

**NPTEL
NPTEL ONLINE COURSE**

**Tutorial-3
Materials Characterization
Fundamentals of Transmission Electron
Microscopy
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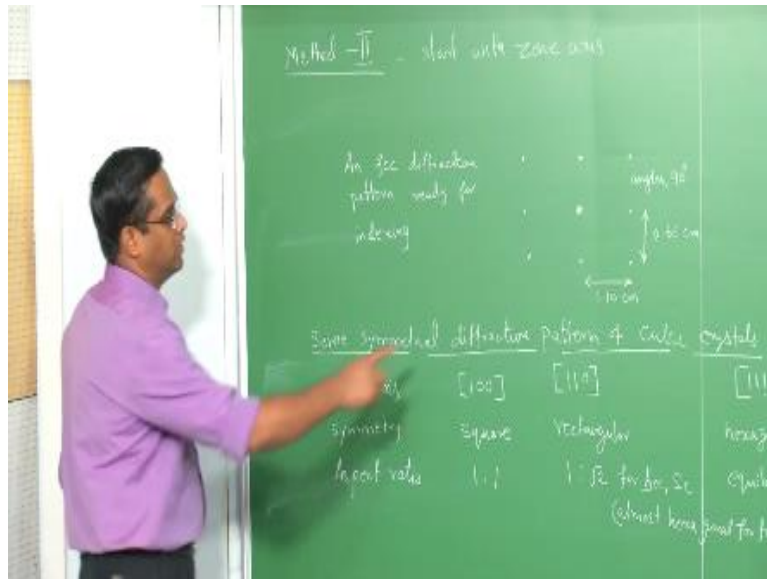
Hello everyone welcome to this material characterization course organized by NPTEL and we have been looking at some of the tutorial classes in the last two or three classes and I have been working out some of the problems which is related to electron diffraction and how to interpret them and her out to index then.

So on in the last tutorial we just looked at some of the single crystal electron diffraction pattern using one method called the ratio method. Where you just to have electron diffraction pattern you start with the pattern and then look at the different ratios you make a table and then you try to find out of these zone axis and so on.

So in today's tutorial class I would like to demonstrate indexing the single crystal electron diffraction with another method called zone access method. Here we first look at the zone axis and from there we will look at the individual planes where it cut contributes to the electron diffraction spot and we can you can also have a comparison at the end how it looks like and how they are related.

Whichever the way you choose the ultimately you are to find arrive at the correct solution you can see in the both the cases they are same and at the end I will also show some simple procedures to look at electron diffraction from a polycrystalline a specimen so let me straight away start with the second method called zone axis method.

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So what we have done is we have taken a simple electron diffraction pattern from the FCC crystal which has got the distance in this direction is 1.10 cm and this vertical distance is 0.65 cm and the angle between these two direction is about 90° and this is one pattern. Similar pattern we have taken for solving the induction in the previous tutorial also.

So that you can compare the both the methods how to see the solution in two different methods so this is a signal crystal electron diffraction pattern so well now we will see how to go about it so before I really get into the technique let me also draw some table where it says the some symmetrical diffraction.

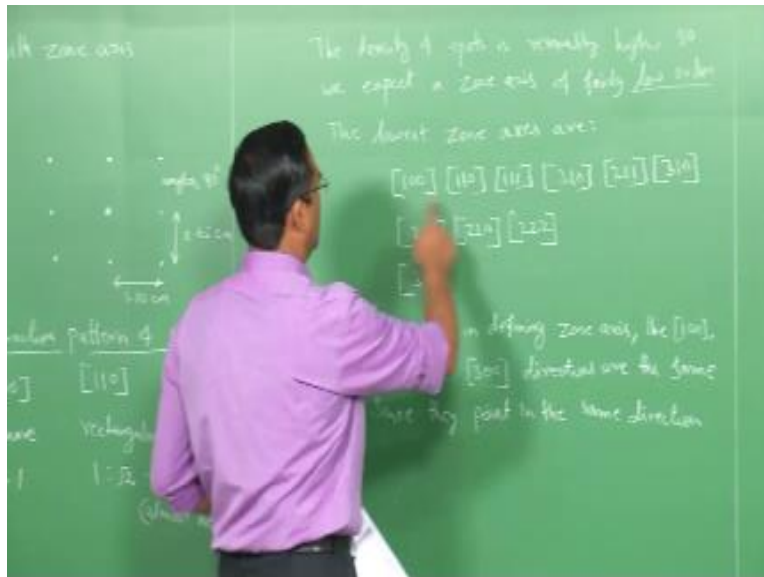
So what I have done here is I have just put some of the symmetrical aspects of by fraction pattern of a cubic crystals so before you look into a solving the single crystal electron diffraction pattern you can have a reference something like this where you have the zone axis for example it is 100 110, 111 in a cubic system then you can look at this their symmetry whether it is square rectangle or external.

