Texture in Materials
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Module - 04
Principles of texture measurements by X-ray diffraction
Lecture - 25
Principles of Pole Figure Measurements by X-ray Diffraction

Good afternoon everyone and today we will continue with the module 4, which is Principles of texture measurements by X-ray diffraction. So, this is lecture number 25, where we will try to understand the Principles of Pole Figure Measurements by the X-ray Diffraction.

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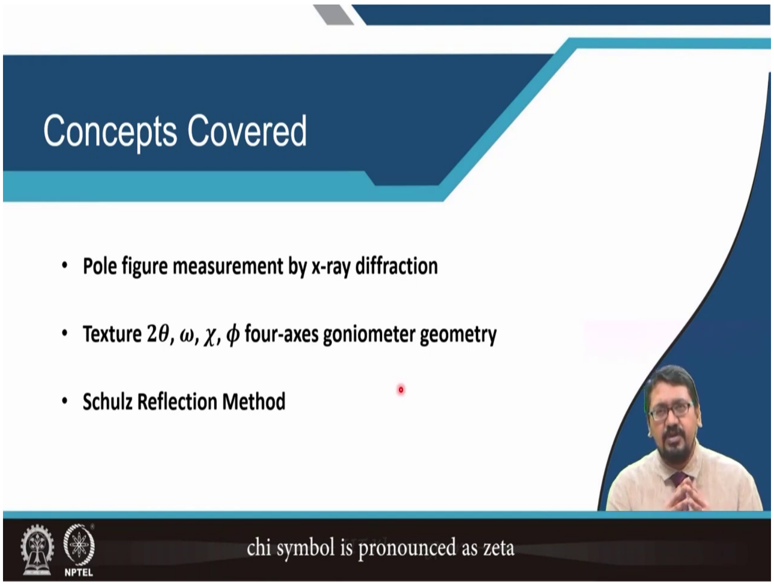

The concepts that are covered in this course lecture is pole figure measurements by X-ray diffraction, texture measurement using 2 theta, omega, zeta, phi four-axes goniometer, its geometric Schulz reflection method. So, before we start this lecture we will I will try to say here is that we understood how continuous X-ray is being generated. We understood that how characteristics X-rays forms when the voltage is increased.

We also understand that we use the mass absorption coefficient mechanism to take away k beta radiation to increase the k alpha radiations intensity. Of course, the k alpha and k beta k alphas radiations intensity is also reduced, but the ratio of k alpha by k beta radiation is increased to obtain a proper characteristic radiation and this characteristic radiation can be utilized for diffraction purpose. So, diffraction methods are used to find out various kinds of things. For example, the various crystals can be diffracted poly crystals or other crystals single crystals where you can get the relationship between theta and the hkl planes based upon the Bragg's law.

On the other hand we can find out various kinds of strains which are present in the material which changes the value of d and therefore, the theta of the material is varied means the peak shifts to the lower theta or higher theta or the peak broadens.

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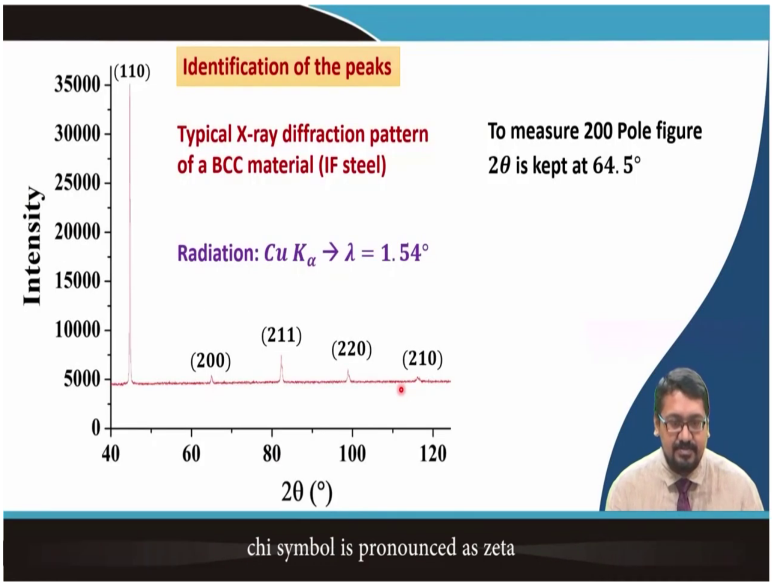

So, these methods as I said when we use a characteristic X-rays we can get and that X-ray is incidenting on a certain metal or an alloy, we can get these kind of characteristic peaks which is characteristic of this particular material on which the peaks are being incidenting. So, here is a typical X-ray diffraction pattern of a BCC material. Basically, this is the interstitial free steel which we have shown in the earlier lecture also and this is the incident the copper k alpha radiation of lambda equal to 1.54 has incidented on it and it is corrected by the nickel filter.

And we can see that various planes hkl planes 110 has a particular 2 theta value right something say in and around 44 or something. And for example, for 200, it is something like 64.5 or something. So, if we do a texture analysis and before we start doing our texture analysis texture measurement from the X-rays we need to know the values of the thetas for the hkl planes for which we are doing the textural analysis.

So, when we do an X-ray texture measurement we first take a particular peak. Say for example, we take the 110 peak and we fix the theta for that particular peak. Say for example, here it is something along around 42 or 44 something like that or and then we measure the whole sample for the particular pole figure and I will show you how we measure the pole figure by alpha, beta rotation. Alpha which is along TD and beta which is along the ND of the sample we I will show that how the sample is kept in the in the X-ray texture goniometer. And then one by one more than one hkl planes are measured. So, first 110 is measured and then 200 and if it requires then 211 like that each plane is measured one by one in the X-ray texture goniometer.

