X-Ray Crystallography

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Lecture - 28
Determination of Single Crystal Orientation by X-Rays

We all know that any property of a polycrystalline material is very much dependent on the property of the constituent crystals. Now each crystal is an isotropic in the sense that any Property in a particular direction of the crystal may not be the same as the value of the same property in any other direction. It is therefore, very much essential to know the orientation of a crystal when we are going to measure a particular property of that crystal.

Now, there are 3 different methods of finding out the orientation of a single crystal by X-rays, namely the back reflection Laue method, the transmission Laue method and the diffractometer method. In my lecture I will deal with the principles of the back reflection Laue method for determining a single crystal orientation. The method will be very much similar in case of the Laue transmission method, and the diffractometer method. I am not going to discuss those 2 methods here.

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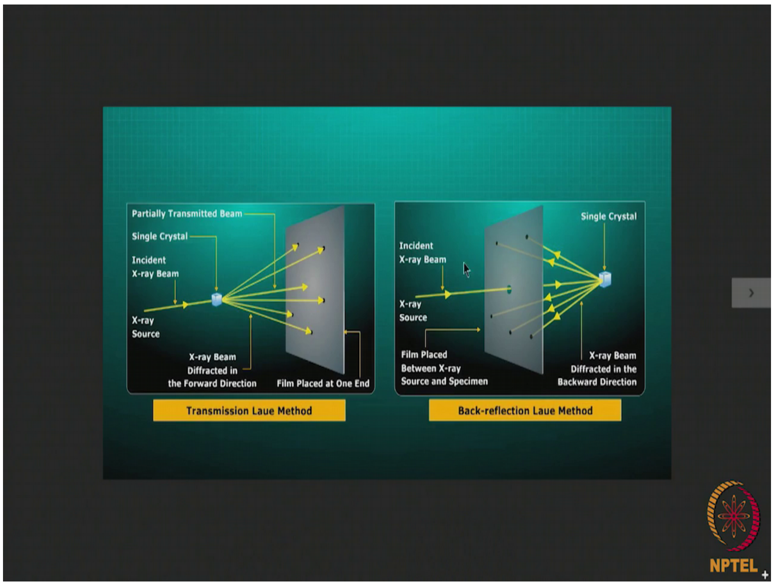

Now when we look at the transmission Laue method here the single crystal is placed in between the X-ray source and the film, and the diffracted radiation from the different crystal planes will intersect the film at definite points or spots.

In the back reflection method, it is the film which is kept in between the X-ray source and the single crystal, and the diffraction refracted radiation from the different planes in the crystal they are allowed to fall in the backward direction on the film. As a result when we read the film in the transmission Laue method we read it from this side and in the back deflection method we have to read it from this side because the backward reflected defracted radiations will cut the film in this direction.

Now, these are the type of diffraction spots that are obtained in case of the transmission Laue method and the back reflection Laue method.

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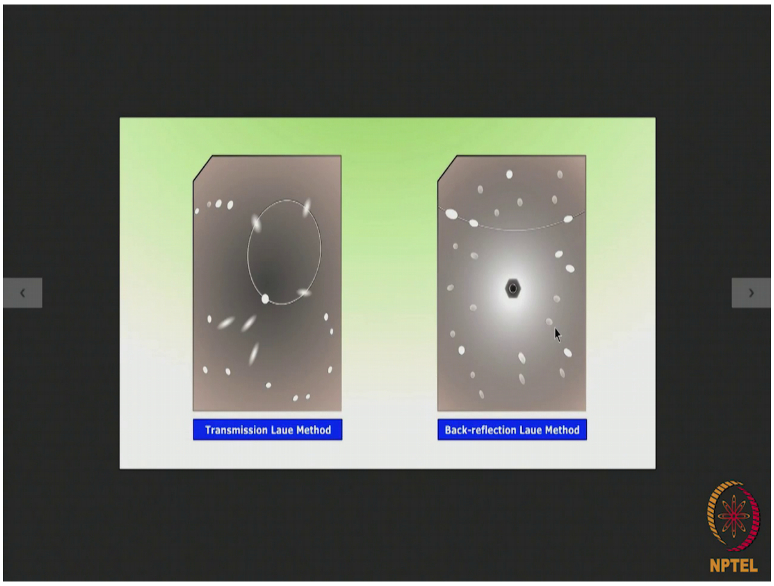

It is quite clear that in the transmission Laue method, this spots that are produced on the film they can be joined together in the form of ellipsis. On the other hand when we look at the back reflection Laue method, this spots or the diffraction spots formed on this film they can be joined together by hyperbolas.

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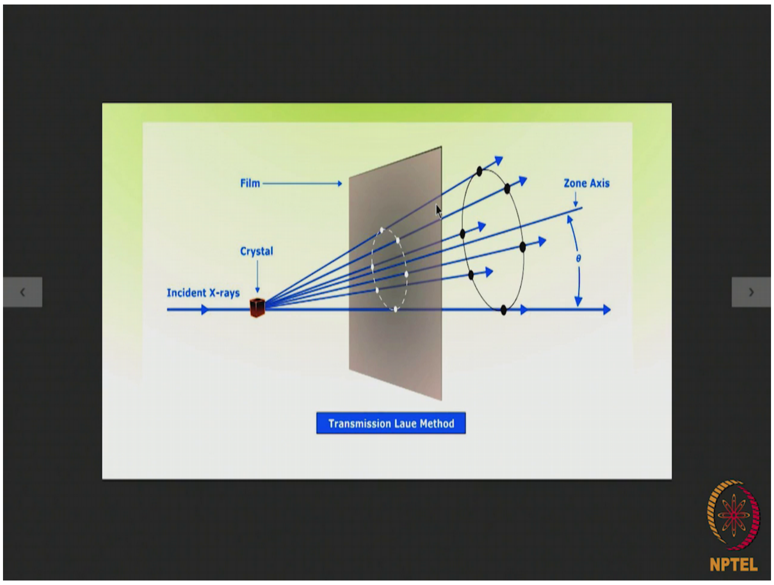

Now the reason for this will be quite clear from these diagrams. This shows that when we have the transmission method then a number of planes which form a zone in the crystal, the diffracted radiations from all those planes in the zone will come out in the form of a cone of radiation. And that cone will intercept the film in the form of an ellipse.

