Indian Institute of technology Madras
Presents

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NPTEL ONLINE COURSE

Tutorial-6

Hello everyone welcome to this material characters course organized by NPTEL and in the last two tutorials we have been looking at the solving the electron diffraction pattern and we have just looked at solving single crystal electron diffraction pattern by choosing two methods one by selecting the diffraction pattern directly and then find out the zone axis are you canal ways start with the zone axis by guessing and then arrive at the particular zone axis with specific indices.

And then, the last one which is slipped over in this tutorial problem is looking at the ring pattern or polycrystalline electron diffraction pattern we will just look at the general procedures how to deal with indexing this poly crystalline electron diffraction pattern so I will just layout some of the general procedures what is being followed and then I will also take up some specific cases a simple system like cubic system and then we can adopt this general procedure to any crystallographic system so let us just start with the, the procedures for solving the electron diffraction pattern in the case of poly crystalline material.

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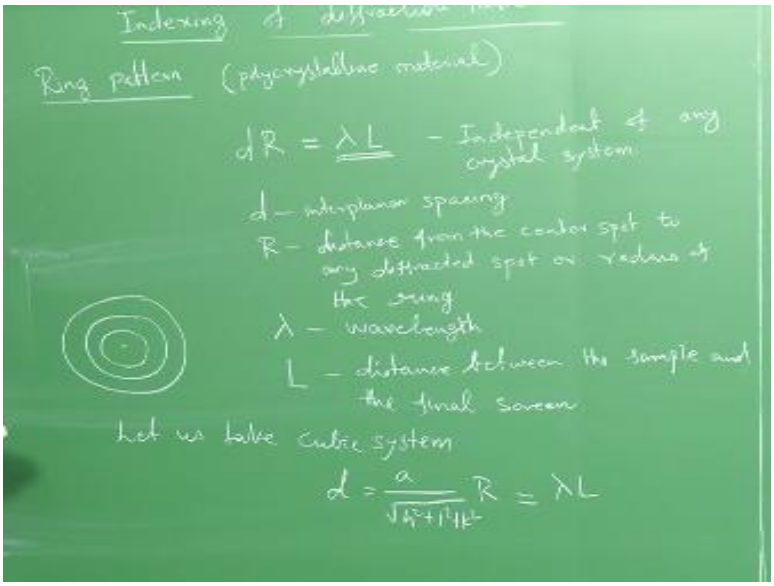

You so we know that this is the relation which we have in the from the geometry of the transmitted and the diffraction system in the electron microscope our dis equal to λ L and D is the inter planar spacing r is the distance from the center spot or a transmitted spot to any of the defected part in the case of single crystal pattern or it is just the dist amine the measure of radius of the ring in a poly crystal linear diffraction pattern lam lambda is the wavelength and l is the, the distance between the sample and the final screen where it appears.

So the, the product of the λ and l is considered as a camera constant which we have already seen and we can calibrate this that I will talk about little later so this is the basic idea with which we are going to work out so in the case let us take the ring pattern so what we do suppose if you have the ring pattern of this kind let us talk a port a general procedure suppose if you have the ring pattern like this then we can now consider a simple system probably a cubicle system so in case you can write let us take one cubic system for example because it just worries a well-known system so let us take that so we can write for that you know that in a cubic system d is equal to λ l.

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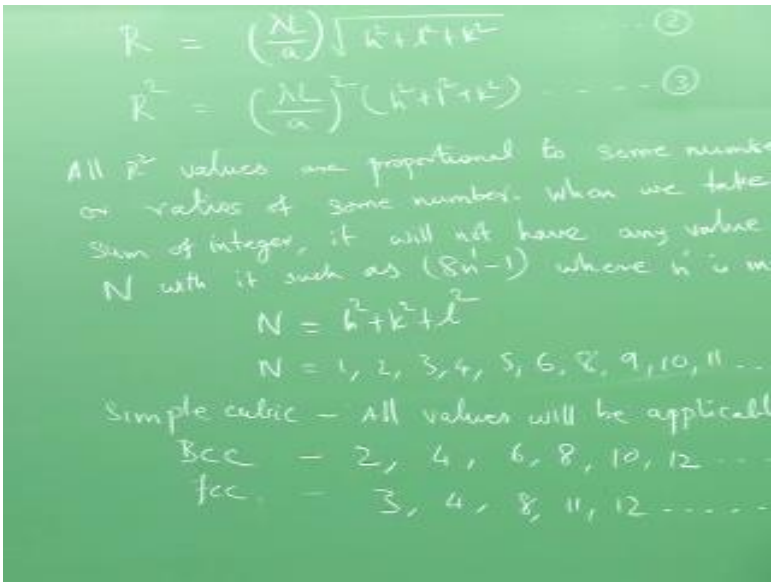

