

A Brief Introduction to Micro Sensors
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Lecture – 13
KOH etching

Anisotropic Etching

Anisotropic Etchants


commonly used:

- (1) 30% KOH at 70 - 80°C
- (2) Ethylene Diamine Pyrocatechol (EDP) at 115°C
- (3) Tetra Methyl Ammonium Hydroxide (TMAH) at 70°C -90°C

They are direction dependent etchants of Silicon

Typical values of relative etch rates by KOH for the three directions are

$(111) : (100) : (110) = 1 : 400 : 600$



Hello, so today we will start from Anisotropic Etching. So, in the last class we have talked about isotropic etching, where in all the directions the material was etched with the same rate right. And now, we will see that the directionality of the crystal arrangement of silicon will have an effect in its etching. So, in that case we will use actually KOH, like KOH is a very popular anisotropic agent.

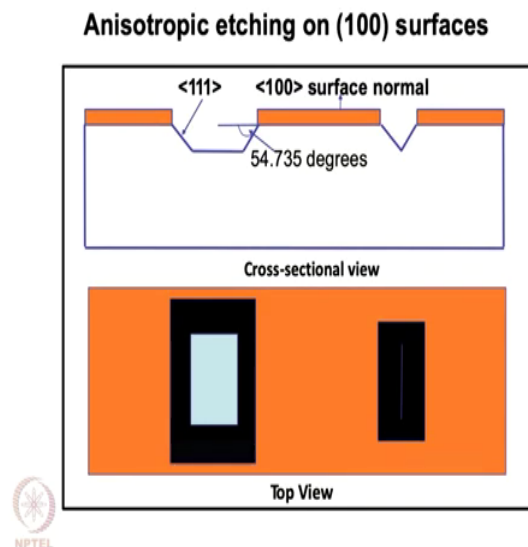
So, mostly used one of the mostly used agent is 30 percent KOH at 70 to 80 degree centigrade another etchant is EDP at 115 degree centigrade. And then we have also tetra methyl amino ammonium hydroxide which is also called TMAH and at 70 to 90 degree centigrade.

So, we can use all these three chemicals for etching silicon, but there are other chemicals also which you can use, but these are mostly used actually KOH and TMAH are the most popular one for silicon anisotropic etching. Silicon etching here like a, these etchants are dependent on the direction ok. So, what happens is along the 1 0 0 or 1 1 0 or 1 1 1, the etching rate is not same.

So, typical values of relative etching rate is given here as you can see that 1 1 1 is to 1 0 0 is to 1 1 0 is 1 is to 400 is to 600 and that difference is pretty huge. So, as you know from our crystals structure itself that in the 1 1 0 direction 1 atom is connected with the bulk with only 1 bond right. So, this is like more probable case, like or the most probable case where the atom can easily get removed from the surface. So, in that case the etching rate is highest like 600 whereas, 1 0 0, it is medium like 400 and for triple 1 case one atom is connected to the bulk with three different bond.

So, it is the most will plane and in that case it is only one. So, it means that if I etch it let us say for 1 hour if I get 1 nanometer for 1 1 1 plane for 1 0 0 and 1 1 0, we will get 400 nanometer and 600 nanometer of etching. But, what we need to remember here that; though the etching rate is pretty small, but still the 1 1 1 plane also get etched it is not 0.

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As I am saying an anisotropic etching. What does it mean? That you can see from this picture. There you can see that; so, this surface is 1 0 0 surface, means it is normal at the 1 0 0 direction. As it starts etching; as it starts etching then, this plane; let us say if I want to draw the surface here, then this surface also will be 1 0 0 surfaces right.

And these surfaces also will have higher etching rate, but before that itself 1 1 1 plane comes. And from our previous slides; we have seen that the 1 1 1 plane makes an angle of 54.74 degree almost with the 1 0 0 plane. So, in that case this is the this angle is 54 degrees; 54 degree angle. And in this 1 1 1 plane the etchant will move very slowly because, once the 1 1 1 plane is open then the etching rate in this direction goes very slowly right. Etching rate in this direction goes very slowly, so, that is why this surface opens up.

And how much is the depth accordingly you can form this kind of u shape or like v shape pattern where the bottom edges both the edges can merge also, depending on the opening. Like here, it will merge at a longer distance; like here it will merge at a longer distance right. So, it after going even more in that silicon bulk then it will merge both the side surfaces. But here, it has merged even much before that because, the opening is pretty small. And this region here at the masking layer which will not get etched by the KOH etching.