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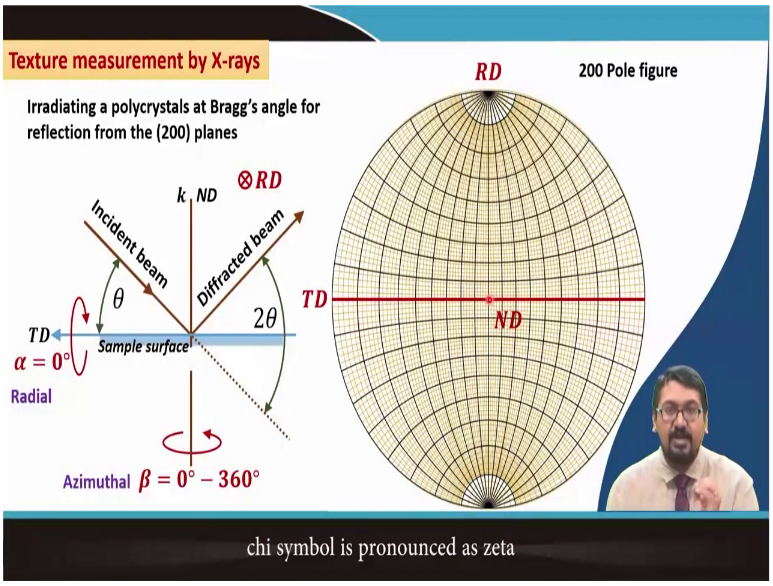

So, let us see how this X-ray texture is being measured. So, we as I said we are trying to measure 200 plane. So, we have kept the theta of that incident angle fixed to 64 4.5 degree based upon the previous diffraction the diffraction experiment in the previous slide right.

So, the incident beam is fixed at theta here is the sample . So, diffraction beam is also at theta. So, the angle between the incident beam and the diffracted beam is 2 theta right. The sample is kept in such a way that the RD ND and TD that means, the important sample reference directions are very well known. Say for example, here the sample is kept in such a way that the RD is going inside the the paper, the ND is vertical towards the top the TD is on this side right. Now, as I said that the measurement of texture is done in such a way that the incident radiation and the diffracted radiation is fixed at a certain theta. So, that it can only measure the that particular hkl plane 200 for example, in this case and this measurement is actually plotted on the pole figure using a Wulff net something like this right.

So, let us say that in this Wulff net RD is in the top, ND is in the center and TD is in the side. It is in the same way as in this case the sample is kept where our ND is in the top and RD is going inside. So, if I rotate the sample and observe it from the above and then we can see if we observe it along ND then we can see that ND is in the center, RD goes to the top and TD remains at the same position.

Now, when there is no rotation. So, we give a rotation alpha another rotation beta. As I said alpha rotation is along TD and the beta rotation is along ND. The alpha rotation is basically known as the radial rotation and the beta rotation is known as the azimuthal rotation. Now, if when you give when we have not given any rotation of alpha and beta that means the intensity basically coming corresponds to the bisector of the incident under diffracted beam which is exactly the ND right. So, the ND of the sample is the same is in the same direction as the direction of the incident beam.

So, when at this position if the sample is irradiated with a copper k alpha radiation lambda equal to 1.54 for theta equal to 60 some 64.5 degree or something then the if any intensity for from the 200 planes comes those 200 planes basically lies perpendicular to this ND directions right. So, all the 200 planes which are lying on the surface of the sample should get diffracted. We should remember that the incident beam and the diffracted beam are not straight like that, but it is basically a divergent beam falling and the convergent beam being diffracted. So, a large area is being irradiated. So, at this position when alpha equal to 0 and beta is equal to 0, the ND where is the position where the intensity is being observed.

Now, if we try to increase if we rotate the beta from 0 degree to 360 degree keeping alpha is equal to 0 then also that the irradiation is along k which is parallel to ND and therefore, even with rotation from beta that is azimuthal rotation along ND from 0 degree to 360 degree, the position of the intensity peaks remains exactly at this position ND.

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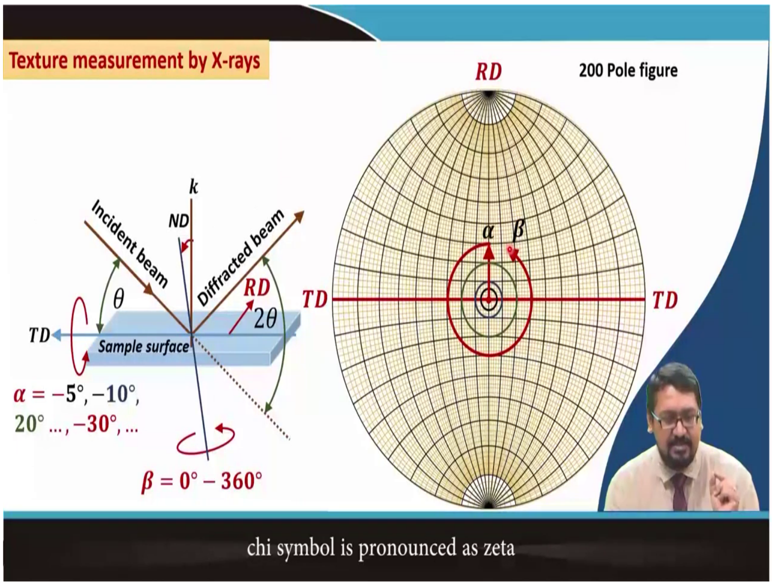

Now, let us say that we rotate the sample by alpha equal to minus 5 degree and why I have said minus 5 degree? Because, we are rotating by clockwise direction that is by the left hand thumb rule. So, what we are getting? We are obtaining a rotation along TD that is alpha equal to minus 5 degree and then while we have rotated by minus 5 degree the ND also will shift from k right.