And this you can write as equation one and we can rewrite this equation R is equal to you so what we have done here is for the cubic system you know that d is related to this the e the lattice constant and then from that we can rearrange this into R^2 value so what we have written here is all r square values are proportional to some number or ratios of some number so when we take this H^2 L^2 K^2 that is as the sum of all the integer it will not have any value of n with it such as for example it will not have 8 n prime minus1 values where n is an integer.

So for example if you substitute this n can have only values like you can you can just put it like this 1, 2, 3, 4, 5, 6 and then 7, 7 will be missing because if you put n is equal to n prime is equal to1 so 8-1=7 will be missing so then it is 8 8,10,11 then the next number will be missing something like that suppose if you put two then it is 16-1= 15 will, will not be there here in this series so this is one way of putting this and, and then we can create a table for all this different n values and what we are now trying to do is we are trying to take a general procedure then we will compare this.

This capital n according to the specific crystal system because we will be able to look at the allowed reflections and then try to match with any one of this the, the sum of the integer values

and then try to arrive a possible solution so we will take up some general area for example in the case of simple cubic all these values all values will be applicable all the values will be applicable but if you go to bcc that is body centered cubic lattice you will have 2,4,6,8,10,12,. so like that the allowed reflection similarly we can write for CC you have 3,4,8,11,12 etc.

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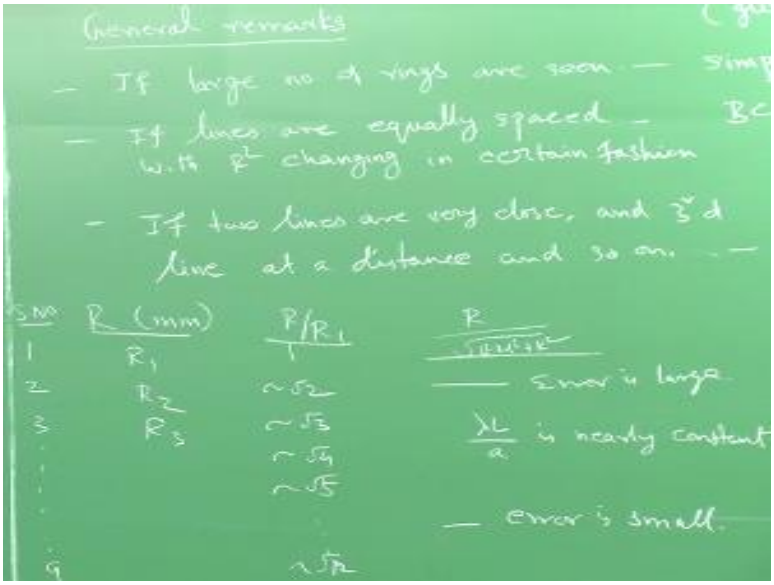

So it will go like this and we can write for looking at the ring pattern some general remarks if large number of rings so seem then this is probably a simple cubic it is only a guess this is BC with R^2 changing certain fashion if two lines voting closed and third-line Dennis case and so on so when you look at the poly crystalline pattern these are some of the general remarks you can look for if the large number of rings are seen in your head diffraction plane and it could be a simple cubic just give you a first idea first impression.

And if the lines are equally spaced with the R^2 changing in certain fashion we will see I will make a table and then tell you what does this meaning when r squared values what you have shown here when it changes in a particular fashion then we can guess this could be a body centered cubic crystal system and if the two lines are very close in a because all the poly crystalline ring pattern will appear like a concentric rings in those pattern if two lines are very

close and the third line is at a distance and this pattern repeats then it could be face, face centered cubic lattice so that is the first impression you can get these are all some of the observations you can keep in mind so that you can quickly come to a conclusion and then later you can confirm that with a proper analysis so that is what we are going to do now for example you can do you measure start measuring.