And if we see after etching; if we see it in the under the microscope, then we will it will look something like this where, this is corresponding to this image is corresponding to this opening which is the cross sectional view. And this region will be the flat region; where I can, where we can easily see the light reflected from the top surface also like get reflected easily. But, this black regions at this slopes right, this black regions at this slopes; where the light is not represent, so it is like this.

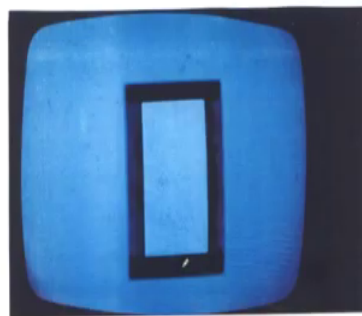
So, from the bottom the light is easily reflected, but from these region light gets reflected to some other place. So, this looks black ok. I will show the real picture in the next slide and then. So, in this case you will you will you can see the bottom side of the sample which is open and which has a lesser in dimension from the top opening whereas, here you will see

only the line because all the 1 1 1 planes have submerged from all the size the 1 1 1 planes have submerged. So, you will see only this kind of state line

And point to be noted here that; you see that the length of the line is also small right. It is not the complete length, complete length is complete length is this much. But here the length of the line is small because, here also we have 1 1 1 planes open. Because, like from this opening it is coming, so 1 1 1 plane and this 2 sides also it will go as 1 1 1 plane.

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Photograph of the etched region showing the sloping side and the bottom surface



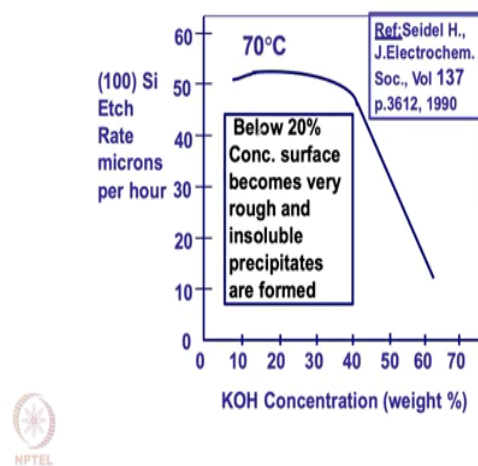
And here now we can see the photograph of real photograph of the etched surface right. There, you can see that this is the top surface, this is the bottom surface and this is the top surface which both are reflective because the light is coming from the from directly gets reflected from the bottom surface as well as the top surface. But this slopes are black, this

black regions are the slope where from the light comes there and then reflected in some other direction.

So, this looks black right and here and the dimension of the bottom surface is also much lesser than the opening that also we can see. And this dimension actually can be calculated from the geometry from this image. So, that will come we will discuss later.

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KOH concentration Effect on Etch Rate



Now, this graph is showing that; how 1 0 0 plane of the silicon etching rate etching rate in 1 0 0 plane changes with KOH concentration. So, as you can see here, according to this graph that for lower concentration we have a very high etching rate right. For lesser concentration we have a very high etching rate it does not change much for till certain concentration, but after that it drops down very fast right.

So, concs below 20 percent concentration surface becomes actually very rough and insoluble precipitation also are formed. So, if it is below 20 percent then, the etching rate is very high it is very aggressive rise etching and in that case; because of aggressive etching the surface also becomes very rough right. So, that the RMS roughness or the we calculate the roughness of the film will be very high. And in that case, some reaction product will be there which will be which will in like precipitate, because that those are insoluble in the solution.

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KOH etching

H₂O has high dielectric constant (80). This polarizes KOH into K⁺ and (OH)⁻. Silicon is oxidized by reacting with (OH)⁻

If KOH Conc is increased, the fraction of (OH)⁻ ions is inhibited and reaction rate falls

Similarly, adding isopropyl alcohol reduces the (OH)⁻ formation and hence etch rate reduces. This is due to the lower dielectric constant of isopropyl alcohol

Etch rate goes up with increase in KOH temperature



So, how KOH etching exactly works? So, H₂O has a high dielectric constant about 80. And it polarizes KOH and it so, it become K plus and OH minus ions like it ionizes to into it is cation and anion. Now, silicon is oxidized by reacting with this OH minus ions right. If KOH concentration is increased the fraction of OH minus is inhibited and the reaction rate falls. Because, as we have a very smaller concentration more number of ions we will get whereas,

if we add more and more KOH then the ions will be lesser like the K^+ and OH^- ions will be lesser.