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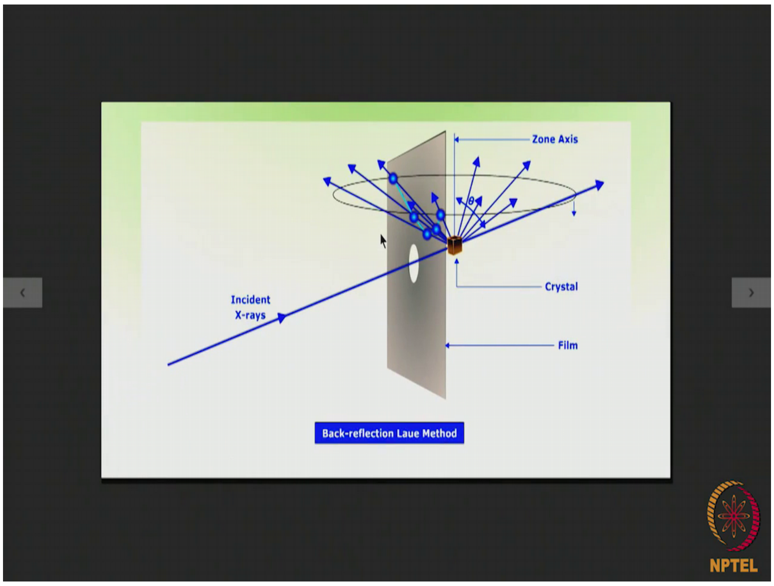

On the other hand when we have the back reflection Laue method, the incident radiation when it falls on the crystal then the planes of the of a particular zone they will be reflected backwards, and as a result they will form a hyperbola on the recording film. So, this is the reason why we get a ellipses in case of the transmission Laue method and hyperbolas in case of the back reflection Laue method.

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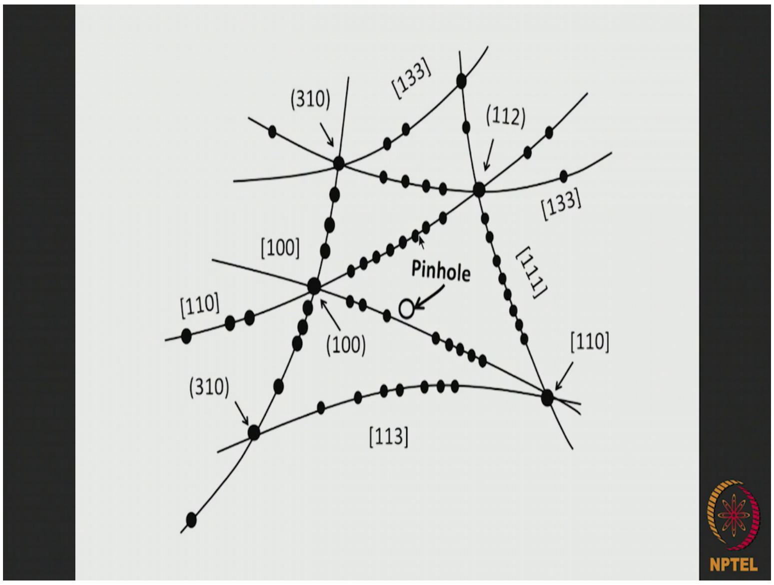

Let us suppose that we have a are an iron sample a BCC iron sample, single crystal sample which is being examined in the back reflection Laue method. So, all the spots appearing on the film can be joined together in this manner in the form of a large number of hyperbolas. For example, this part here on this line they represent the zone 1 1 0; that means, all those planes which belong to the zone for which 1 1 0 is the zone axis their diffraction spots will lie on this hyperbola.

Similarly, all those planes which form a zone for which 1 1 1 is the zone axis their diffraction spots will lie on this hyperbola. In a similar manner the planes forming the 1 0 0 zone will be lying in this manner on this hyperbola. The planes forming the 1 1 3 zone their diffraction spots lie on this hyperbola etcetera, etcetera. Now when it put the specimen in the back deflection Laue method, the x radiation is allowed to fall on the crystal surface in a perpendicular manner, and in that respect the perpendicular to the surface of the crystal will be lying right at the center of this region whenever we get a spot on the X-ray film in the back reflection Laue method.

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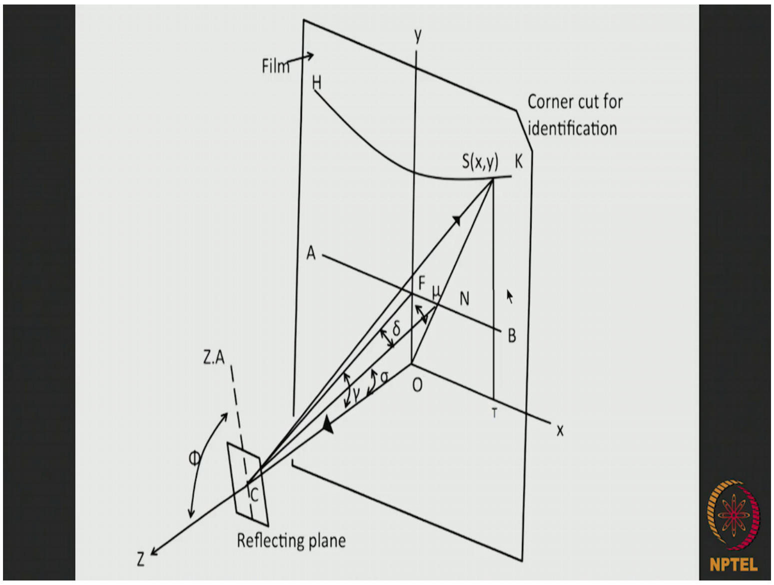

It is possible to find out the value of theta or the Bragg angle for that particular spot.