Now, the bisector of the incident beam and the diffractor the diffracted beam which is k is little separated that is 5 degree separated from the ND. Now, this new ND is there which can be rotated through beta rotation from 0 degree to 360 degree. Like that the alpha can increase to 10 degrees, 20 degrees and 30 degrees and each time the beta has to be rotated from 0 to 360 degree. Let us see that when alpha is rotated by minus 5 degree by the clockwise direction what happens when the beta rotates from 0 to 360 degree. that I am showing a small black colored circle in the this pole figure. We are trying to draw a pole figure using this Wulff net.

This Wulff net I forgot to mention has graduation in orange and graduation in black and if the graduation in black it is 1, 2, 3, 4, 5, 6, 7, 8, 9. So, it is a 10 degree graduation. It starts from 0, then 10, 20, 30, 40, 50, 60, 70, 80, 90, right. So, it is it is a 10 degree graduation because we know that from if it is 0 then here it is 90; if it is 0 here it is 90 right and then it is divided by the orange smaller grids of the Wulff net.

Now, we have calculated that from here to here that is half from here to here is 10 degree then here to here is 5 degree. So, after a 5 degree rotation of alpha the alpha reaches this position. At this position, how it reaches this position? Because that we are measuring at the bisector of the incident beam and the diffracted beam which is 5 degree away from ND on that side right. So, the position is now here it is just 5 degree away that side.

Now, this is a position where beta is basically 0 right and while the beta is increased from 0 to 5 and 10 and it goes to 90 and then 360, it goes through this line and it rotates like this and it reaches here at 360 because beta is equal to 360 means, beta is equal to 0. And then we can see that we at the next go we will increase alpha by 10 degree and rotate along ND that is the beta rotation from 0 degree to 360 to trace this whole position and then we will rotate then we will go to 15 degrees and then we will go to 20 degrees and do the same thing and then we will keep on tracing this whole Wulff net for the positions of the hkl planes with respect to alpha and beta.

Now, to let that while we are tilting these specimens from alpha equal to 0 degree to 5, 10 and 15 and 20, 30 and we are going up to 90 degrees if possible then the bisector between the incident beam and the diffracted beam is k. So, the planes which are perpendicular to the k is only being measured right that plane will always have an angular deviation from ND in a similar manner as we move through alpha and beta and that is what we are plotting in this pole figure right.

Now, you look into this that let us say; let us when we go for alpha equal to 30 degree and I am showing this because it is easier to understand here it is having a larger circle and it is easier to understand here. So, when we are go we are here then alpha is equal to 0 and then we are going here that is alpha equal to 10, 20 and 30 degrees; that means, the sample is rotated slightly rotated by alpha by minus 30 degree this side. Then when we are rotating along beta and we are going like this and like this and like this and like this we are going and reaching this 360 and we are tracing that at any point if we can get that particular hkl plane for this particular Bragg's angle theta. So, let us take this alpha equal to 30 degree and see.

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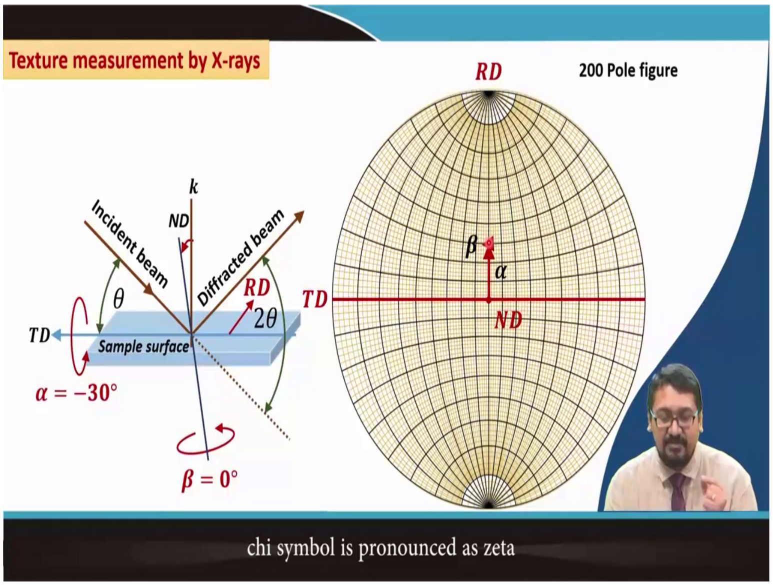

So, when we reach alpha equal to 30 degree we are here and we are observing the intensity at k that is bisector between incident and the diffracted beam which is this position this position right, alpha equal to 30 beta is equal to 0 right.