So, in that case, the reaction rate will fall. Now, we can add isopropyl alcohol or IPA that also reduce OH^- formation and hence the etch rate reduces. This is due to the lower dielectric constant of the IPA. And another point; important point is that, etch rate goes up with increase in KOH temperature. So, if we increase the temperature of the solution then the etching rate also increases.

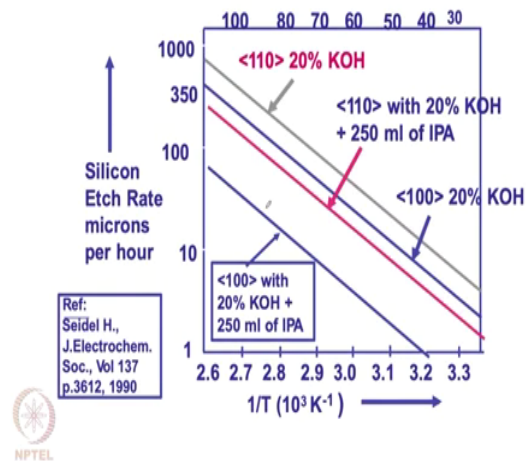
So, the important to note here is this concentration is one kind of control which we have, which we can select and accordingly can choose how much etching rate we want. Then adding IPA is another kind of control which you can which we can add and in specific concentration and then we can control the etching rate temperature is also another control knob or another handle for them. And why we need to control the etch rate, because in some kind of application; we will need very small etching, very small amount of etching let us say 2 nanometer, 3 nanometer etching or lesser 10 nanometer etching and very fine etching like the surface should be very small surface should be very smooth.

So, in that case, we can use very slow etching rate. So, that the surface will be smooth or in some cases, where we need like we need to etch probably 1 micron or 1000 nanometer right and in that case initially, for the for let us say for first 900 nanometer. We can go with very high etching rate, keeping the concentration higher.

So, in that case, surface will be etched very like very fast and it will be little bit of rough, but after that the later on we can use even higher etching rate or you can use IPA to reduce the etching rate and accordingly, we can generate smooth surfaces. So, the final surface will be much smoother and also we will we can finish the process faster.

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KOH concentration Effect on Etch Rate



Now, in this graph, we are showing different effect of different parameters like temperature, concentration and different planes etcetera and IPA also on like 1 KOH etching. So, in this case, all the cases if you see the first case is 1 0 0 plane with 20 percent of KOH right and 250 ml of, 20 percent of KOH and 250 ml of IPA. And this is how with temperature the etching rate changes and you see this is the this is plotted with 1 by T.

So, with temperature the etching rate actually with increasing temperature; the etching rate actually increases, because in this direction 1 by T is increasing; that means, T is decreasing. So, as we are decreasing the temperature etching rate falls right. Now, 1 0 0 plane with 20 percent KOH and 250 ml of IPA has the lowest possible etching rate then, 1 1 0 plane with the same KOH concentration.

So, then we can see that 1 1 0 plane have the higher etching rate than 1 0 0 plane right with the same KOH concentration and same amount of IPA. Next case is, where we have the same 1 0 0 plane has same percentage of KOH, but we are not adding any IPA. So, as we discussed earlier that adding IPA reduces the OH minus ions in the solution.

So, now we have more number of OH minus ions in the solution. So, this will oxidize that silicon even faster. So, the etching rate is even higher. So, etching rate is not only higher than 1 0 0 plane for that condition it is also higher than now 1 1 0 plane which was etched using IPA. And if we now, use the same condition for the 1 1 0 plane like same 20 percent KOH without IPA, then 1 1 1, 1 1 0 plane also have now the highest possible etching rate it is higher than all the other cases.

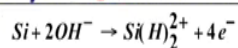
So, there we can see that KOH concentration is already a parameter what we discussed in the previous slide, but that IPA as well as the temperature and the directionality of the which direction we are etching, then all of these are different different parameters for controlling etching rate.