Say for example, here we show schematically. So, this is the direction of incident x radiation along o z and here, we have got our sample and say this is a particular plane within the single crystal sample which belongs to a zone for which this is the zone axis. Now the diffracted spot by this plane falls at the point S say, now what will happen to the plane normal the normal to the plane which is CN is incident over here on the film.

Now this particular spot which has got the coordinates x and y and which can be easily measured on the film this is the x coordinate this is the y coordinate it can be measured very easily and knowing the value of the specimen to film distance and the coordinates it is possible to figure out how much will be the value of theta the Bragg angle for the plane which is deflecting a spot at the point s.

But the problem is, if we remember Bragg’s law. Bragg’s law says, lambda is equal to 2D sin theta. So, lambda in case of the Laue method is not a monochromatic radiation. It is a poly chromatic or white radiation a mixture or various radiations. Therefore, from the Bragg equation even though we know the angle theta for this spot it is impossible to figure out the D value, because lambdas are can kind of. So, many different values; that means, we cannot identify the plane from which this diffraction spot has horizon.

Now, if we consider this plane to be rotated around this axis, then by that process all the different orientations of this particular plane belonging to the same zone will be produced. And accordingly the diffraction spots will all appear on this hyperbola. So, all the diffraction spots from a number of planes forming a zone will appear on this hyperbola. And what happens to the plane normals for all the planes belonging to that particular zone? Their locus will be on this straight line which is perpendicular to the y axis.

Now, let us draw a perpendicular for S on y x it is t and what we find will find now is knowing the x and y coordinates of the spot S and the value D from the specimen to film distance, we can find out the angular parameters for our plane normal here. Say this is the plane normal here CN. So, if we look at it this plane normal is tilted from this axis by an angle lambda. And it is away from this central point by an angular measure of delta.

So, we have a situation where if we draw the perpendicular or the normal to this plane CN, then CN will be tilted you know about this axis by an angle gamma, and it will also be shifted by an angle delta from this central location. So, these angular parameters which define CN or the plane normal of the given plane here, this values can be easily found out from the measured values of x and y on the film and by knowing the specimen to film distance which is capital D.

Now, from this diagram we find that x is equal to o t which is nothing but OS sin mu.

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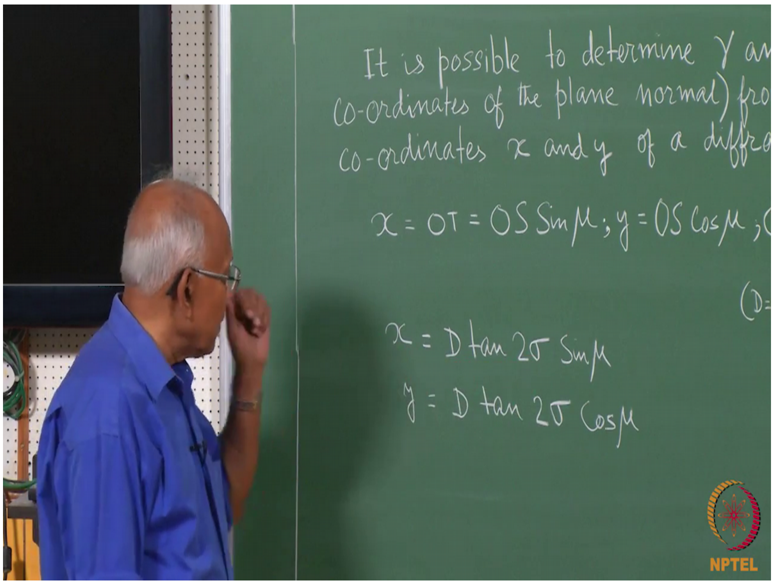

You see this angle is mu here and automatically this angle will also be mu. So, we can write x is equal to o t is equal to OS sin mu similarly y will be OS cosine mu and what is OS by the way OS is OC tan to sigma and OC is nothing but capital D which is a specimen to film distance. So, OS is equal to D tan 2 sigma.

Now, this is the value of sigma over here and this is the value of mu. Now then x can be rewritten as x is equal to D tan 2 sigma sin mu and y is equal to D tan 2 sigma cosine mu. Now again in the triangle FNO, if we look at the triangle FNO here we can write down tan mu is equal to FN by FO which is equal to CF tan delta by CF sin gamma equal to tan delta by sin gamma.

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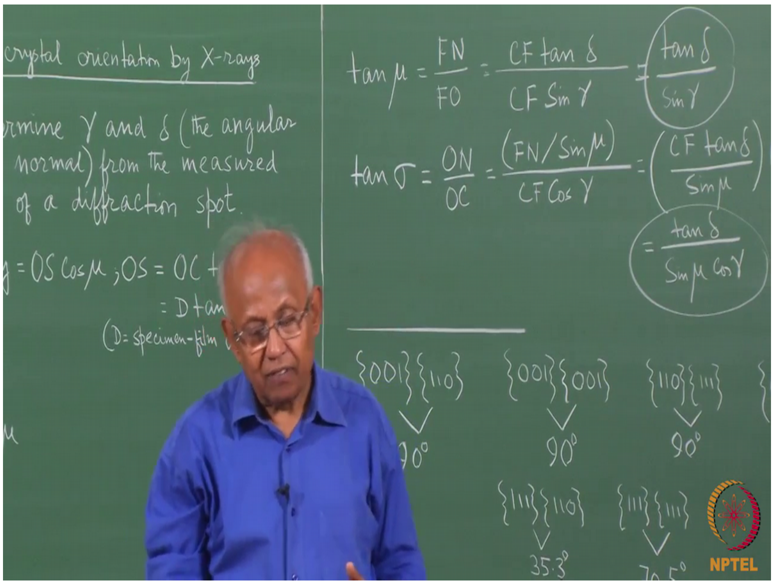

Similarly we can write tan sigma is equal to o n by OC equal to FN by sin mu divided by CF cosine gamma that equals CF tan delta by sin mu into one upon CF cosine gamma.

