Texture in Materials

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Module - 04
Principles of texture measurements by X-ray diffraction
Lecture - 27
Limitations and Errors in X-ray Texture Measurement and Corrections

Good afternoon everyone and today, we will continue with module 4 which is Principles of texture measurements by X-ray diffraction. This is lecture number 27 and in this lecture, we will learn the Limitations and Errors in X-ray Texture Measurements and Corrections and their corrections.

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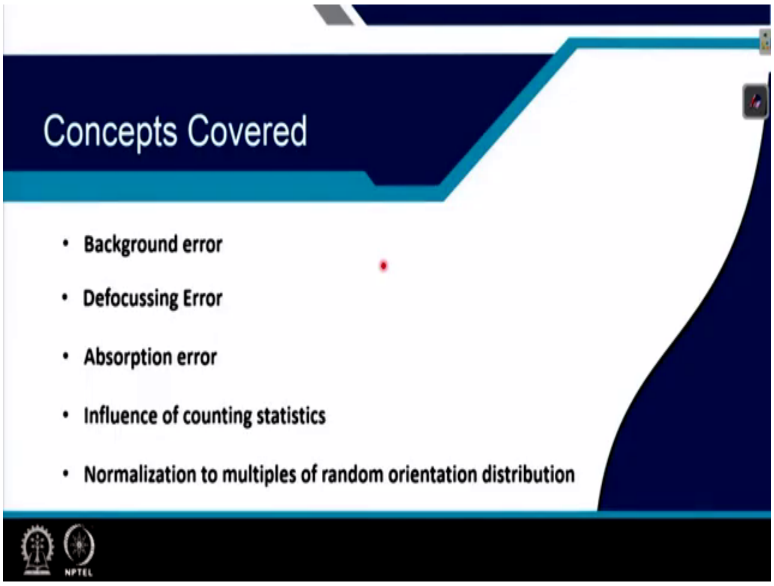

So, the concepts that will be covered in this lecture are background error, defocusing error, absorption error, the influence of counting statistics and normalization to multiples of random orientation distribution, MRD as we call it in abbreviated form.

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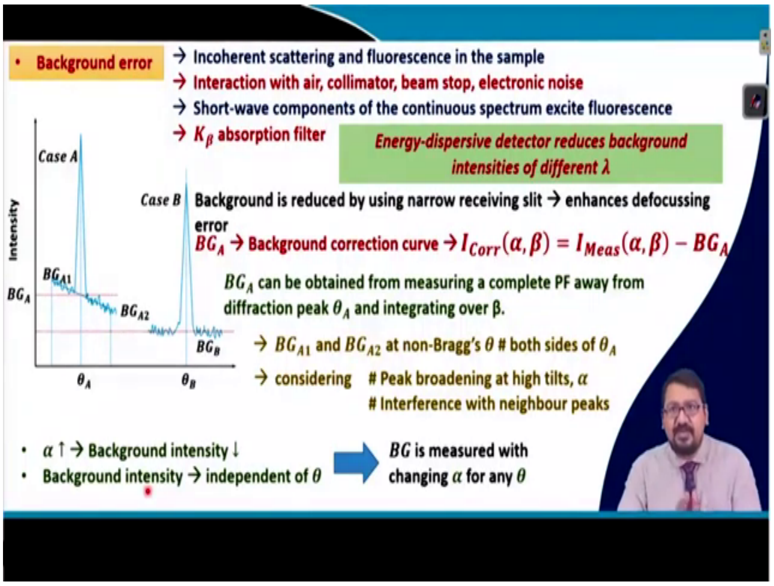

So, that the first thing that happens when we calculate pole figure is that along with the peak, there comes the background. So, here is one case, the first case, case A, where the background BG A 1 and BG A 2 have a little difference. So, BG A 1 is little larger than BG A 2. On the other hand, in the case B BG that is background B is constant in both the directions. So, for different measurements of theta or different measurements of alpha, the background may be different in either sides or same in both the sides right. How the background is created. The background occurs because of the presence of incoherent scattering and fluorescence in the sample. There could be interaction with the air, collimator, beam stop and electronic noise, which can create background.

