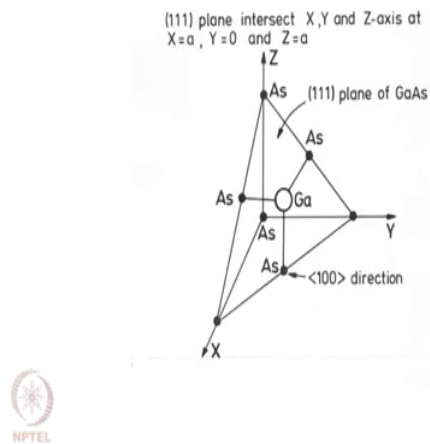


A Brief Introduction to Micro Sensors
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Lecture - 12
Si etching

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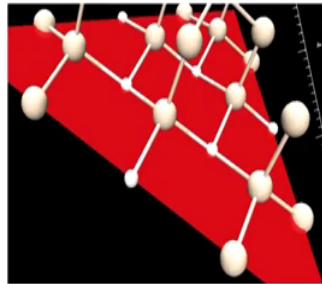
Truncation of GaAs cube with $\langle 111 \rangle$ plane



So, as we are discussing the crystal structure of silicon or gallium arsenide we have seen that and in different directions like whether you were taking 100 110 or 111 you have different kind of crystal arrangement. What does it mean? Different kind of crystal arrangements means that how many atoms or 1 atom is connected to how many atoms inside the bulk that number is different.

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(111) family of Planes



- (111) Plane intersects X,Y and Z axis at $X = a$, $Y = a$ and $Z = a$
- Each atom is tied to the substrate with THREE bonds
- One dangling bond and three bonds connected to the bulk are present per atom



Used Crystal Viewer Tool@ Nanohub



So, now we will see that how it looks exactly in real life like, let us say this is a crystal structure so you have at different corners atoms are sitting and then you are cutting it with 111 plane that means, you are cutting it like this somewhere. So, if this is X axis, this is Y axis and let us say this is Z axis; then X axis Y axis and Z axis all the axis are cut by this plane at 111 or a a a right.

Now, as you can see so this is this plane is 111 plane and it is cut X Y Z axis in like it is X equal to a, Y equal to a and so there is a mistake this is this will be Y equal to a ok. This direction or the plane has cut the XY plane in along this line right and this is 100 direction this is 100 direction, right.

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The angle θ included between two crystallographic directions $[u_1 v_1 w_1]$ and $[u_2 v_2 w_2]$ is given by

$$\cos \theta = \frac{u_1 u_2 + v_1 v_2 + w_1 w_2}{\sqrt{(u_1^2 + v_1^2 + w_1^2)(u_2^2 + v_2^2 + w_2^2)}}$$

Angle between (111) and (100) in degrees is
 $\theta = \cos^{-1}(1/\sqrt{3}) = \cos^{-1}(0.5773) = 54.735^\circ$



Now, to calculate the angle between 2 crystallographic direction like u_1, v_1, w_1 and u_2, v_2, w_2 from coordinate geometry we can see there this is the formula like $\cos \theta$ is equal to $u_1 u_2 + v_1 v_2 + w_1 w_2$ divided by their own distance is this actually you know that if there are 2 vectors a and b then $a \cdot b$ gives you $a \cdot b \cos \theta$ right.

So, $\cos \theta$ is equal to $a \cdot b$ divided by a into b like the mod of a into mod of b . So, this is just like this the same formula right. So, this is the dot product and this is divided by their own length, correct. Now the angle between then, the angle between this 111 plane and 100 plane is but like it will be 1 it will be on the top side it will be 1 and they there you will have like 3 into 1. So, it is 1 by 1 by root 3 that will give you 54.735 degree.

So, now the point is that while you are having this let us say this is the block and this is let us assume that this is the 100 plane right, this is the 100 plane then this 11 plane 111 plane will

cut like this and these angle like what the 100 plane and 111 plane has made this angle is 54 degree or 54.73 degree.

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Line Describing the intersection of the planes
 $(u_1v_1w_1)$ and $(u_2v_2w_2)$ is $[uvw]$ given by

$$u = v_1w_2 - v_2w_1$$

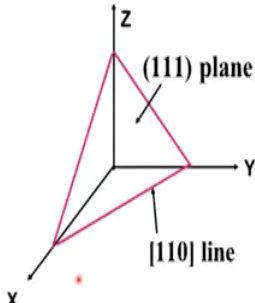
$$v = w_1u_2 - w_2u_1$$

$$w = u_1v_2 - u_2v_1$$

(111) and (001)

planes meet on a **[110]** line

XY plane is parallel to (001) plane



Credit: Prof. K.N. Shrivastava, IIT Bombay

So, as we are discussing this is the 111 plane and then this 111 plane intersects this 111 plane intersects the like XY plane at 10 line right at 110 line correct because here if you see then if you draw a if you draw a plane at 1 at this 110 line then it will be parallel to the Z axis right.