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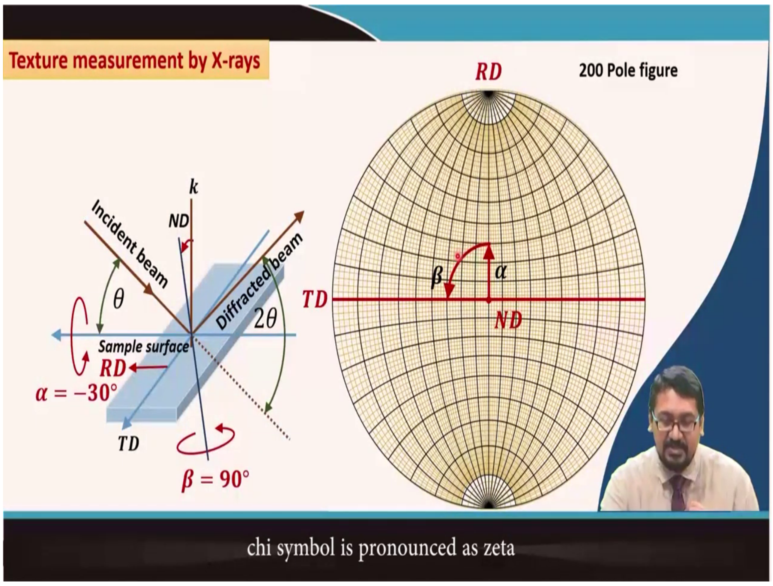

And once we rotate it once we rotate it and increase the beta from 0 to 90 degree the sample is rotated like this . And when the sample is rotated now the TD is here and the alpha is basically sorry the RD goes here right. So, the ND remains the same the position of the ND remains the same just tilted by 30 degrees from the k right. So, that when we are rotating the ND comes here and then we are measuring from beta equal to 0 to 90 degree throughout.

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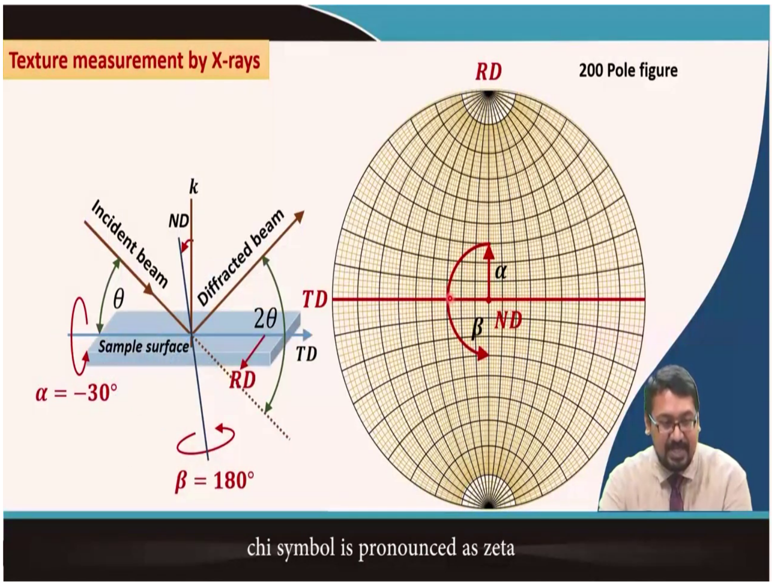

And then we are going again and we can we rotate b t sorry beta and we can get the intensities of that particular hkl plane 200; 200 for this case up to 180 degree. And you are seeing that the sample is rotated. This time the TD is here. The RD is now in the front right and then we can go again.

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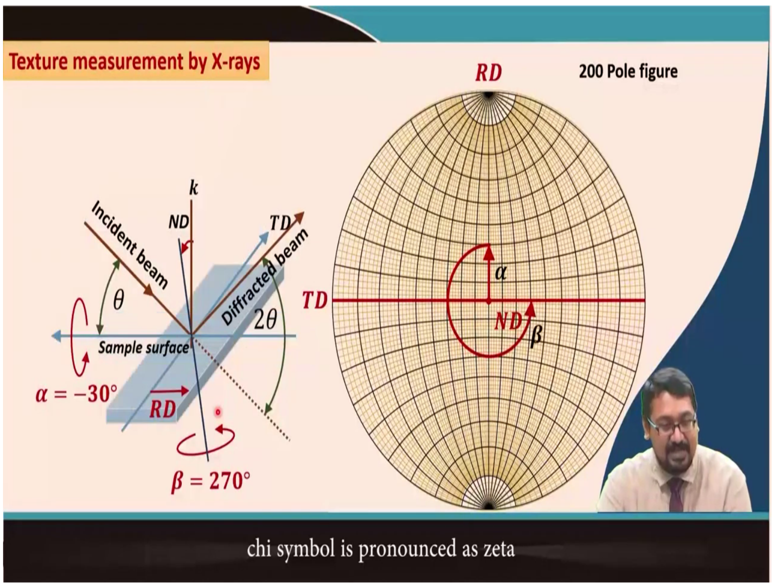

And now, that TD is going backward, where RD is on this side and then we can see that we are tracing the whole sample and we have gone up to 270 degrees.