On the other hand, short wave components of continuous spectrum also excites fluorescence, which creates background. Background is basically created when K beta absorption filter is used because in that direction, the background along with the K beta is removed; but on the other direction, on the higher of lambda, there is still a background. Now, energy dispersive detector, is used to reduce background incentive intensities at different wavelengths right. But , the background is mostly reduced or can be reduced technically, instrumentally through using a narrow receiving slit.

But you will see that by using a narrow receiving slit will increase the defocusing error; defocusing error, we will teach in the next slides, coming slides. So, what we basically do?

The background that we have here is basically removed by subtracting the background or the average of the background from the measured intensity of the peaks for the whole alpha beta.

So, the intensity of the alpha and beta, the corrected intensity is equal to intensity measured minus BG A or BG B whatever it is. So, now, BG A can be obtained you will see by measuring the complete pole figure away from the Bragg's diffraction peak. So, we have to measure the complete pole figure, the alpha beta scan at a theta which is away from theta which fulfills the Bragg's law and then, you have we what we do? We calculate the background for increasing alpha and then, we just have to integrate it over the beta. Because the background error does not change with the change in the beta from 0 to 360; only it varies when the alpha is increased or the theta is increased.

Now, the BG A 1 and BG A 2 are non Bragg's theta on both side of theta A which is the Bragg's condition right. Now, considering we while we calculate this background correction or the background error, we have to consider the peak broadening which occurs when the alpha is increasing. So, we should remember that the intensity peaks at Bragg's angle are very sharp when the alpha is less and when the alpha increases the intensity peaks broaden right. Total intensity of that peak remains same, but the intensity peak reduces and it gets broadened. So, secondly, while we are calculating this background, we have to be very sure that there must not be interference between the neighboring peaks.

So, that as alpha increases, the background intensity basically decreases right. Background intensity basically is most of the time independent of the theta and therefore, the background is basically measured with increasing alpha for any particular theta and then, is integrated over the beta right and that is how we calculate the background and then, we subtract the background from the measured intensity to get the correct intensity for alpha and beta for obtaining intensity peaks for a pole figure.

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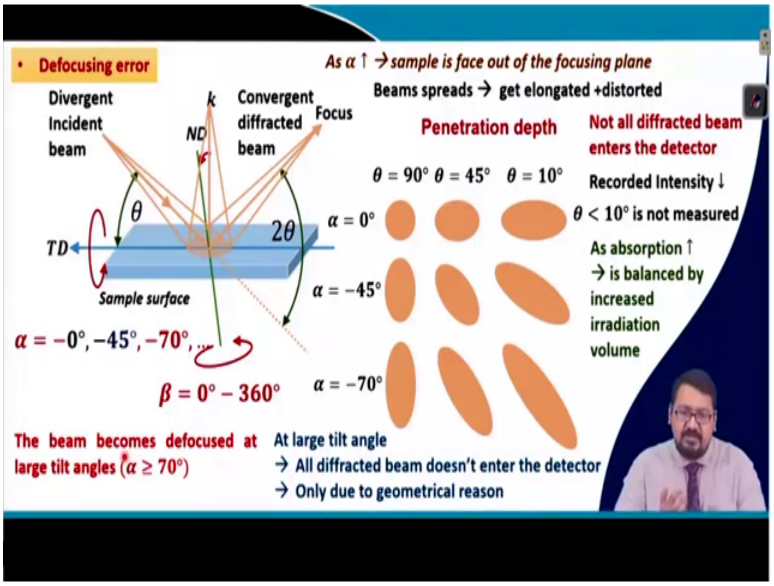

The second most important error that occurs during the calculation of the pole figure, calculation of the alpha beta scan is defocusing error. the setup schematically looks something like this right. This is the schematic of the setup. We have the divergent incident beam. So, here is the X-ray beam which is divergent is falling on the sample and here is the convergent beam which is diffracted from the sample and is going to the detector. So, the detector is at some position, where the convergent beam is focused right.