So, that leads us to tan delta by sin mu cosine gamma. So, we see that if we can measure the value of x and y and the specimen to film distance, then it is quite possible to figure out the values of gamma and delta from these relationships. So, knowing the values of x and y it is quite possible to figure out the values of gamma and delta. So, what we are doing here, we have got say for example, a plane in which belongs to a particular zone with this as the zone axis. So, this is the plane belonging to a particular zone in the single crystal having a zone axis given by this. So, from all those planes in the zone the diffracted spots will fall on this particular hyperbola and the plane normals. The normals to all those planes will fall on this particular line A B.

So, what we are trying to do is you see we have already seen that knowing the value of theta we just cannot identify this particular spot, because we are using not a monochromatic radiation, but if polychromatic radiation the Laue method. So, we cannot identify the planes which give rise to this part and all the other spots from the planes of that zone. But we know that any plane normal bisects the angle between the incident radiation and the diffracted radiation. So, this is the incident radiation direction and this is the diffracted radiation direction. So, the plane normal CN bisects this angle.

And if even if we cannot find out the value of find out the identity of the planes which give rise to the diffraction spots, it is possible to find out the locations of all the plane normals on this line. And these can be converted because plane normals are nothing but poles this can be converted into a series of poles on a stereographic projection. And once we can project we can have the plane normals of all the planes belonging to a particular zone. And if we can plot them in the form of in the form of stereographic projection then by measuring the angle between those pores and by having a table which shows the interpreter angles between the different planes it is possible to identify the planes which have given rise to the diffraction spots. So, this is what we are going to do now.

You see if we look at this particular diagram for this particular zone of planes the diffraction spots will appear over here and the plane normals will appear on this line. So, we find for these particular zones the plane normals will be tilted away from this line by an angle gamma, and for this particular spot it will be shifted from the center by an angle delta. So, this gamma will be the same for all the planes which have given rise to diffraction spots on this line. So, all the planes of the zone which give rise to the diffraction spots on this particular hyperbola, their plane normals will be tilted by an angle gamma from this line, and you know for this particular spot the value of delta is here for another spot over here the value of delta will be on this side etcetera etcetera.

So, we see that, you know just as at different values of gamma we can have the you know the you know for a particular you know series of planes belonging to a zone we will have a particular hyperbola as the locus of the diffraction spots and a particular line is the locus of the plane normals for another zone we will have another particular hyperbola for the diffraction spots and another particular line for the plane normals.

So, you see that what we can do from this diagram, we see that you know the gamma and delta values for a particular hyperbola can be it will be single gamma, but deltas will be different. So, you see that for different values of gamma we will have different hyperbolas here from left to right now, and for if we consider the same value of delta in the plus side here on the minus side over here, and if we can you know draw lines through equal values of delta then they will form another series of hyperbolas from top to bottom. Just as you can have the hyperbolas of the diffraction spots you know from left to right.

Similarly, equal values of delta will give rise to hyperbolas running from top to bottom. And if we remember this it is possible to construct what is known as a Greninger chart.

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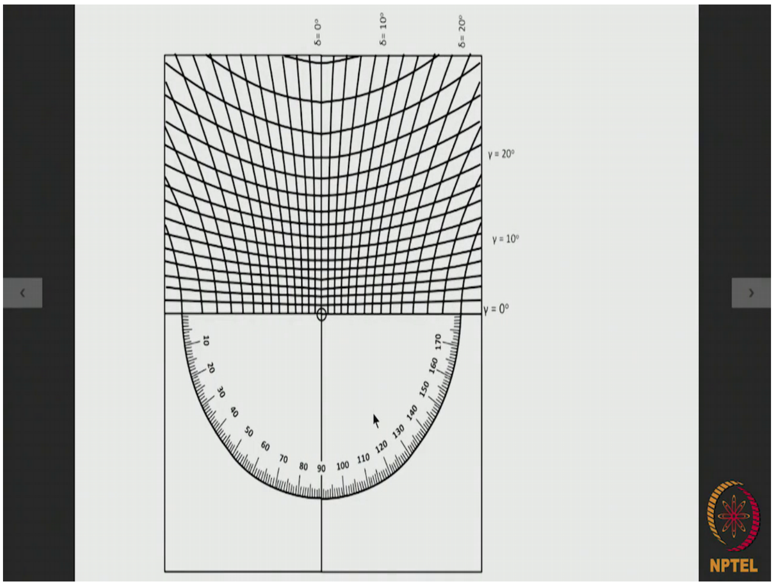

So, you say that in the Greninger chart we have got these are all the different gamma values you see. So, this is gamma is equal to 0 gamma is equal to 10 degrees, gamma is equal to 20 degrees gamma is equal to you know, this that then comes delta is equal to 0 here then delta 10 degrees delta x with 20 degrees etcetera etcetera.

Now, this Greninger chart can be drawn keeping in mind the particular distance between the specimen and the film. And for a particular distance D between the specimen the film you will have a Greninger chart of a particular dimension. Now the advantage of the Greninger chart is, if we put the Greninger chart on top of the diffraction spot pattern then for all the spots which appear in the on the hyperbolas in all the diffraction spots from those diffraction spots we can measure out the corresponding gamma and delta values of the plane normals.