Then you can immediately recognize some of the indexing aspects of them so it is quite useful to have a table like this to compare the single crystal electron diffraction before you indexing so and also you can look at the aspect ratio if it is the square pattern then it is 1:1 and if it is rectangular 1:BCC and simple cubic and it is almost acceptable for an FCC and if it is for 111 it will be a equator .

So some of these symmetrical aspects will readily give you a clue about this kind of the origin of this electron diffraction so you can readily go to the reference book or any handbook where you can compare the single crystal electron diffraction pattern which is given in D for ready reference so that way it is easy to solve them.

But in this case let us see whether it follows any of this symmetry this particular single crystal electron diffraction pattern since it do not follow a noon of this then we will take the other method of solving it.

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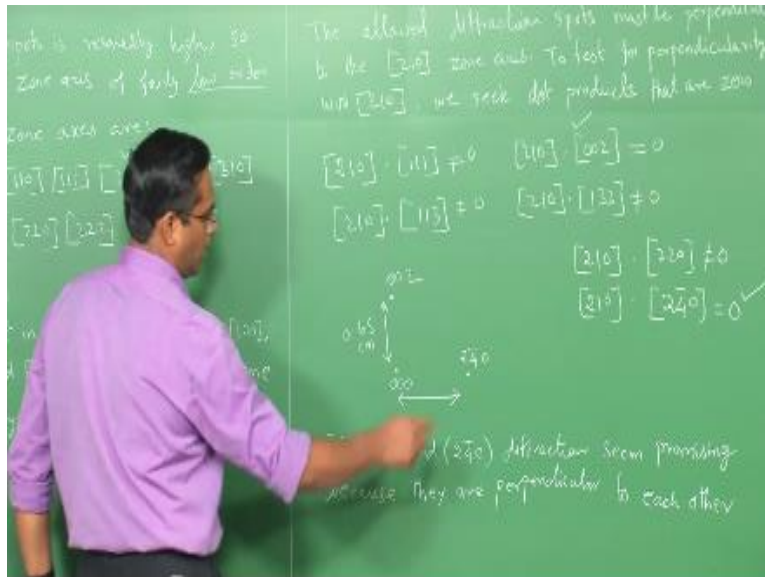


So what now we have to do is since the method itself says that will start with zone axis we have to select some zone axis by guessing first of all we have to look at whether this pattern is in falling with any of this symmetry and you also have to look at the kind of patterns so that means the density of this partition reasonably high so we expect a zone axis of fairly low order finally low order.

So we have to now look at the lowest zone axis what are possible so we will start with the 100, 110, 111, 210, 211, 310 etc and you can also look at this 200, 2, 20, 2 22 and 30 0 and so on and please note that in defining any of the zone axis the 100, 200 and 300 directions are the same since they point to the same direction so you have to look at some of the lowest possible zone axis.

Since this pattern does not fall into any of this we cannot take this 100 because it is not falling into this any of this symmetrical or aspect ratio in the table so what we will do is we will select be the next lost the first three we leave it we take the 210 as the zone axis as a guess work.

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So what I have written here is we have now taken the next lowest order zone axis 210 so the allowed diffraction spots must be perpendicular to this zone 2 and 0 axis to test that for the perpendicularity with the 210 we seek the dot products that are 0 that is what the condition the plane which is lying in any zone axis according to the V zone law all the plane should be perpendicular to this direction.

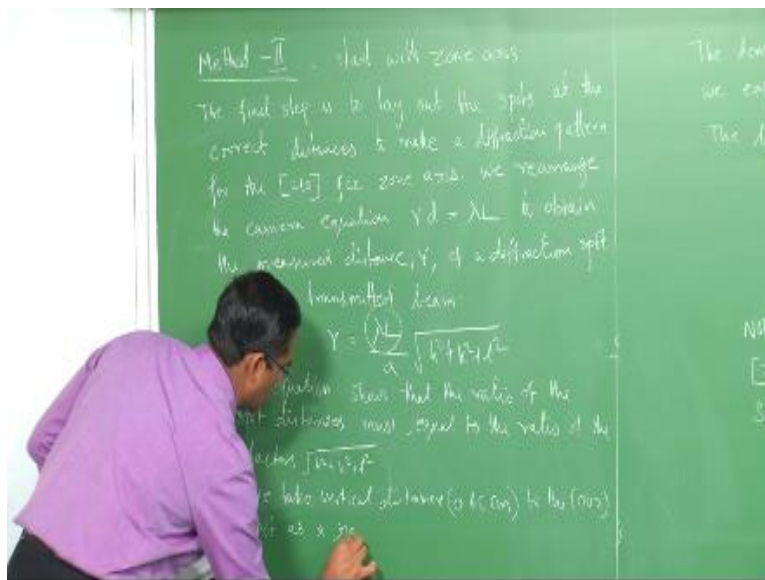
Then we can say that they all the planes are lying in this zone axis so we will simply say that so now if you look at this the lowest index planes which are lying in the zone axis we check this whether these two are in perpendicular so they are not zero the dot product is non zero that means they are not lying in the zone axis or the plane is not perpendicular to this direction.