So, theta is the Bragg's angle, as I said alpha is the rotation that occurs along the TD. So, alpha is equal to 0 and then, it increases to 10, 5, 15 and 20 and 45 and then, it goes to 70 in the minus because it is in the using the Right Hand Thumb Rule. Sorry, it is not in the Right Hand Thumb Rule, it is in the Left Hand Thumb Rule. So, it is minus. Now, that when the alpha is 0; that means, there is no tilt and the theta is at 90 degrees; that means, when the the source of the X-ray is kept here and it is falling over here. So, the theta which is the angle between this incident beam and the TD is 90 degrees, then that as per geometry of the electromagnetic radiation incidenting on the surface at 90 degrees, the incidenting beam will have exactly circular circular, what you call it?

Shape falling over the sample. Now, as this is the point, where the focusing is 100 percent true because alpha is equal to 0 and there is the theta is 90 degrees, but that when we increase the theta.

Now, see in this case, that when alpha when theta is equal to 90 degree, the incident beam and the detector should be at the same position to get any kind of intensity or diffraction which is not possible; definitely, theta has to be less than 90 degree to get a diffraction. So, as theta is decreasing and it reaches say for example, 45 degrees. So, once theta is decreasing from 90 degrees to 85 and 80 and it goes to 45 degree, slowly and slowly defocusing starts. So, the beam starts to get elongated horizontally .

So, as this beam the incident diffracted incident beam elect X-ray beam is the theta is decreased from 90 degree and it goes like this and it is falling like this and it goes like this to become 45 degree, the the beam becomes elongated while it falls on the surface. When it becomes higher at theta equal to 10 degree, it becomes too much elongated because when the beam is falling from this direction, the beam will be spreading out over the sample and it looks something like this. Now, what happens when alpha is increased? So, when alpha is increased, so there is a rotation of 45 degree in this direction.

So, when it is rotated by this direction means by the Left Hand Thumb Rule ok, what will happen that the beam when theta is at 90 degree, the beam which was initially circular, it will become elongated vertically and when the alpha will increase to 70 degree, it will further elongate right. When both theta and alpha is increasing, you will see the elongation occurring at a diagonally and it keeps on increasing and once that as the sample is rotated by alpha this, this is the sample at alpha equal to 0. When the sample is in such a way that it is in the most focused condition as the incident beam is diffracting and it is falling on the surface and then, it is getting focused here fully.

As the alpha is increased from 0 to 10, minus 10 and then 30 and 45 whatever, then what happens that apart from this line of the sample, the plane this plane on that side and the plane this side of the sample becomes both defocused because the sample is now rotated by a certain angle by alpha.

So, the sample is face out of the focusing plane; that means, that except these line which is getting focused here on the detector neither the plane which is on that side of the line and this and towards my side of the line is getting focused properly at the detector. So, two thing is happening; first the focusing is gone, second the beam is highly spread out. So, it is highly elongated and distorted.

So, two things that has to be considered; first is the penetration depth. The penetration depth will have will change. Second, not all diffracted beam, now the diffracted beam is highly spread, so not all diffracted beam will now enter into the detector and neither it is neither it will entered in fully focused form right. So, as the beam has a large spread while after the diffraction, so all the recorded intensity may decrease, because the receiving slit may have a constant diameter right. So, it takes only a part of the incident beam sorry the diffracted beam because now the diffracted beams radius is much larger. So, the recorded intensity will reduce.

Secondly, when theta becomes very small is less than 10 degree, it cannot be measured because the spread here has become so large when as alpha will start to increase, the spread will be larger and larger. So, now, we comes to the penetration depth. Now, that in case of reflection geometry the problem of absorption is not that much. The spread increases, the absorption increases; but it is balanced by the increase in the irradiation volume. So, penetration depth will have no issue in case of the reflection geometry, but it will come it will become an issue, when it is a case of transmission geometry that is for example, in synchrotron radiation or in neutron diffraction.