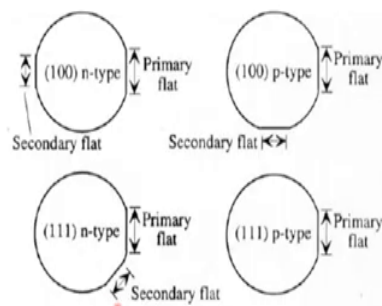
So, this is 110 line where it is intersecting and another thing is so as we already know that XY plane is parallel to 001 plane right because at 001 plane it has cut the Z axis at 1 or Z equal to a let us assume, then XY plane is parallel to the Z plane and another point very important point in this regard is like 100 plane let us say this plane then 010 plane and 001 plane will be the two different plane which will be perpendicular to this 100 plane right. So, this plane if it is 100 plane then there are multiple planes we can draw like infinite number of planes which

are perpendicular to the 100 plane right like this plane is also perpendicular, this plane is also perpendicular, this plane is also perpendicular.

But in that, there are only two planes which will be like the which will be the basis plane right. So let us say, if this the if this is 001 plane which has cut the Z axis and then this is 100 then this will be 010 right. Now, the important point is that all of these plane are called like 100 plane family and crystallographic direction wise they all have the similar kind and the crystallographic direction wise they all have similar kind of property similar kind of material property. Like the way we are discussing earlier that in different direction; in different direction the atoms are connected to the bulk by different number of bonds and in this family of planes like a plane an atom which is in 100 plane let us say the crystal is cut in 100 plane then the atom at the surface will have same property if the crystal was cut in 010 plane or in 001 plane.

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**Flats on Standard Commercial silicon wafers
used to identify crystallographic orientations**



Credit: Prof. A. N. Bhattacharya



Basically, while the silicon wafers are prepared or manufactured they are manufactured in a cylindrical shape like in ingots. So, the single crystal silicon ingots are first formed like it is it will look like a cylinder and then different slices like circular disk are chopped off from the cylinder, so these are called wafer. These are usually like 400 500 micro meter of thickness and it can be its diameter can be 2 inch, 3 inch, 4 inch, 6 inch or even 12 inch diameter, so these are like thin very thin plates.

Now, as we discussed the different planes have different kind of material property right, the atoms represent in different property have different kind of material property.

Now, 1 ingot once prepared ingot is like a cylinder as I am saying then it can be cut in different direction, whether we are cutting at 100 direction or 110 direction or 111 direction depending on that the silicon material will have different kind of properties right and to understand that we introduced this primary flat and introductory and secondary flat. What is primary flat?

So, there is a long cut, so this is not properly circular that there is a long cut at the edge which is called primary flat and then there is another small cut which is called secondary flat. Now, for 100 wafer for 100 wafer and n-type wafer you also are aware of the term doping where we can dope the silicon semiconductor to be p-type or n-type, p-type semiconductor will have more number of positive charge carriers or holes and n-type semiconductors have more number of negative charge carriers or electrons.

Now, for a 100 cut and n-type silicon the primary flat and the secondary flat are exactly opposite way. So, by this; this seeing this arrangement without even asking we will know that this is 100 n-type silicon. Similarly, if the secondary flat is present at the perpendicular direction to a primary flat then it is 100 p-type, it is 100 p-type.

Again if the p the primary flat is very close to the secondary flat, then it is 111 cut and this is n-type right and if there is no secondary cut no secondary flat then this is 111 p-type. So, like this we can identify which kind of silicon wafers our sample is.

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Relevance of crystal planes and 'primary flat' to Bulk Micromachining

- The primary flat is provided passes through the 110- line.
- The (111) plane intersects the (100) on the 110-line.
- (111) is the cleavage plane of single crystal Silicon. Scribe through the line parallel to the primary flat and then press the wafer gently, it will cleave along the (111) plane
- As we shall see later, this primary flat is also used to align the patterns for anisotropic wet chemical etching of silicon



So, now we will discuss about this flat and one important thing, I will go back to few slides back like where I have shown that 111 plane intersects the XY plane or the let us say 001 plane you can call it 001 or 110 100 plane in the along the 110 line and the cut what we make on the silicon wafer is along that 110 line and why is that? That we will come later, but currently already from the crystallographic orientation as we have discussed you know that 111 110 plane has very now like 1 atom on 110 plane have only 1 atoms connected to the bond or the bulk.

