That is you have an inclined sample holder and with this background, we can go over to the epitaxial reactor configuration.

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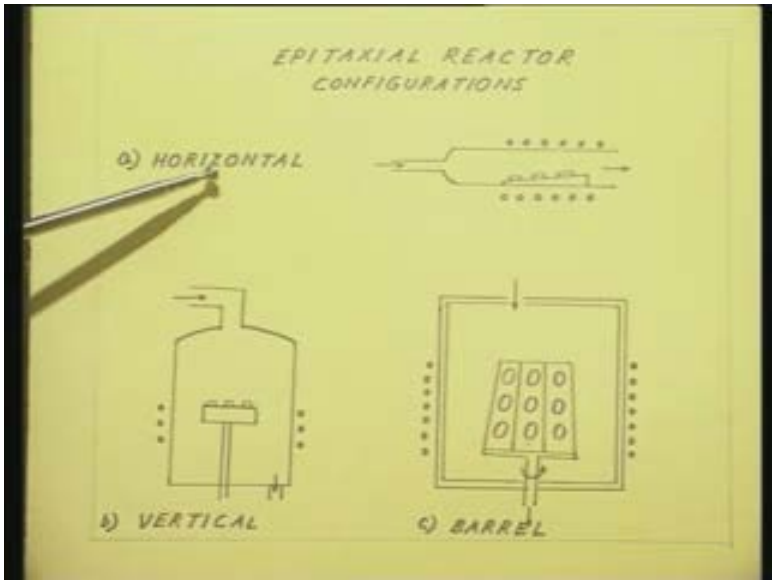

The first reactor is a horizontal reactor which is just like the one I have drawn already on the board. Notice that the gas flow is parallel to the surface of the sample and the samples are placed on an inclined plane. This is a vertical reactor, figure number b, where the gas flow is in a direction normal to the surface of the sample, it is perpendicular to the direction of the sample. Here the boundary layer problem is less acute, but you have the problem of design complexity. The reactor design is more complex compared to the simple horizontal reactor and also in a vertical reactor you cannot place too many samples at a time and finally for better throughput, for mass production what is used is a barrel type reactor.

In a barrel type reactor, the reactor holder is a barrel or a drum and in this drum, small initials are carved out, right, small grooves are carved out and in each of these grooves the sample can be placed. See, this is the groove for placing one whole silicon wafer. This is for mass production. So, you put one whole silicon wafer in each of these grooves. You can rotate the barrel for better uniformity of deposition. Remember, a barrel reactor is also essentially a horizontal reactor, because the gas flow is parallel to the semiconductor sample surface. In a vertical reactor, the gas flow is in a direction perpendicular to the semiconductor surface. In a horizontal reactor, it is parallel to the

semiconductor surface and even in a barrel reactor, it is parallel to the semiconductor surface. So, these are three basic reactor designs. In a barrel reactor of course, you can put a lot of wafers, at the same time you can achieve better uniformity by rotating that barrel and that is most universally used for commercial epitaxial layer production. So, this is what vapour phase epitaxy is, in general; this is what vapour phase epitaxy is.

In tomorrow's class, we are going to discuss the vapour phase epitaxy of silicon in particular. That is now you know, what are the problems of vapour phase epitaxy is and based on these problems how the reactor has to be properly designed. With this background, we are going to discuss the particular chemical reactions involved in a silicon vapour phase epitaxy.