So similarly for this on the other hand 002 this is zero that means this direction or a plane must be lying in this direction so these two are not perpendicular and similarly these two directions are not perpendicular but 24^{-0} is perpendicular to 2 and 0 so from this we have some idea these two cases you have for example this there is a possibility that this plane and this plane can be lying in the zone.

So let us see how to proceed further so we can take this so what is the next step so now we have taken one zone axis and then we looked at all the possible direct directions or plane normal is which is dot product is equal to 0 then we found that this plane normal are perpendicular to 210 then we can assume that these two planes diffraction planes appears promising because of this perpendicularity.

The next job is we have to just put this two planes in a respective distance to make sure that they are exactly lying in 210 so for that we have to check with the other parameter to keep these two planes in a correct directions how to check that so that is our next step so what i will do is.

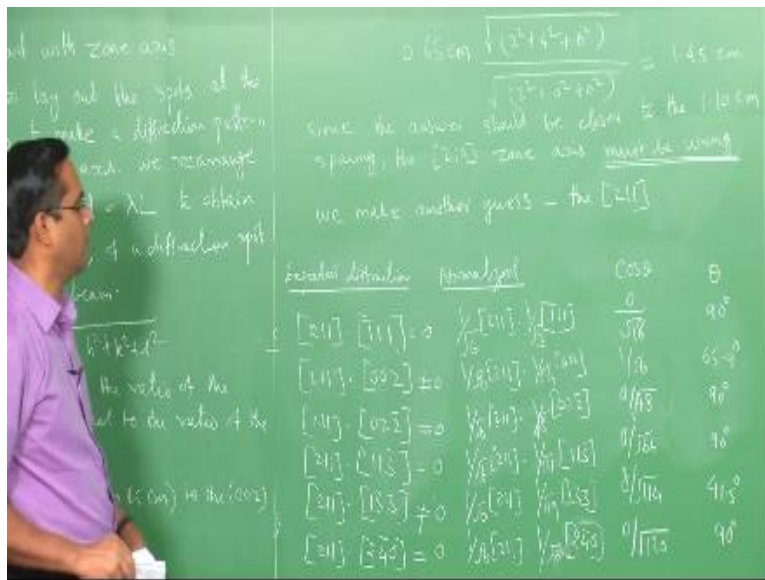
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So what we have done is the final step is to lay out the spots are a correct distances to make the diffraction pattern for this 210 FCC is own axis so we rearrange the camera equation our d is equal to L to obtain the measured distance R of a diffraction spatter pattern spot from the transmitted beam that is this distance whatever we have measured this one and one point 10 in this the other direction.

That we will put so if you in this equation suppose if you know the camera constant already then we are directly we can put it but if you do not know the camera constraints since it is a constant number you can take the ratio of these two planes and then you can try to find this distance so let us what we are going to do so the above equation shows ratio of the spot distances must equal to the ratio.

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So what we have done here is the equation of the camera constant shows that the ratio of the spot distance must equal to the ratio of the factors square root of H square plus K square plus a square so that means since we do not know the camera constant we can adopt this method. So in this particular case we take the vertical distance which is 0.65 centimeter to the 002 plane from the transmitted spot that is 000.

As a reference distance then we can put that as the you know ratio of these two planes then what we get is 1 point 4.5centimeter that means this is vertical distance what you will get us in a horizontal distance that is 1.4 5centimeter but what you have to now look at it is whether this is matching with what we have originally had in the problem.

It is slightly higher than the initial distance which is 1 point 10 so that means this zone axis selection 203 020 is not correct so far it was good but once you want to place the two spots 002 and 2/0 in a correct distance and we are not getting the close distance what we have actually measured so that means this particular zone axis is not the correct zone axis for this part.

So this is one clear-cut example of a wrong indexing okay that is how it should be measured so what we should now do we can write so that means the 210 zone axis must be 0 so we have to tie again with something else so how to move further we make another guess so we will make another guess that is the next in the line in the original possible zone axis after to10 it is to 11 so we take this and then try the same procedures like what we have done.