So, this is more this is most unstable plane whereas 111 plane or the triple 1 plane is the most stable; most stable plane right because 1 atom is connected to the 3 atoms at the bulk. So, while we make the cut; the cut is automatically formed like the open surface at the cut is 111 plane and the along the line 110 line actually the cut is made. So, about that we will discuss

while we go to actually etching and cutting so, this is one thing. So, 111 plane intersects the 100 plane at the 111 at 110 line ok.

Now, the 111 plane is called cleavage plane because if we scribe through the line parallel to the primary flat or even perpendicular to the primary flat and press the wafer gently then the wafer will break along that line because along that line we have already the 110 line which is most fragile and then it meets the 100 line like the 1 or triple 1 plane which is which meets 100 plane at this line. So, it will just break at that point.

So, as we see while we will see the lithography that time also we will discuss this that usually the primary flat is also used to align the pattern for anisotropic wet etching or anisotropic chemical etching.

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In Summary

- Atoms on the (110) Plane of Si are bonded to the next (110) plane with 1-bond
- Atoms of (100) Plane of Si are bonded with 2 – bonds to the next (100) plane of the crystal
- Atoms of (111) Plane of Si are bonded with 3 – bonds to the next (111) plane of the crystal

As a result of the above binding strength is highest in the (111) direction, it is considerably lower along (100) direction and the lowest along the (110) direction



In summary, like the atoms on the 100 110 plane of silicon are bonded to the next 110 plane with 1-bond or only 1 atom is bonded. Atoms of 100 plane of silicon are bonded with 2 - bonds to the next 100 plane of the crystal and atoms of triple 1 plane of silicon are bonded with 3 - bonds to the next 111 plane.

As a result of the above as a result the binding strength in 111 direction, is the highest whereas, it is considerably lower for 100 and the lowest in case of 110 direction. And because of that the etching rate or the damage of the wafer along the 110 direction is the highest whereas, 111 direction it is the lowest.

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Micro machining Processes

- Bulk micromachining
- Surface micromachining



Now, micro machining processes, in that there two specific different process: one is called bulk micromachining and another is called surface micromachining.

So, before we go to micromachining we need to understand one thing that is currently in macro scale we make different devices or different structures and for that we have already some specific processes are set up in industry right like you can if we want to make this so if we want to make this then this has some kind of mould where the material will be put in that mould and then it can be made from that or also like if we want make some particular kind of design on that then accordingly we can cut it or we can polish it or we can we can scribe it to make exactly that pattern.

But this is possible only because this is in micro; only because this in macro scale right, but if we need to do the same thing like while we are making a macro scale device or nano scale device then we need to make this kind of structures in very small line scale like micro meter scale or in nanometer scale like, in the first module we have talked about cantilevers and membranes right if we want to; if we have to make a cantilever which has a like length of 100 micron with the 5 micron and let us say the thickness is something about 500 nanometer to 1 micron.

Then using our popular macro scale processing techniques it is not possible to make this kind of devices and for that we need to use a separate kind of process flow and that is called microfabrication or micro machining. Now, in that case the material is also very important as we are discussing earlier also that the material is very much important and this silicon is chosen as the material because it has externally properties like it has good mechanical properties also and good electronic properties also.

And another important point is as we just discussing last few slides that it has a crystal single crystal structure right and in that single crystal structure it has a very defined property in each of the direction and accordingly we can make our device according to our requirements. So, let us first go to micromachining bulk micromachining.

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Bulk Micromachining

- Start with bulk Silicon wafer
- Remove(etch) material until a final surface required is created
- Wet chemical etching , etch-stop layer and Wafer Bonding methods are used
- Dry Etching - RIE



So, in bulk micromachining we start with bulk silicon wafer. So, as I was saying that for silicon like manufacturing of silicon is done by processing this ingots right which is cylindrical in shape and then we cut the ingots parallelly, slice by slice and then each of these slices are used as a for like microfabrication or as silicon wafer.

Now, we can remove material until the final service; final surface is created. So, there are different techniques which a few of them we will discuss in the part of this course that how we can remove a material and while we are removing some material let us say if this is some material and then I am removing some point some material here from the circular like some circular this kind of region I remove. Then it will create a circular hole or a hole in this block right.