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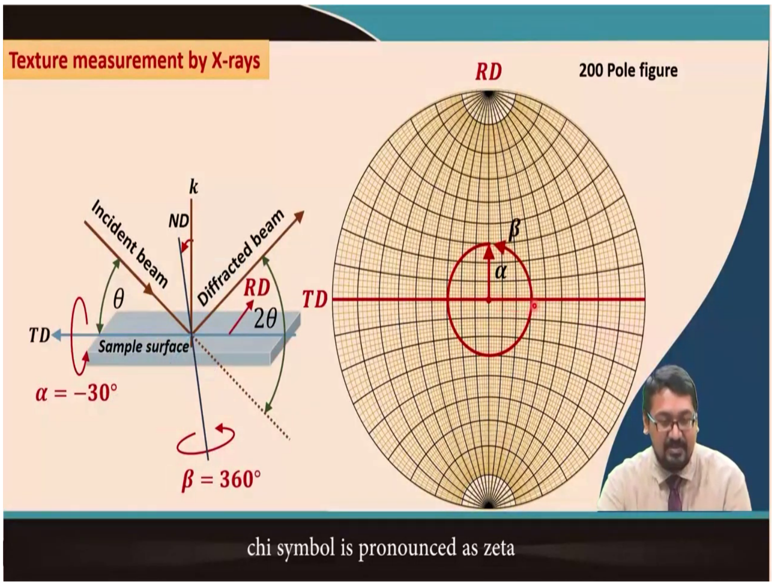

And then we are going up to 360 degrees and like that we will increase alpha from 30 degrees to 6 means 35 and go up to 90 degree and we will trace the whole alpha beta to measure the intensity point of this 200 planes. And like that we will have to measure the intensity points of 110, 200 and other hkl planes in order to get the complete information of the texture of the material.

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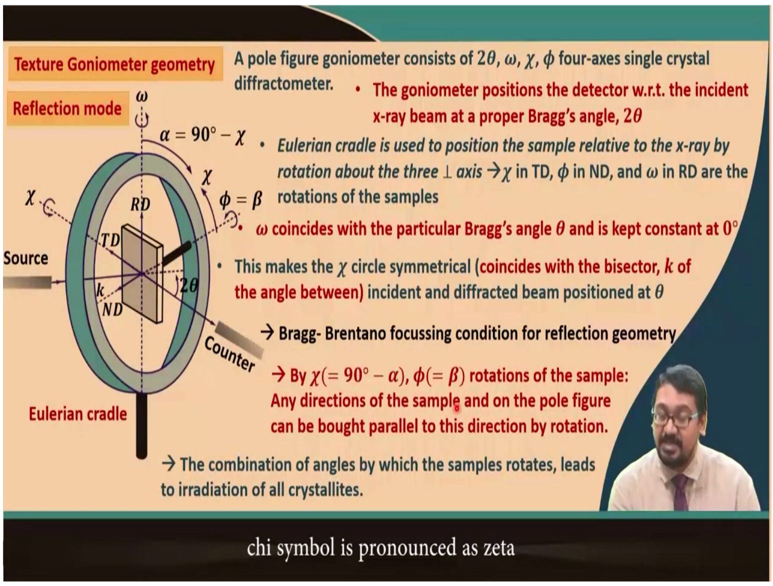

So, that this kind of alpha beta rotation is done using a texture goniometer with a certain geometry. This goniometer, see most of the case is being used in a reflection mode because the X-rays that are produced generally in laboratories are not so intense or does not have the lambda smaller or it is not short wavelength and they are not hard enough to penetrate even the thinnest specimen in order to obtain diffraction pattern from the in a transmission mode.

But is neutron diffraction or even synchrotron diffraction can be used to get transmission mode of this kind of diffraction experiments. Now, let us look into this condition, where we are showing the schematic of this texture goniometer. And this texture goniometer you will see that we have shown a circle and this is known as the the Eulerian cradle.

If the source of the X-ray is here and the sample is kept here, the sample is always kept in such a way that it the RD is most of the time is it is kept on the top vertical. The TD is along here, the ND is in the front, the source is kept at a particular theta to the counter 2 theta to the counter for that particular hkl plane that has to be measured and that what we said. And that along RD there is a omega scan. The omega scan basically coincides with the 2 theta, which is the Bragg's angle 2 theta and it is fixed it is first rotated and it is fixed for that 2 theta and the omega is made equal to 0 and kept fixed for that particular sample.

Then another rotation which is most important rotation is the rotation along TD and this is basically the zeta rotation. And this zeta rotation as I said is according to the right hand thumb rule as that these are a goniometer which is a four-axes single crystal diffractometer consisting of four angles; 2 theta, omega, zeta and phi. As I said omega which is a rotation along RD is made so that it means it takes and confronts the 2 theta to get that 2 theta and then we make the omega equal to 0.

The most important rotation is along TD which is the zeta rotation. The zeta rotation is basically, again a right handed thumb rule rotation and therefore, as I said in the earlier slides that alpha is the clockwise rotation that is by the left hand thumb rule. Therefore, the relationship between zeta and alpha is zeta is equal to 90 degree minus alpha or alpha is equal to 90 degree minus theta. So, this zeta rotation is along TD and along the ND the rotation is known as the phi rotation which is again anticlockwise or means as per the right hand thumb rule and the phi rotation is equal to the beta rotation that we have observed in the earlier slide. So, this is how the texture goniometer geometry looks like.

So, the goniometer position the detectors with respect to the incident beam in a proper Bragg angle 2 theta as I said, the Eulerian cradle is used to position the samples relative to the X-rays by rotation about these three X-rays. So, zeta along TD, phi along ND and omega along RD; so, these rotations are the samples are used to keep it in the Bragg's condition. As I said earlier the omega basically coincides with the Bragg's angle theta and after a certain Bragg angle is kept for a certain hkl planes the theta this omega is made into 0. So, what we understood from this is that the incident beam and the the diffracted beam is such a way that the the bisector of that beam that we were showing by the bisector k right lies in the in the zeta circle right.