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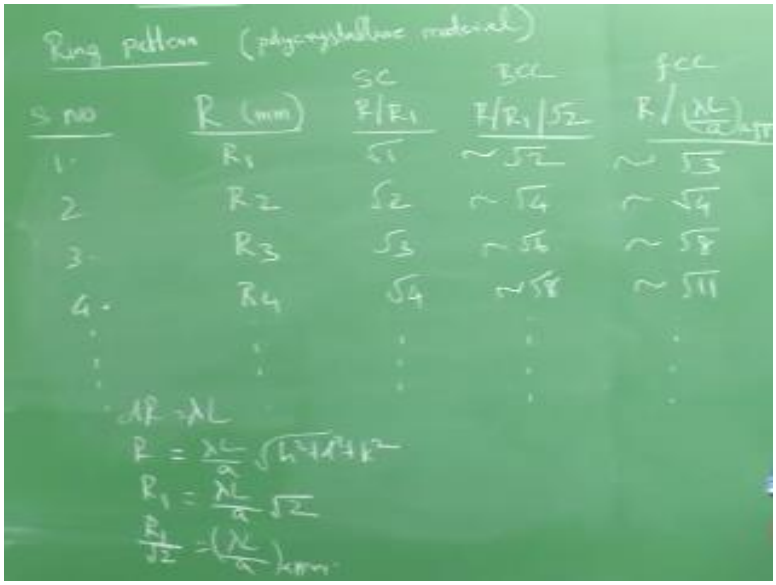

So the another general in observation is suppose if you measure the Rings different rings in a poly crystalline pattern like this I'm going to take the ratios with the smallest radius and then tablet it like this so the measurement is to be very, very careful whether it is a single crystal pattern or a polycrystalline pattern because each person will have its own you know human error in measuring that so when you in a general case like this so the initial values the error will be large at the beginning then as you go down the error is small.

so it is always to try to take different links and then take an average for a to minimize this error so that is one general remarks one should have for example we can now try to tabulate this in a general for you see what we have now done is just whatever I have mesh we have written in there in the previous section we have just start comparing with the a simple cubic system one is a

simple cubic this is a body centered cubic this is a face centered cubic you can see that your simple cubic varies with this r by r ratio and then what I mentioned is your the other ratio is goes in this fashion suppose if you do this kind of at a blazing and then for an FCC it goes like this so you know that again you can do this lambda L you know that r =λ/a and R1=λ L/ a and then $r1^2$ /λ L/ a approximate.

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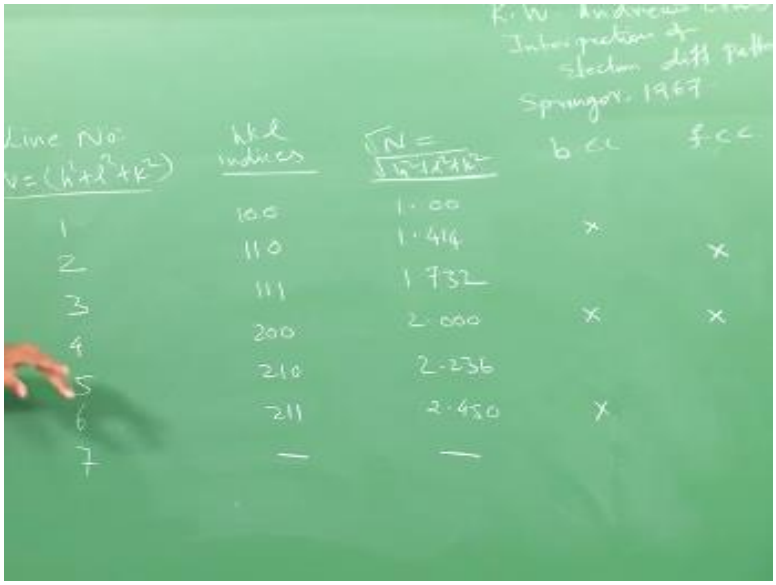

So that is how you calculate for this particular system so this will be a an approximate number but then it will follow this trend for the key in the case of FC now based on this I will just follow we can also make table with simple you can also make table like this, this is from the book AW and Bruce I tell some book interpretation no electron diffraction so this example which I am going to give table going to show KW and rules at all interpretation of electron diffraction pattern published by Springer science in 1967 and very useful table which i am going to show this.