So, removing material is very much important and we will see few of the processes and then we can select a particular region which we want to remove and the another region we do not want to right. So, there are some masking layers or etch stop layer which we can put where we do not want to etch. Let us say, if I want to write a pattern like a here then only according to the a pattern I will etch rest of the region I will just mask it so that will not be etched

Then there are some bulk like dry etching technique also. So, other than using only liquids only chemical liquid chemicals we can uses gases also for etching away the material. So, that is these are called dry etching techniques and RIE or reactive ion etching is one of the very popular technique for dry etching and depending on our requirement we can also bond two efforts. So, let us say we have a; we have a device such that on one device we have made some patterns and another device we have made some other patterns and for our application we need to bond it together. So, that is that also is possible using bulk micromachining techniques.

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Wet Chemical Etching: (a) Isotropic (b) Anisotropic

Isotropic etching (Non directional etching) :

Etch Rate in X and Y Directions is equal to that in Z direction (eg)

('HNA' etchant HF : HNO₃ : CH₃COOH)



- HNO₃ oxidizes Si to SiO₂.
- HF reacts with SiO₂ to form H₂SiF₆ which is soluble in water
- Acetic acid is a moderator. It has dielectric constant << than that of H₂O and hence prevents dissolution of HNO₃ into NO₃ and NO₂ and allows formation of species directly responsible for oxidation of silicon



While I am discussing about bulk micromachining, the first point is that: wet etching or wet chemical etching. In that, wet chemical etching can be of two types; one is isotropic and another is anisotropic.

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Isotropic or Non-directional Etchant Etching



(a) Isotropic etching without agitation



(b) Isotropic etching with agitation



Credit: Prof. K. N. Bhut, IIS Bangalore



So, in isotropic etching what happens that in all the direction etchant will etch the material in all the direction equally. So, let us say here this regions are properly protected with some extra layer. So this region, so this is the material or your substrate or your sample which you need to etch and this is this brown color regions are like etch stop layer. So, this will not this will not allow this regions to be etched this regions will not be etched.

But, this regions will be etched and in all the direction if we take this direction; this direction or in the other direction all the direction the etching rate is same. So, you can see that as it has gone down vertically to some etching distance it has gone sideways also and after certain time it will start etching from the bottom of the etch stop layer also. So, this is there is under etching or there is under etching or side etching also right and in that case all of the sides are etched; are etched equally with same rate and if you change the chemical if we if; we change

the chemical and take different chemical then we can use it with some different rates, but in all the direction the etching rate will be similar will be same.

Now, we can also etch it with some kind of agitation. What does it mean? Agitation means we can sonicate it with some particular frequency. Now while we are sonicate it, we are allowing the liquid to move sideways even more freely. So, those liquids will get in contact with the surface more frequently and also whatever is the product of the reaction that also will be removed very easily. So, in that case the etching rate sideways will increase even more and we will get more etching rate

So, one of these example; one of these example is silicon etching with this HNA etchant. What is HNA etchant? HNA is hydrofluoric acid, nitric acid and acetic acid. And hydrofluoric acid and nitric acid reacts with silicon to make this SiF_6 product which is soluble in water. So, it easily get dissolved and then the NO goes out.

So, the silicon is etched by this etchant. Here what happens is actually, HNO_3 oxidizes the silicon to SiO_2 or silicon dioxide and then the silicon dioxide reacts with HF and makes this SiF_6 product which is H_2SiF_6 and this is very much soluble in water right, this fluoride is the fluoride is very much soluble in water so it easily get dissolves and accordingly silicon will get etched in all the direction equally, in all the direction in an equally right.

Here the acetic acid is a moderator; that means, that into it actually does not do any reaction, but it has a dielectric constant which is very much lesser than the ϵ two. So, it prevents the HNO_2 ; HNO_3 to break into NO_3 or NO_2 and allows formation of species that are directly responsible for oxidation of silicon. So, it does not allow or actually reduces the rate of HNO_3 dissociation disassociation dissociation ok.

So, acetic acid actually reduces HNO_3 dissociation and because of that HNO_3 will be more capable of making silicon to SiO_2 and that SiO_2 will react with the HF and ultimately will make this dissolvable product. So, accordingly H a accordingly we can get silicon etching. And here, one important point is; as I was saying earlier that, the silicon has different etching

rate in different different direction. So, then the HF reacts with the SiO_2 HF reacts with SiO_2 and forms this H_2SiF_6 product which is actually dissolvable in water.