So, the beam becomes totally defocused, when the tilt angle alpha becomes greater than 70 degree. The most important thing to remember is that at large tilt angle, all diffracted beam does not enter the detector because the receiving slit has a certain dimension right and therefore, it is only due to the geometric reason, this defocusing error basically occurs.

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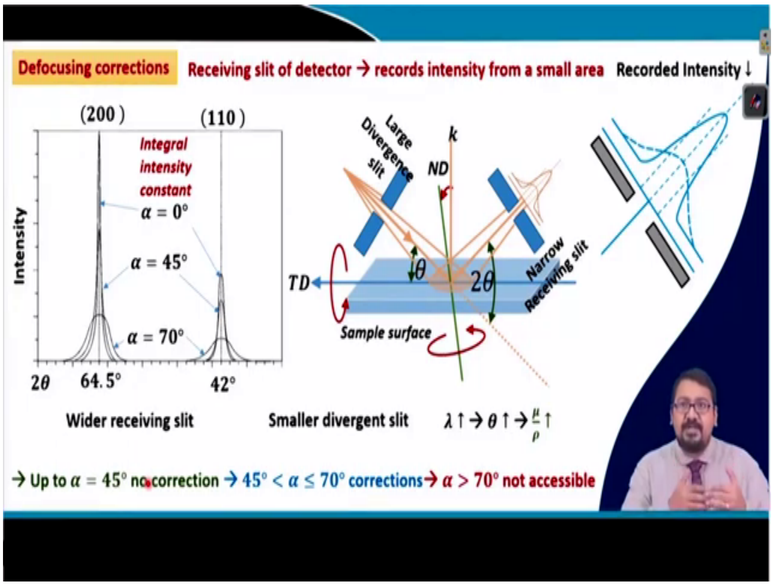

So, now, we will look into defocusing error correction. that when we do an alpha beta scan to obtain a pole figure for a certain theta. Say for example, for either an hkl 2 0 0 or for an hkl 1 1 0. Say for example, this is for a certain body centered cubic material which diffracts at a 2 theta of 64.5 degree for 2 0 0 hkl and 42 degree for 1 1 0 and say that we assume that in both the case, the intensity or the texture of 2 0 0 and 1 1 1 or sorry 1 1 0 is almost the same.

Now, let us look into this figure. that in order to obtain a better intensity, a optimum result from the diffraction; the X-ray beam which is basically divergent, we use a large divergent slit so that larger and larger beams enter and falls on the specimen. On the other hand, in order to reduce the background error, we use a very narrow receiving slit.

But as I said, in the last slide that when the theta is high and the alpha is low, the peak is sharp right. You can see it here, the peak is sharp and you can see that for a higher theta, the peak is sharp for a particular alpha say alpha equal to 0 and for a lower theta, the peak is basically it has a less peak considering the same intensity.

But the total area of this peak for both the case will be same, if the intensity of the texture is same for the hkl plane 200 and 110 and this is a hypothetical situation that we are talking about. Now, when the alpha is basically increased or the theta is basically reduced.

Say for example, in this case, let us keep the theta same and let us increase the alpha, what happens that as for example, in this case, when the alpha is 0 for hkl equal to 200 and for 2 theta equal to 64.5, the intensity peak is very high, the integral breadth is very low. At alpha equal to 45, the intensity peak reduces a lot and the integral breadth increases.

Now, the area under this curve, both this curve remains the same because it is for the same peak and when alpha increases to 70 degree, the integral breadth increases to a much higher extent; whereas, the peak decreases much largely.

Now, what happens that because we use a narrow receiving slit as the integral breadth of the intensity peak increases with the increase in the alpha, the narrow receiving slit restricts the entry of the total peak in order to remove the background and therefore, the recorded intensity is from a very small area and thus, the recorded intensity basically decreases.