This is line number you can put n is equal to H^2 a square and hklin Jesus root n is equal to 0 da Vinci FC so take this one so you have 1 0 0this is 2 1 1 is little14 this is 1111.7 point to me tell me so for 20xx fine so 2.36 so what I have drawn a table is general indices and this is your n

value and this is square root of N and which is corresponding to you are allowed reflections in the both systems so you can see that as I mentioned in the beginning you see that 8-1=7 is absent here and you can see that in BCC reflections are tabulated here.

And a similarly FCC reflections are tabulated here and that so you can compare the, the result standard tables which will be available so whatever the table you generate from this our values or r squared values you can relate that with the again by putting these tables of course you have to remember that this is a simple cubic system for other crystal systems you will have a particular d spacing relations so you have to accordingly make a table and then compare this ring pattern so that is the one important aspect of analyzing this we can now write general rules this is again from be you.

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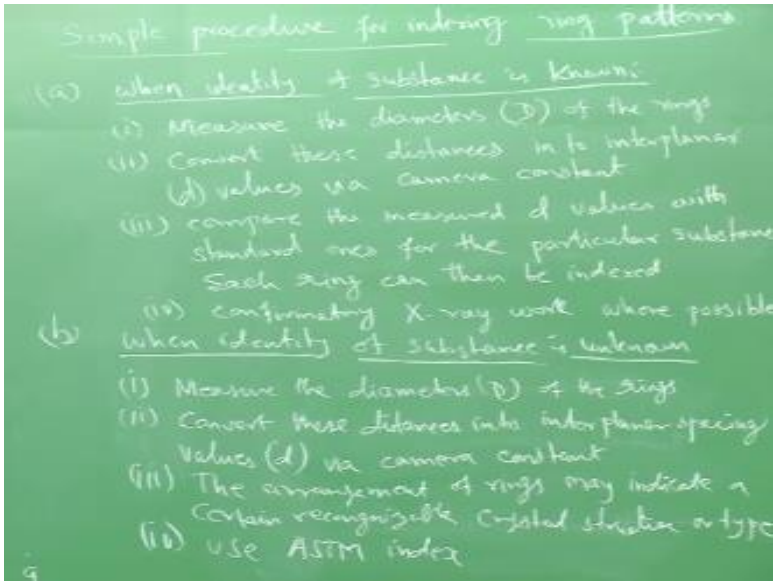

So what I have returned here as a simple procedure for indexing ring pattern there are two cases you have to remember one is when the identity of the substance is known then you just follow this procedure first you measure the diameters of the ring D and convert these distances into interpreter spacing values that is d very small be yr Tamara constants that is what we have done

here in this table that is what we have done and compare the measured D values with the standard ones for the particular substance and then each thing can be indexed like this.

You have to just measure the distance and compare the D values and, and Confirmation can be done even with an x-ray work if possible or if it is necessary and if it is a unknown substance then follow these procedures so similarly measure the diameters first then convert them into diameters through camera constant and then look at this some of the general remarks what we have stated already the arrangement of rings may give some clue that means whether they are a large number of rings are do they have some they do they fall some particular symmetric fashion as something like your FCC.

You have a two closely spaced lines will be there and then third line will be with some distance and so on so you look for some clue from the arrangements of the ring and then use the index where you have the camera constraint d value with the standard crystallographic data are the D spacings index you can look at them and then also finally you can do x-ray diffraction to confirm that whatever we are analyzing from this so.