And because of that silicon gets etched very easily with this HNA etchant and here the acetic acid is a moderator; that means, that it does not etch or does not react actually with silicon or even any by product of the reaction, but it reduces the dissolution of HNO_3 because it has a very less dielectric constant than water. So, the HNO_3 will not dissociate into NO_3^- and NO_2 into the atmosphere. So, it will be available for oxidizing the silicon to SiO_2 .

Now, another important point is here as we are saying that this HNA etchant is etching in all the direction equally, but as we have seen from; as we have seen from our crystallographic orientation class that in different direction silicon has different etching rates. What does it mean different etching rates? It means in a particular direction let us say if this is 100 direction then in that case 1 atom is connected to 2 atoms at the bulk right so and whereas, if this is an 111 plane let us say if this is 100 than this size this direction or this direction will be 111 and in that direction 1 atom is having more number of bonds or all three bonds to the bulk. So, that is more stable.

Now, if we put this material here then this is; this is 100 direction and in this direction whereas, this is actually 1 this is another 100 family where as 110 there will be there will be this plane has 11 111 which will be the less lesser etching rate and in that case it should have; it should have been; should have been that the one direction it is more etching and another direction it is different kind of etching.

But, it is not it has not happened in this case because etchant is a such an aggressive such an aggressive agent that it easily reacts with silicon and completely etch it away in all the direction. But, in the next class we will see the examples where we will use the cleavage or TMH as the cleavage or TMH for silicon etching and there we will see that in the 110 direction and 111 direction have very different etching rates and accordingly different kind of geometries are created using this anisotropic etching. Whereas, in HNA case of hydrofluoric acid nitric acid and acetic acid combination this is isotropic etching.

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HNA Etchant formulation and etch rates at 22 °C

HF	HNO ₃	CH ₃ -COOH	Etch Rate μm/min	Masking Film
10	30	80	0.7 -3	SiO ₂ 30 nm/min
25	50	25	4	Si ₃ N ₄
9	75	30	7	SiO ₂ 70 nm/min



Now, as I am saying that this HF HNO₃ acetic acid are like a mixture which is which is silicon very aggressively very easily. But depending on the different concentration it has different etching rate, like if we use 10 is to 30 is to 80 HF HNO₃ and acetic acid then we will have an etching rate of 0.723 micron per minute that 700 nanometer to 3 micrometer per minute. Whereas, if we use more HF and more HNO₃ and very less lesser amount of the C C double OH or acetic acid then in that case it will be 4 micron per minute.

And here you should understand one thing that acetic acid is like just a moderator got it or it actually just controls the reaction. So, that the HNO₃ will not dissociates dissociating into the solution, but it does not actually reacts.

So, increasing HF and HNO₃ in with respect to acetic acid actually increases the etching rate. 9 percent of HF and 75 percent of HNO₃ and 30 percent of acetic acid then we get an etch

etch rate of 7 micrometer per minute so more acetic acid is we will make the HNO_3 we will make the silicon to more in SiO_2 and that SiO_2 we will get etched by HF and we will have say more amount of silicon etching.

Here, we see that in the first case, where we have like 10 is to 30 is to 80 or 1 is to 3 is to 8 that time SiO_2 is if we use SiO_2 as masking plane then that will be also as used as 30 nanometer per minute. In the second case, we can use Si_3N_4 or silicon nitride as the masking plane the etch rate there is very small. Whereas, in the last case where we have a lesser amount of HF in that case we can use SiO_2 also as the masking plane, but here the etching rate is of SiO_2 is 70 nanometer per minute.

So, by the time it will etch the silicon to 7 micron it will etch SiO_2 to 70 nanometer. So, if we what we need to understand in this term there that; let us say; let us say I need a I need silicon to be etched about let us say 700 micron and for etching 700 microne if I am using the last recipe or this third one then, for 700 micron etching I need 100 minute of etching in this concentration right.

But, in this 100 minute; in this 100 minute this SiO_2 also will get etched till 70 into 100 that is 7000 nanometer or 7 micron and so, 7 micron etching 7 micron SiO_2 film at least is required to protect the Si from etching and say depositing 7 micron SiO_2 or silicon dioxide film is very difficult so this is not a proper solution.

And we need to so keep in mind all the time that, even though we call all one layer as masking layer all or the etch stop layer that layer also has very has a etching rate which might be very small, but depending on the type that may also get completely etched. So, next time we will talk with we will start with anisotropic etching.

Thank you.