So, what we do, we prepare a chart called the Greninger chart, and when you put the Greninger chart you know on top of the film with the diffraction spots then we try to have the diffraction spots on a particular hyperbola to lie on a particular you know, hyperbola or the Greninger chart. And we can read out from these spots the values of corresponding gammas and deltas. So, as I wrote over here it is possible to determine gamma and delta directly. What are gamma and delta these are the angular coordinates of the plane normal from the measured coordinates x and y of a diffraction spot.

So, we have got diffraction spots in the form of hyperbolas and using a Greninger chart and putting it on top of the film we can locate all the spots on a particular hyperbola on the Greninger chart, and read out the corresponding gamma and delta values. So, this is how this is how what we obtain here.

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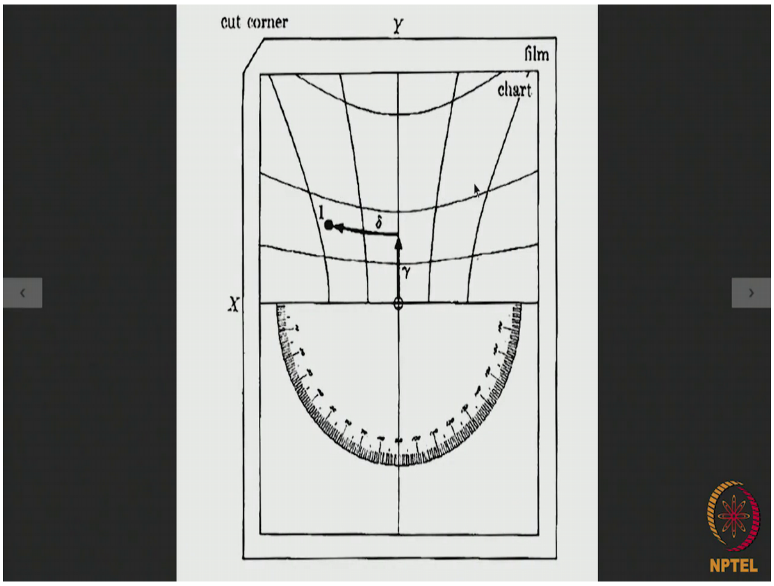

So, you see that we have got the film at the bottom and we have got the Greninger chart on top. So, for a particular series of planes belonging to a zone all these parts will appear on this particular hyperbola. And say this is one of the spots. So, for this spot this is the value of gamma this is the value of delta.

So, you see that the use of the Greninger chart using the Greninger charts straight away, we can measure the gamma and delta the angular parameters of the plane normal of the plane which gives rise to this particular diffraction spot. Now as I told you once we get the gamma and delta values of the plane normals then it is quite possible quite easy really to plot those plane normals on a stereographic projection.

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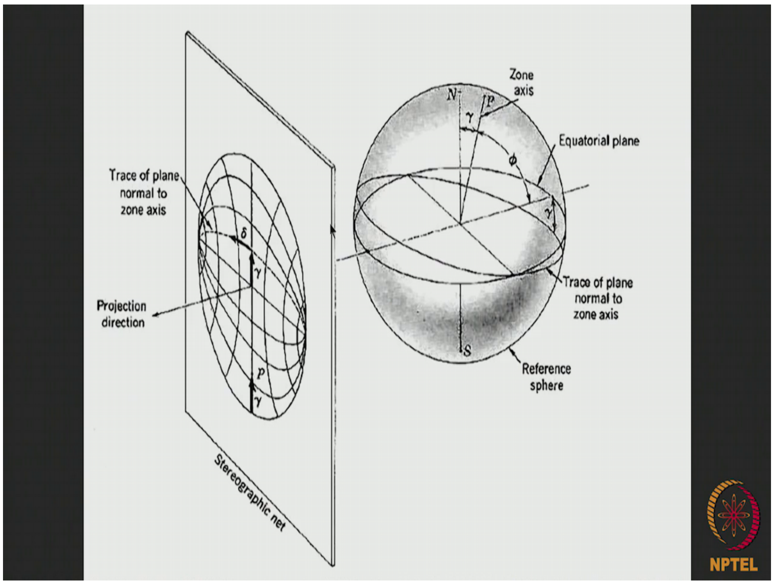

Now as you can see here say we consider our sample which is a very crystal single crystal which the very, very small a point crystal and consider a big reference sphere with the single crystal right at the centre.

Now, say this is the equatorial plane within the reference sphere, and say the particular trace of a plane which is normal to the zone axis you have been talking about is this one. So, this is the trace of a plane you know belonging to a particular zone for which the zone axis is known, and this is the equatorial plane of the sphere reference sphere. Say they are separated by an angular distance of gamma. So, if this is the projection plane if we consider the film as the projection plane.

Now, you know you will have the walnut put in this position now as you can see here, in the walnut the north and south are not are put in a horizontal fashion, why? Because you know the poles of a plane belonging to a zone you know the all the traces you lie on this longitude lines only. So, you know if we have the number of a planes belonging to a particular zone and in that case and if the corresponding value of you know of tilt is gamma, then this is the location of the trace of all the poles all the planes that will belong to that particular zone.

So, you see that we can plot all the poles of the plane normals in this manner on a particular the longitude line knowing the value of gamma and delta. Well for a particular hyperbola of diffraction spots gamma will be the same and the different spots will have different delta values. So, all those spots can be drawn on this line.