Now, in that case, actually one thing to I would like to mention that if IPA we can select that whether we would like to add or not, temperature we can decide, but usually the substrate we are bound by the design right. What kind of if the design of my device needs 1 0 0 plane then you have to use 1 0 0 plane. If it is 1 1 1 plane then we need to use 1 1 1 plane because there are different parameters which will also depend on this crystallinity. So, that is not on our hand much, but we can select different kind of temperature and give which percentage to control the etching rate.

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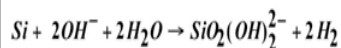
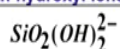
Reaction of KOH with Silicon

Silicon reacts with $(OH)^-$ and gets oxidized, and 4 electrons are ejected from each Si atom



Simultaneously, water is reduced, leading to evolution of H_2

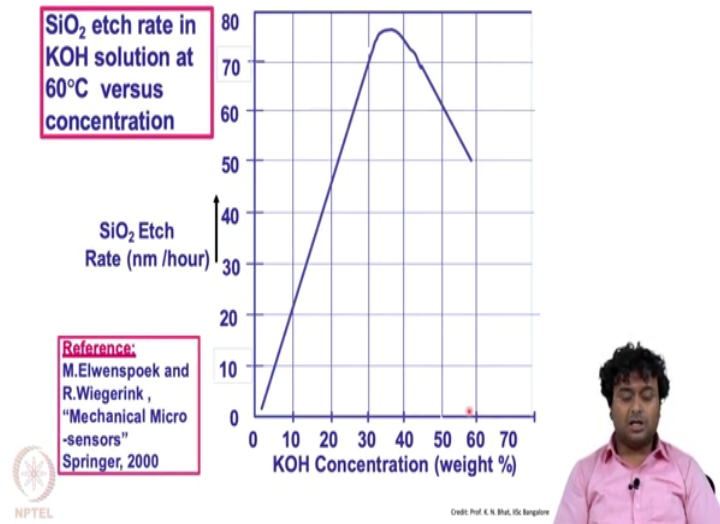
The complexed Si further reacts with hydroxyl ions to form a soluble silicon complex



So, what are the different reactions what actually happens while silicon is H plus KH? First of all the OH minus ion oxidizes the silicon at and it forms this silicon hydride element. Now, simultaneously water is reduced also like OH minus and OH plus is also there. So, water is reduced leading to evolution of H_2 . The complex silicon further actually reacts with hydroxyl to form a soluble silicon complex which is silicon dioxide with 2 OH minus ions right.

So, this is the final product with very with easily dissolves in the solution and so, and the silicon gets etched. So, this is the final reaction is silicon plus hydroxyl ions which are coming from the KOH and water it you will get this dissolvable reaction product and with that hydrogen.

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From silicon we move to silicon dioxide etch rate with KOH. And silicon dioxide etching rate with KOH is also very much important, because most of the cases we have so, first of all silicon has always some native oxide layer on top which is the silicon dioxide, because it gets exposed to the moisture and then the dangling bonds easily reacts with the water and it forms kind of a native oxide layer. This silicon dioxide also is used as the masking layer.

So, in the previous one of the slides on like this slide; we have shown this masking layer right. So, which is, which will not allow does silicon to get etch below it right and this is this can be actually silicon dioxide. So, we can put silicon dioxide on and some according to our design. So, how we can do that using lithography that; we will discuss in some other class, but the point is here that silicon dioxide can act as an oxide as an etched stop or the protective layer or the masking layer.

As I was saying that even though the masking layer have for the masking layer or the protective layer; the etching rate is small, but still there is some amount of etching. So, like here you can see that the silicon dioxide have a etching rate of like below 70 or to 80 nanometer for different different KOH concentration. And for different KOH concentration the etching rate varies, but it is below 70 to 80 nanometer.

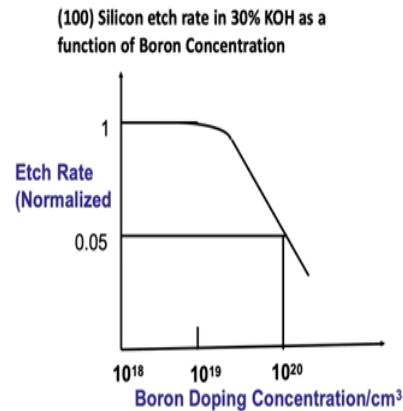
Now, if you go back and see it for KOH for silicon then, we see that the etching rate here is given in terms of microns per hour. It is, you can see that this is like 40 or 50 microns per hour. Whereas, for SiO₂ or silicon dioxide it is 60 70 nanometer per hour. So, the etching rate for SiO₂ is almost 1000 times smaller than the silicon.