So, it is such that the zeta circle is basically symmetric around this bisector between the incident beam and the diffracted beam. So, under such condition, where the zeta circle is symmetrical between the incident and the diffracted beam when it is positioned for a certain theta b that is the Bragg's angle for a certain hkl plane this means that this is in the proper focusing condition for the reflection geometry to obtain diffraction for a certain hkl plane and is known as Bragg-Brentano focusing condition.

So, zeta rotation along TD and phi rotation along ND is the one which is done simultaneously so that to scan the whole alpha beta because zeta is 90 degree minus alpha and phi is basically beta. And so, any direction of the sample or any plane of the sample can be bought perpendicular to the k, which is basically the bisector of the incident and the diffracted beam that is can be bought in the Bragg-Brentano focusing condition.

Or you can say that the pole figure the poles of the pole figure can be brought parallel to the direction of by this rotation right. So, a combination of angles by which the samples rotates means mainly alpha and beta. Of course, omega and 2 theta has the significance leads to the irradiation of all the crystallites present in the poly crystalline material, which the all the planes can be radiated which becomes parallel sorry perpendicular to the k that is the bisector between the incident and the diffracted direction during the alpha beta rotation. So, that the whole poly crystal can be scanned while the alpha and beta is changing.

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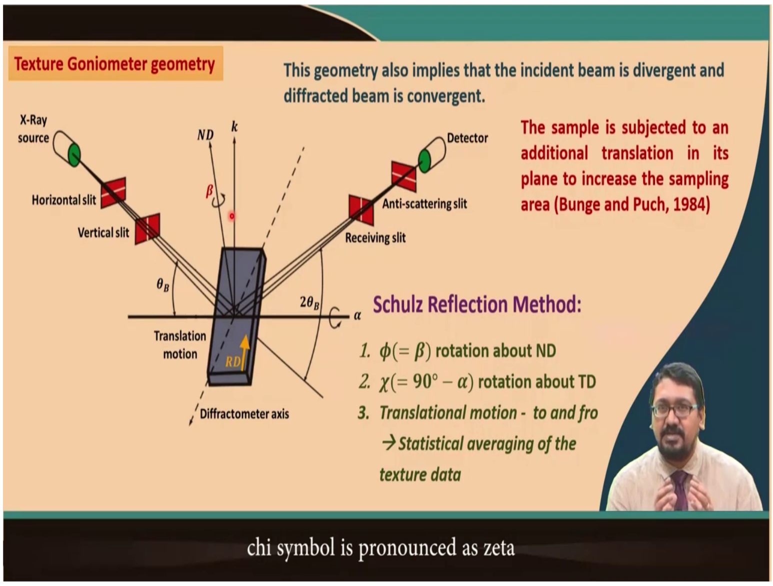

So, that the texture goniometry geometry consists of a X-ray source, a source which has a divergent beam and a convergent diffracted beam. And you in order to make this divergent beam and the convergent beam smaller various kinds of slits are used. horizontal divergence horizontal slit and vertical slits and then receiving slits and anti scattering slits are used in front of the detector.

The sample is kept in such a way that the RD is kept vertical, the TD is kept so that the alpha could be rotated along TD and the ND in which the beta is rotated. So, that the sample additionally is subjected to a translational motion or a reciprocating motion so that a large area of the sample could be irradiated. So, that a large volume if irradiated we can get a larger information and it will be more representative of the sample. Because by while we are saying pole figure measurement using X-ray texture we are saying that we are basically measuring global texture of the material which should be representative of the property of that material. So, along with all this and when the Bragg-Brentano's focusing condition is met the method which has to be satisfied to carry out this X-ray texture measurement that is the pole figure measurement is known as the Schulz reflection method.

So, the one thing the that has to be satisfied in this is that phi is rotated along ND, zeta is rotated along TD and there has to be translational to and fro motion for statistical averaging and if all these three things are satisfied in an X-ray textured goniometer then the texture measurement are complies the Schulz reflection method.

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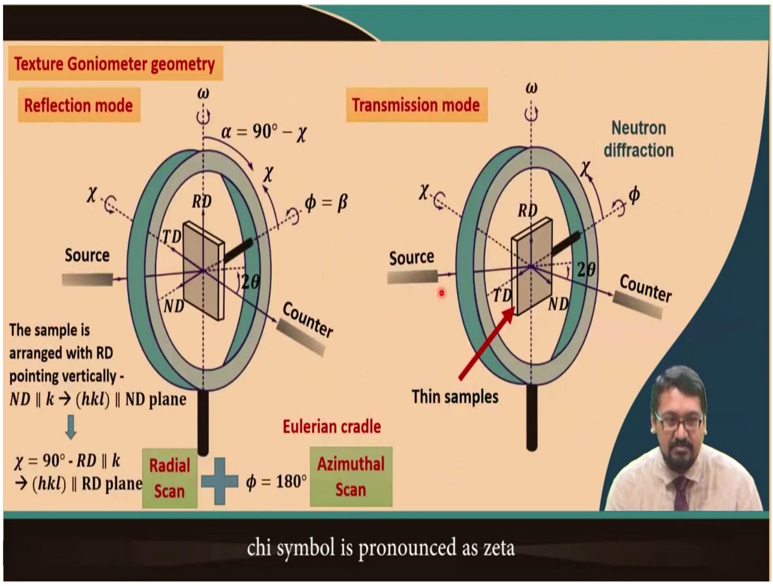

So, while we are talking about the texture goniometer geometry we are showing that we have shown this in terms of the reflection geometry right. In case of neutron diffraction or synchrotron diffraction transmission mode can be also utilized and you can see that there is a subtle difference between the samples which are kept in the Eulerian cradle in case of reflection geometry and in case of transmission geometry.