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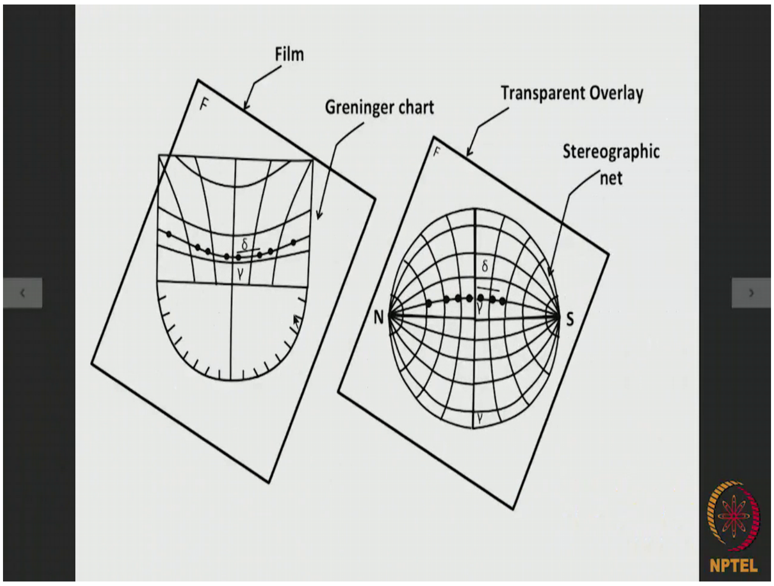

Now using the Greninger chart on top of the film as I have already said, this is the location of all the spots on a particular hyperbola on the film. Now this is the value of gamma for this particular hyperbola and the values of delta from the center will be different for the different spots.

Now, as I have already shown, these can be plotted you know on a transparent overlay over a stereographic net in this manner. So, we can have all these you know the plane normals you know all the plane normals for this particular you know planes which have given rise to diffraction spots those plane normals can be plotted in this manner. So, we have to remember one thing here we have got these are the positions of the actual diffraction spots on the film.

Now, when we put the Greninger chart on top of that and we measure out the value of gamma and delta for the different spots these gamma and delta refer to not the actual spots, but these are the angular parameters of the plane normals for those planes which give rise to those diffraction spots. And those plane normal positions can now be plotted in this manner in a stereographic projection.

So, we are we have got here. So, this part say is the plane if the location of the plane normal for the plane which give rise to this spot here. Say this dot here is the location of the plane normal for the plane which has given rise to this diffraction spot here etcetera etcetera. So, what we have got here by using Greninger chart from the film shows you all the diffraction spots from a large number of planes belonging to zone. But using the Greninger chart we find out straight away from those values the gamma and delta values of their plane normals.

So, using these values all the plane normals can be plotted in a stereographic projection. Now once we have got the plane normals after all what are plane normals these are the poles or planes. So, we can measure the angle between the different poles here all the plane. And you see if we have you know with us a values of in depth you know internal angles for the different planes in the cubic system because we are working on the cubic system, then comparing these values between the poles it is possible to identify the planes which have given rise to those diffraction spots in the back deflection Laue method.

Now, we will take up a concrete example to show how it works.

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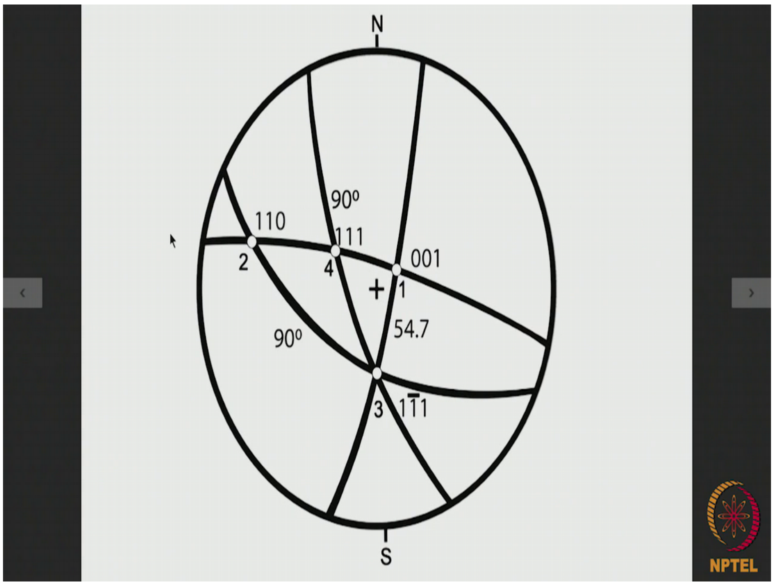

Say for example, we have a situation that this is the projection stereographic projection of a number of plane normals which we got by using the Greninger chart. And suppose this is another line you know which shows this is another line showing the poles belonging to both of the planes belonging to a particular zone. Say for example, between this pole and this pole if we find out by measurement it is about 90 degree say, and between this pole and this pole again we find it is 90 degree. Say this is another longitude line another great circle which shows the poles belonging to the place of another zone and suppose this angle is 54.7.

So, we have got a situation where we have got the longitude lines showing the poles of different zones. So, this shows the poles belonging to a particular zone, this shows the poles of another particulars zone, and this shows the poles of a third particular zone. Say this is designated 1, this is designated 2, and this is designated 3. Suppose we measure out the angle between 1 and 2 and it comes out to be 90 degrees. Again between 2 and 3 it comes out to be 90 degrees. And between one and 3 it comes out to be 54.7 degrees.