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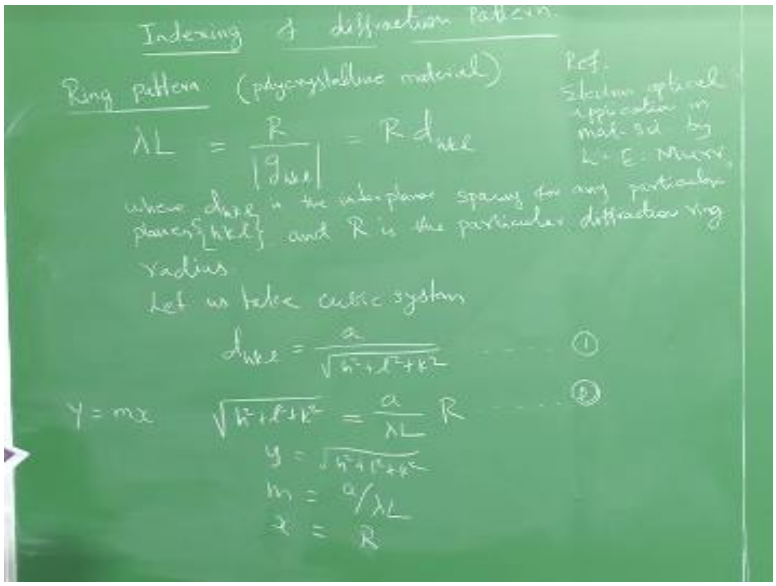

So these are some of the standard procedures for analyzing the ring pattern now we are talking about camera constant so I will now just briefly tell how to do the calibration for the camera constant in the case of analyzing this polycrystalline material so same procedure Canberra constant is λ L can be written as our G H K L=R up this is this particular method is from reference electron of a compass application vacation in material things why so here again what we have trying to do is this is from the reference electro optical application in material science by L le Mor and what we are going to do is from this relation how to do the calibration of the camera constant.

So our this is ji ji hkl is a diffraction vector which is related to our D H K L and D hkl is the enterprise missing for a particular system for a particular flame hkl and r is the particular diffraction during radius and if you take cubic system then we write hkl then we can rewrite this into so this form of the equation appears like a straight line equation so we can say that suppose if you put this into y =MX type then we can write $y=x^2=1^2=a^2m=\lambda 1r=x$ are I would say X = R let us be uniform here.

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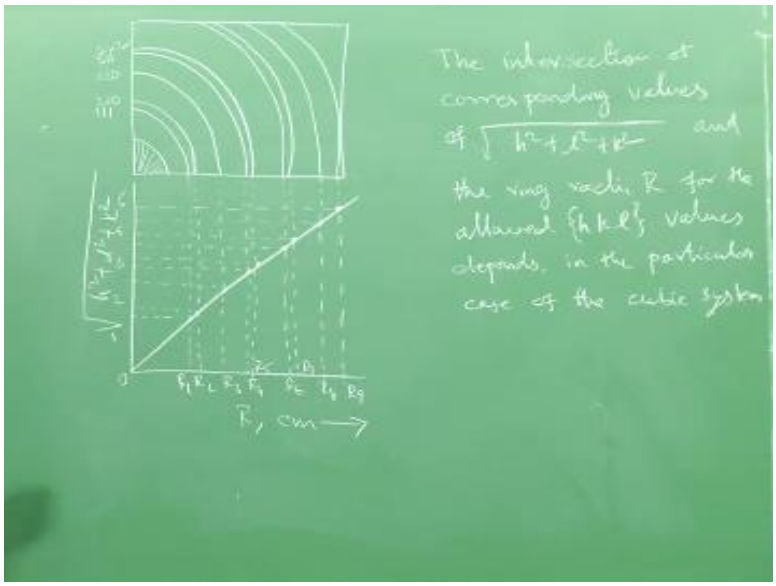

So we can put it like this and we can now draw one schematic to show this suppose if I have the diffraction pattern like this and then you so what we have done here is so we have taken some a cubic system equation and then we see that if you write this equation in this form it is straight line equation so we have taken one polycrystalline pattern which actually appears like this in the case this is the particular case of nickel.

But then the message what you are trying to get from this is if you plot this $H^2+ L^2+ K^2$ versus are the intersection so the intersection of the corresponding values of this square root and the ring radius for the allowed values depends upon the particular cubic system but in this system it is nickel so like that if you plot then you will the slope is nothing but your camera constant if it is done for a loan system then you have the value of camera constraint.

And if you can perform use this camera constant for a given acceleration voltage and you can use that camera constant for analyzing the unknown pattern if you operate the microscope in the same condition then that is valid and this is another way of calibrating the camera constant so that you will have that value you can just simply plug in and then find out the D spacing for the unknown material so this is a another way of looking at the same procedure but then it gives you a kind of a simple procedure to plug in this D values and find out the camera constants. Thank you.

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