In case of the transmission geometry the sample is kept like this and now that even though RD is kept vertical and along which omega is the rotation. So, as to fix the 2 theta and then by rotating the omega we fix the 2 theta for a particular hkl plane and then omega is bought to 0 and it is kept fixed. The rotation of the phi is now is done along TD. Earlier in case of the reflection case it was done along ND and the zeta rotation which was earlier along TD is now basically along the ND. So, there are subtle geometrical differences between the the reflection mode and the transmission mode and based upon that one can easily calculate the changes in the that occur and can calculate e for the transmission mode the alpha and beta.

So, if we look here what I have written is, the sample is arranged with RD vertical in case of the reflection mode that and that is what we learned in the last slide that keeping RD vertical and ND is initially kept parallel to the k that is the bisector between the incident and the diffracted radiation.

And that when the ND is rotated the hkl planes which are basically perpendicular to the ND perpendicular to ND; that means, the hkl planes which are parallel to the ND planes actually get diffracted at ND that is parallel to k. Now, there is a zeta rotation. So, this is basically summarizing where the zeta can rotate from 0 to 90 degree and at 90 degree the RD becomes parallel to k if if you rotate zeta and the RD will become parallel to k when zeta is equal to 90 degree.

And basically at 90 degree then we are basically measuring the RD plane right. So, the hkl plane which are basically parallel to RD and this is known as the radial scan as I said earlier. Along with this if we do an azimuthal rotation and that is phi is taken from 0 to 180 degree to 360 degrees then what happens? Then the rotation along ND. So, a combination of zeta and phi that is alpha and beta gives the whole pole figure of the sample.

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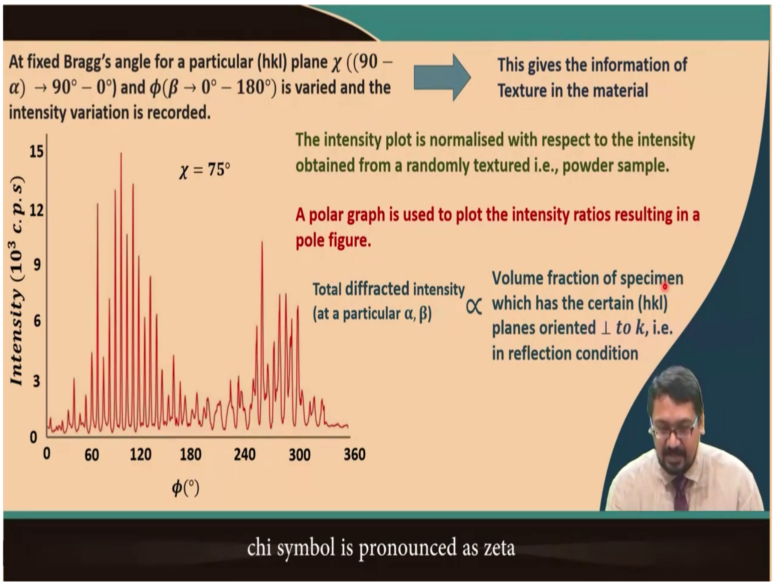

And in such a situation if we plot; if we plot intensity plot for a certain zeta and while we are rotating the phi and that is from 0 to 360 degree for a certain element or certain kind of poly crystalline material we can get an intensity plot which looks like this right something like that.

So, this is something very what how you will say a schematic and it will differ for different material for different processing conditions and all these things. And this plot basically is later plotted for every zeta that is zeta equal to 0 and 5 and 10 and up to 75 and maybe beyond for phi equal to 0 to 360 to plot the the whole pole figure for that particular hkl plane.

So, to summarize we fix a particular hkl plane and the Bragg's angle for it is constant and then we vary the zeta from 0 to 90 degree or 90 degree to 0 degree and we vary the phi from beta equal to 0 to 60 degree and we record the intensity. This recorded intensity is plotted in a pole figure and gives the information of the texture. This intensity plot is basically normalized with respect to the intensity obtained from the random texture that is the powdered sample. What it is done is that the texture of the powdered sample, a sample of the similar condition is powdered its strain is released by annealing. And then because the powder will have random texture the texture of the same hkl plane is actually calculated and this intensity will be same all over and this intensity is normalized to 1.

And when this intensity is normalized to 1, the intensity obtained for a real life sample that is a textured poly crystalline material is basically normalized with respect to this intensity. So, the polar graph from this kind of several graphs can be plotted and then the pole figure can be obtained right. So, the total diffraction intensity at a particular alpha beta point is proportional to the volume fraction of the specimen which means for which that certain hkl planes oriented perpendicular to the k and that means that is the reflection condition. Means, the volume fraction of that hkl plane which can be radiated that which are actually perpendicular to the bisector between the incident and the diffracted beam is basically shown in terms of intensity right.