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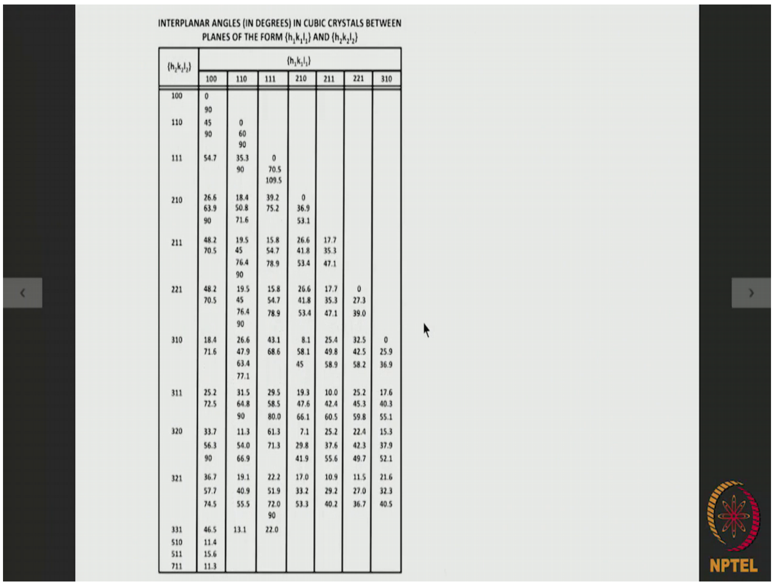
INTERPLANAR ANGLES (IN DEGREES) IN CUBIC CRYSTALS BETWEEN PLANES OF THE FORM $\{h_1k_1l_1\}$ AND $\{h_2k_2l_2\}$

$\{h_2k_2l_2\}$	$\{h_1k_1l_1\}$						
	100	110	111	210	211	221	310
100	0 90						
110	45 90	0 60 90					
111	54.7	35.3 90	0 70.5 109.5				
210	26.6 63.9 90	18.4 50.8 71.6	39.2 75.2	0 36.9 53.1			
211	48.2 70.5	19.5 45 76.4 90	15.8 54.7 78.9	26.6 41.8 53.4	17.7 35.3 47.1		
221	48.2 70.5	19.5 45 76.4 90	15.8 54.7 78.9	26.6 41.8 53.4	17.7 35.3 47.1	0 27.3 39.0	
310	18.4 71.6	26.6 47.9 63.4 77.1	43.1 68.6	8.1 58.1 45	25.4 49.8 58.9	32.5 42.5 58.2	0 25.9 36.9
311	25.2 72.5	31.5 64.8 90	29.5 58.5 80.0	19.3 47.6 66.1	10.0 42.4 60.5	25.2 45.3 59.8	17.6 40.3 55.1
320	33.7 56.3 90	11.3 54.0 66.9	61.3 71.3	7.1 29.8 41.9	25.2 37.6 55.6	22.4 42.3 49.7	15.3 37.9 52.1
321	36.7 57.7 74.5	19.1 40.9 55.5	22.2 51.9 72.0 90	17.0 33.2 53.3	10.9 29.2 40.2	11.5 27.0 36.7	21.6 32.3 40.5
331	46.5	13.1	22.0				
510	11.4						
511	15.6						
711	11.3						

Now, if we have a chart which gives us an idea of the angles between different planes and different between 2 different types of planes in the cubic system. We say that 90 degree angle can be between a 0 0 1 and 1 1 0 plane or between a 0 0 1 and a 0 0 1 plane or between a 1 1 0 and 1 1 1 plane. So, here we find that between with this pole and this pole it is 90 degrees, between this pole and this pole it is 90 degrees.

So, what are the possibilities? Possibilities are from this you can easily see that 0 0 1, 1 1 0 is 90 1 1 0 and 1 1 1 is also 90. So, if this is 0 0 1 tentatively and if this is 1 1 0 tentatively, then angle between 1 1 0 and 0 0 1 as we have seen here is 90 and angle between 1 1 0 and 1 1 1 is also 90 degrees. So, we see that this is the case where angle between this pole at this pole that is a if it is a 1 1 0 and if it is 0 0 1 then it is 90 as we have measured and between this pole and this pole again between a 1 1 0 and a 1 1 1 plane is it 90 degree.

So, we have written down against one as 0 0 1 against 2 as 1 1 0 against 3 as say one bar 1 1 whatever it is. Now you see this does not you know this fixes the location of point to unambiguously. But you see it does not fix the location of the points 1 and 3 unambiguously, but this could be there; that means, 1 1 1 could be there 0 0 1 could be here you know, in that case also this angular relationships are satisfied. So, in order to be convinced that this is really a 0 0 1 and this is the 1 1 1 location we take one more longitude line which shows the you know locations of the posts of another particular zone passing through this.

So, what we find we measure the angle between this particular point 4 and 2, and we found out that it measures 35.3 degree and this 4 and 3 the we find measure the angle between the 2 and we find it is 70.5 degree, but you see 35.3 degrees the angle between a 1 1 1 and a 1 1 0 plane. So, naturally it automatically tells us that this must be a 1 1 1 plane, in a similar manner you know 70.5 degree is angle between a 1 1 1 and a 1 1 1 type plane.

So, this must be a 1 1 1 type of plane. So, that leaves us the position of these spot as 0 0 1. So, now, we have a situation that this spot is a 0 0 1 spot this is a 1 1 1 pole this is the 1 1 0 pole, and this is say one bar 1 1 pole. Now our job is to find out what is this particular pole here, because this is the pole which is nothing but the perpendicular to the surface of the crystal. So, the normal to the surface of the crystal is given by this central point here. So, our job is to find out what particular pole is this what is the you know position of this particular pole, and that will give us an idea of the orientation of the crystal, because if I if we know the perpendicular the pole of the surface crystal surface we know what is the orientation what is the you know HKL of the plane parallel to the surface of the crystal.

So, this is the next job that we are going to do.