And this is a very important point, because we can use, we can use silicon dioxide as an protective layer and let us say; if we are etching for one micron in that period in that time 1 micron of silicon let us say we need to etch it for; let us say we need to etch it for 10 minutes for 1 micron of silicon etching.

Then in the 10 minutes SiO₂ we will get etched only likes 5 6 nanometer right. Only 5 6 nanometer of h SiO₂ will get etched in that 10 minutes. So, that much of oxide layer is good enough to protect the silicon layer. Because, SiO₂ layer will protect the silicon layer until it is by itself it get etched by the KOH right. So, if we deposit a like 100 nanometer thick silicon dioxide layer. So, let us go to; let us go back to this picture again. So, this is let us say a silicon dioxide and I am etching about 1 micron of silicon.

And for that let us say we need for that we need 10 minutes of time. Then in that case, this silicon dioxide can be up, let us say 50 nanometer or 100 nanometer also. Because, in that 1 hour this will get etched only by 5 6 nanometer. So, thickness also we will get reduced. Let us say this will come to some place; this will come to some place like this right. Little bit lesser the thickness of what it started with, but ultimately it will; it will still protect this region, it will still protect this region. So, silicon will not get etched right. So, SiO₂ has a small much smaller almost 1000 times smaller etching rate on the KOH etching compare to silicon.

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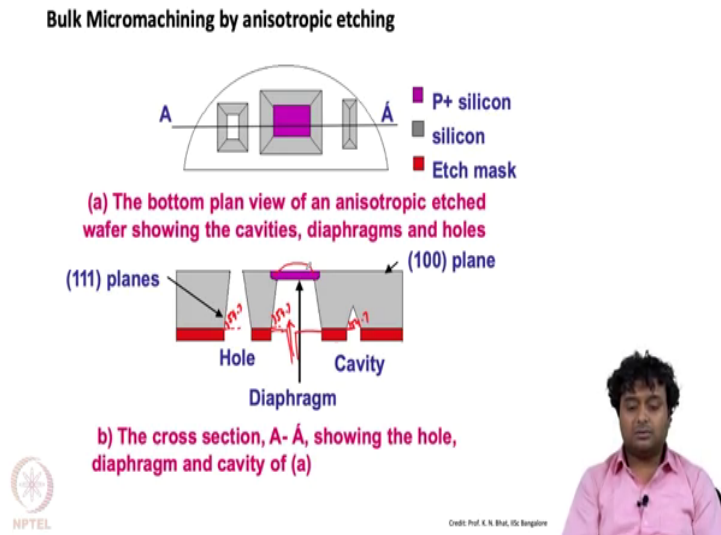
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Now, the next way to control the etching is boron doping. In case of boron doping, what happens is initially, let us say if it is 10^{18} like 10^{18} atoms per centimeter cube. And in that case, we have the usual etching rate of KOH like without doping or with doping it does not matter much. But as we increase the doping rate like 10^{19} to 10^{20} , then the etching rate drops down drastically.

You can see here that; this is normalized with respect to the maximum etching rate and their it stays at 1 for certain till certain doping concentration, but after that it drops down drastically. So, we can also; we can also just dope boron in certain region to reduce the etching rate in those regions. So, then that case, that will act as a masking layer.

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And now, we will see that; how we can use this KOH etching for creating different different geometries. And we will use the etched of layer also for creating this kind of geometries. So, let us assume that this is a silicon wafer like half of the silicon wafer, you can see this half circle right and there we have three different opening. Here, we have a small opening and then we have a little bigger opening and then again, we have a small opening right even smaller than this the first opening.

Now, in this case, the first case; if the cross sectional view is drawn in the bottom figure here. Consider, this is the cross sectional, this is the cross sectional view of the same geometries here. This is the this right side is the etching like the masking layer and we have etched the KOH, we have etched the silicon from this direction from the bottom right from the bottom and then this is the hole opening and this is the bottom hole. So, let me just right down here.

So, this is let us say; if it is 1 0 0 plane then, this is the angle which is 54.7 degree ok, 54.7 degree. All of this angles of 54.7 degrees, but this angles are 54.7 degrees.