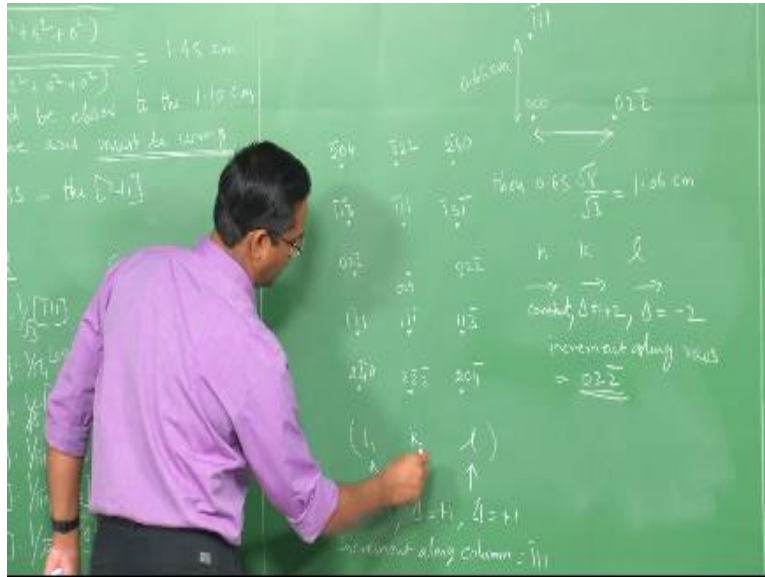
So what we have done here is since we are making an another zone axis to 11 as the new zone axis then we need to explore all the low index planes or their plane normal perpendicular to this zone axis so for that we have created a table the expected diffraction and they are all normalized with their indices like this so that you find out the $\cos \theta$ value from there you actually find the theta that is the angle between the two planes.

So it is quite straightforward so you take this first 111 plane normal and which is the dot product is 0 and then you see that $\cos \theta = 0^{18}$ which is nothing but the θ is 90^0 these two are perpendicular to each other so similarly you try 002 which is not equal to 0 and then you get you see that the planes are also not 90^0 apart that is purple not perpendicular to each other but you have 022 / . product is 0 similarly you can prove that angle is 90^0 .

So similarly 113/ these two are dot product is 0 and then you can see that angle is $\cos \theta$ is 90 degree I mean $\cos \theta$ is 0 and θ . So like that you have 133 which is not perpendicular to each other but you get a finite sum some angle which is other than 90^0 the fourth one is perpendicular to each other so you get about 90^0 between the two planes.

So like that it is a one standard way of putting in a table so that you readily work out the planes or the plane normal which are perpendicular to this zone axis so from now we can just want to assigning the indices to the.

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So now what you see from this relation similar thing suppose if you take from this table the first two sorry the first two perpendicular planes and if we can put them in their respective distance according to this distance is coming to one point zero six centimeter which is very close to what we had in the measured distance which is 1.10.

So that is that means this calculation is correct we can now rest assure that this type this zone accesses a correct zone axis so now we can just expand this so once you have this the basic two planes and then if you if you can expand the reciprocal lattice with the linear combination of these vectors suppose if you consider this as a vector and this vector you can add this.

To get this similarly you can expand this with this for example you can you can keep on adding this with this vector and then get this so something like this for example you add these two you get this $2/2 + 2$ for $22 + 2 = 0$ so similarly you can just add these two as a linear combination and then to get this and you can add these two to get this similarly.

So it is a combination linear you can just expand this whole net as much as you want then still you will see that all the planes will lie in 211 zone axis so that is the solution of our finding the

indices of all this single crystal electron diffraction pattern but what you can see here is there is you can see that your HKL HS constant in this direction and K is $\Delta+2LS$.

So you can also have some idea from this basic vectors the HKL values in this direction that is the increment as you go from this transmitted beam to right and side the increment towards this direction that is H is constant and K the difference is $+LS-2$. So which is nothing but $022'$. Similarly the increment along this column you have $\Delta=-1$, $\Delta=+1$ in the case of $k\Delta$ is $=+1$ in the case of L.

So the total you will have $111'$ as an increment when you move in this direction so that is another way of cross check this and though it looks little bit longer procedurally as compared to the ratio method and both the methods are useful in indexing as in the crystal pattern like this so finally what we can do is we can also now show that probably you can do it as an exercise because you can take any one of this plane and then make sure that they are all lying in this 211 that means their cross product.

I mean dot product should be 0 you can check that and what I will like to do in the next tutorial we will look at the polycrystalline electron diffraction pattern you will get a ring pattern so I will work out the procedures how to index those polycrystalline pattern using some simple procedures and then you can also practice for the sum of the known crystal system and then we will also compare that procedure how to apply it for the unknown crystal systems. Thank you.

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