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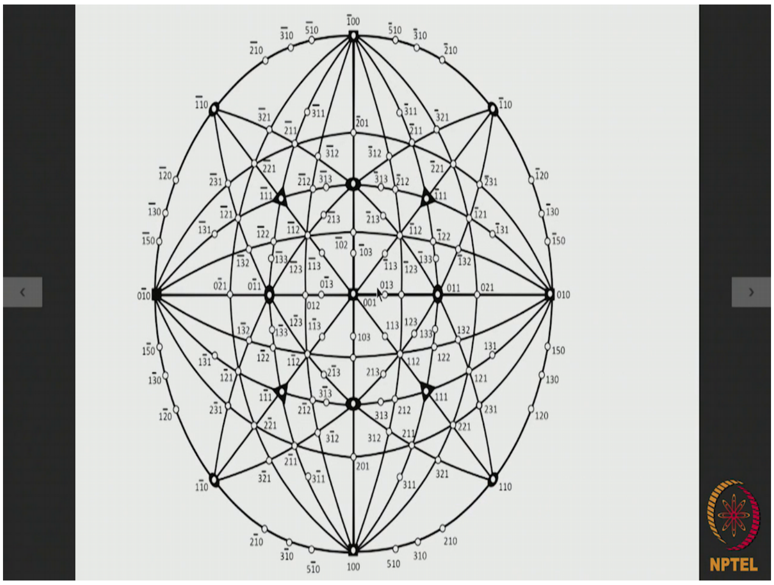

Now you see, when you look at is 0 0 1 standard stereographic projection, you can easily locate that this is the 0 0 1 position this is the 1 1 bar 1 position and you know you can have all those poles up to bar 1 bar 1 0. So, knowing this you can we can give tentatively you know the locations of all this flow poles and mark them. Now we are interested in this pole. So, what we are going to do how to find it out.

Now, what we do is here is this is our 0 0 1 and this is our bar 1 bar 1 0. So, our you know pole for which we are trying to figure out who identify that pole it lies somewhere over here. So, in that case what we can do instead of choosing the entire 0 0 1 stereographic projection. We can choose only this particular unit triangle which is defined by the poles 0 0 1 bar 1 bar 1 1 and 0 bar 1 1. And we know that the pole which we want to identify lies straight over here.

So, we draw the outline of this unit triangle over here.

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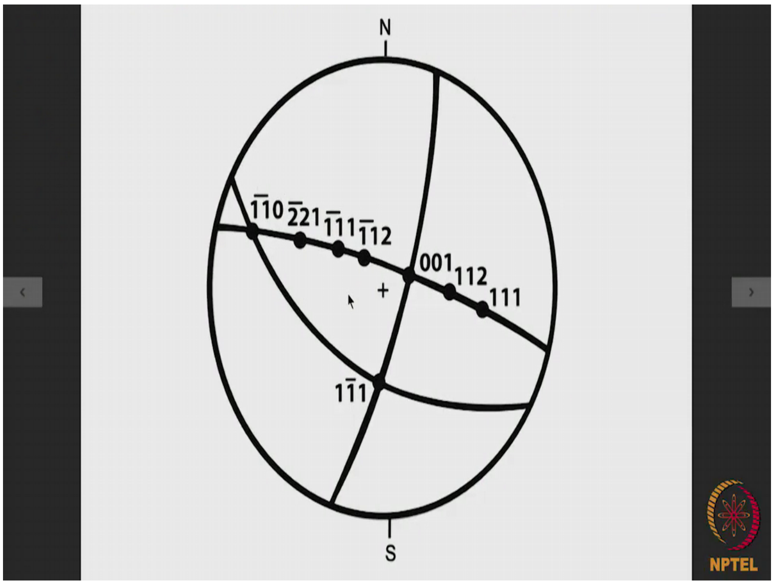

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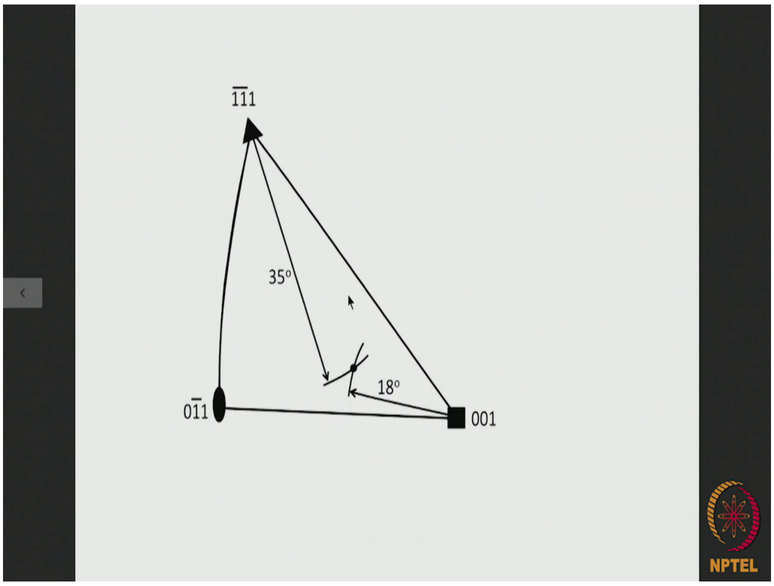

And this is the pole identity of which we want to figure out. Now it is very easy to find out what is the angle between this pole and the unknown pole here, this world and the unknown pole over here, this pole and the unknown pole or here. And we can measure the distance and from a table which shows the values of the interpreter angles in the cubic system. It is quite possible to pinpoint which particular pole is this because this pole is nothing but it designates the normal to the crystal surface.

So, when we can find out, but which pole is this say it is the HKL spot, then we can easily say that the crystal surface has the orientation the plane the crystallographic plane parallel to the crystal surface has the orientation h k m. So, this is the principle behind the determination of single crystal orientation using X-ray method.

Now normally when we want to have a reasonably accurate measure of the orientation the back reflection Laue method or the transmission Laue method is used. But when we have to examine the orientation of a very large number of samples, then the diffractometer method will be somewhat advisable because it can analyze these very, very quickly if not with this much of accuracy.