Now, with this angle here, the silicon wafer thickness is such that; that I will get the through hole. So, this is completely. So, light can pass from the top to bottom right. This is the through hole then, in the next case; the hole size is even bigger. The opening is even bigger. So, there also I should get a through hole, but what we have done in the other side or the opposite side where we are etching we are etching from the bottom side right and when the opposite side or the top site we have doped boron in this region.

So, as we have doped boron the etching rate in this region becomes the smallest. So, the KOH will not be able to etch in this region of silicon. This region of silicon will not be etched by KOH. So, this becomes like a thin membrane; this becomes like a thin membrane. So, if we have let us say 500 micron thick of silicon wafer and then this region can be like 50 or 100 nanometer which is boron doping has happened and odd even little higher. Let us say 100 or 200 nanometer.

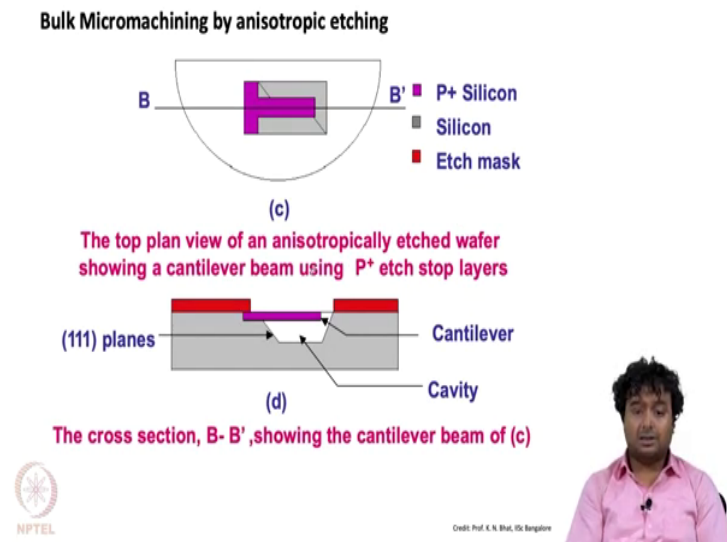
So, in that region we get a very thin like 500 micron nanometer or 1 micron thick membrane which will not get etched. Whereas, this is the smallest opening hole, so here both the sides meet much before it can reach the bottom side of the plate. So, this through hole, this membrane and this v shape hole all are made using the same KOH etching by just using A etched stop layer here.

And different kind of hole opening and this different different geometries like membrane, through holes or even this v shaped hole have different different applications; which some of some of which we will see in the in the next module. But, this like one kind of a like one kind of application for the membrane is this is very much useful for pressure sensor.

So, we can connect pressure port here; can connect a pressure port here like this. And then, if the if the air pressure is applied here then, this pressure will build up and that will make the membrane into bent like this; that will make the membrane to bent like this and then we can we can measure the deflection of the membrane by some characterization technique by like

optical or electrical and can measure that; how much is the deflection and also how much is the pressure of the like pressure of the chamber which I want to measure. So, that this things we will discuss in the in the next module.

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So, this is another application. So, we have seen in the module 1 that we have used cantilevers for different kind of sensors right. Now, how you can make a cantilever? We can make the cantilever such that; that the only in it using the cantilever geometry this region this violet colour region or pink colour region is only boron doped. So, this region will not get etched, but all the sides will get etched. Now, all the sides as it get etched cantilever varies not much. So, the material or the liquid can go below it.

So, like this is the cantilever, let us say; this is the cantilever and then this is only doped with boron. So, this will not get etched, but all the sides are open. Now, from this side the liquid

can like sink in and can etch the bottom side of the silicon of the bottom of the cantilever; which is used like the normal silicon. Because, the boron doping only will be in top view nanometers; like few 100 nanometers such like that. So, it will not go at the bulk right; it will not go at the bulk. So, that bulk silicon can get easily etched.

So, ultimately, we will have; let us say this regions, the other regions are also boron doped. So, this will also not get etched, but from bottom of the; from bottom of the cantilever the material will get etched. So, now, this will be like a free cantilever this will be; so now, this will be like a free cantilever. It can easily bend because it has a vacancy or empties empty space below it. So, it can easily bend and this kind of device we can used for different kind of sensors.

Thank you.