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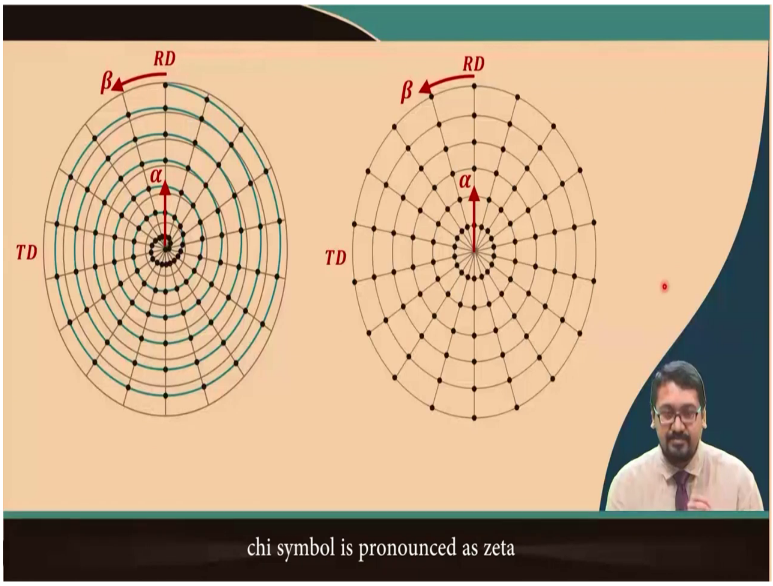

So, here we are showing two methods. This, the left one is the one which was initially done. So, stepper motor was used to vary both alpha and beta slowly and slowly at means different resolution. So, this we have shown very roughly. And so, if we vary it like that a spiral is obtained and there are various points which are shown here.

This points indicates that every point the texture the machine is stopped for a certain moment for radiation. So, it is radiated and the diffraction from that particular hkl plane is obtained and then alpha beta is changed. And in this way it is scanned throughout the hole pole figure from 0 to 90 degrees. And of course, the measurement cannot be done up to 90 degrees because after alpha equal to 70 degrees the the in the incident beam become almost parallel to the sample surface and so, the the intensity of the diffraction cannot be obtained. And we will come to that and this is known as the focusing error we will come to that in the later slides not in this lecture in different lecture.

So, what is it? That this kind of spiral is obtained where the sample is stopped at certain alpha and beta and then the intensity of the diffracted beam for a certain theta for a certain hkl plane is actually noticed. And thereby the whole pole figure was measured and this is was done earlier, but now things have changed and the machineries and the we can do a much higher resolution scan and many things.

So, here nowadays what happens that initially the alpha is rotated by few degrees and then beta is rotated from 0 to 360 degree to get a scan and then again the alpha proceeds by 2.5 degree or 5 degree and the beta is rotated by 360 degrees to get another scan. And in this way the earlier spiral is nowadays not done, it is done in terms of circle keeping alpha fixed and the beta moving and in this way the whole pole figure is actually measured a nowadays.

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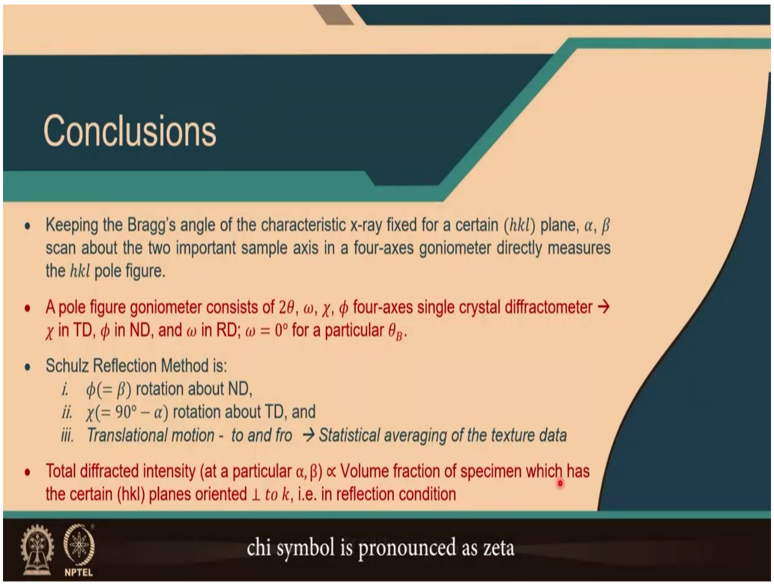

So, we can conclude from today's lecture that if we keep we have to keep the Bragg's angle constant for a particular characteristic X-ray for a certain hkl plane. And then the alpha and beta scan has to be done about the two sample important sample axis that is that is TD and ND in a four-axes goniometer and this directly measures the pole figure that particular hkl pole figure. And this is done for several hkl poles that shows a positive Bragg's diffraction right.

Now, the pole figure goniometer basically consists of this four-axes as I said 2 theta, omega, zeta and phi and this four-axes single crystal diffractometer the zeta is rotated about TD, phi along ND, omega along RD and omega is rotated as I said along RD to keep to bring the theta to theta b condition for that particular hkl and then omega is made equal to 0.

The most important thing that we should understand that the method of measuring the pole figure is known as the Schulz reflection method, where phi which is equal to beta is the rotation along ND has to be done; zeta which is equal to 90 degree minus alpha that is the rotation along TD has to be done. And the third a translational motion of the sample to and fro for statistical averaging has to be done to obtain the texture data. Most importantly this has to follow the Bragg-Brentano focusing condition.

Finally, the total diffraction intensity at a particular alpha beta is proportional to the volume fraction of the specimen which has a certain hkl plane oriented perpendicular to the bisector of the incident under diffracted beam that is the k and that is the reflection condition.

Thank you. Thank you very much.