The same thing happens for the different theta for another h k l plane. So, that the in order to carry out defocusing correction, what we need to do is that we need to use a wider receiving slit; a wider receiving slit will help us to take the whole intensity, while the peak has been broadened. A smaller divergent slit, a smaller divergent slit will help us to obtain a smaller divergent incident beam.

And thereby, the receiving slit or the detector will the intensity will not reduce that much when the narrow receiving slit is used in the diffraction diffracted beam and therefore, almost all the intensity will enter the detector. But a wider receiving slit and a smaller divergent beam both will increase the background.

So, one can think like we can increase the value of lambda, the wavelength. So, we can use a different instead of a particular characteristic wavelength, one can use a wavelength which is of higher lambda right, X-ray beam often higher lambda so that one can increase the Bragg's angle theta and thereby, one can get a reduced defocusing error.

But it will also increase the absorption mass absorption coefficient. Of course, it will not affect in case of the reflection geometry and it will affect mostly, in case of transmission geometry. So, up to alpha equal to 45 degrees, there is in most cases there is no correction needed.

From alpha equal to 45 to 70 degrees, one can do a correction and then, one can obtain the corrected curve or the correct corrected intensity removing both defocusing and the background. For alpha greater than 70 degrees the defocusing is so much that further correction is not possible.

Therefore, while we do the pole figure measurement, alpha beta measurement, the alpha is measured, the pole figure is measured up to alpha equal to 70 degree and not beyond that.

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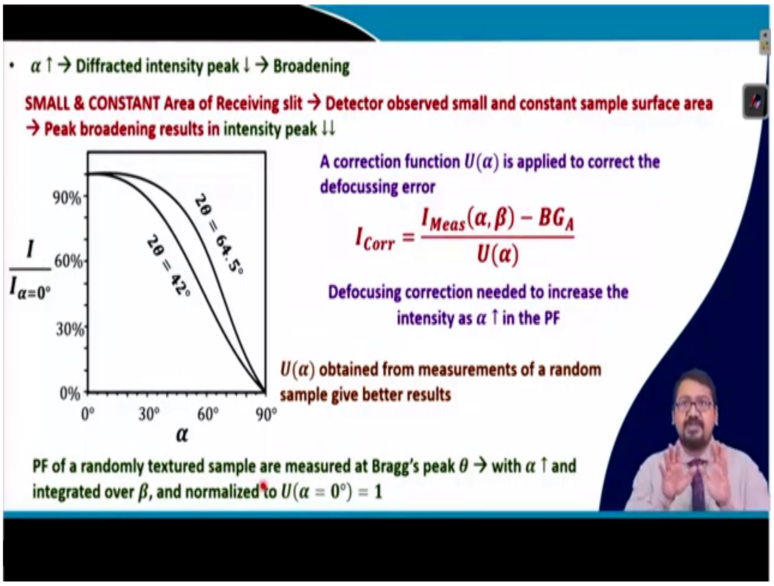

So, if we look closely as alpha increases, the diffraction peak basically intensity diffraction peak intensity basically decreases and this accompanies with the broadening. So, us as I said, in the last slide that a small and a constant area of a receiving slit, the detector will only observe a small and a constant sample area and the because of the peak broad broadening, the intensity that will enter the detector because of this narrow slit will keep on decreasing and therefore, what will have; what we will have? We will have a decrease in the intensity overall intensity.

So, if we look into the intensity with respect to the change in alpha and it is normalized by the intensity at alpha equal to 0, then we find that for different angles means the different Bragg's angles, 2 theta angles, the change in the intensity curve is not that different. Yes, there is a slight difference. So, what it is done? A correction function is U alpha; say U alpha is a correction function is applied to correct this defocusing error.

So, the intensity corrected is equal to the intensity measured minus the background correction divided by U alpha which is the defocusing correction right. So, the defocusing correction is needed to increase the intensity as alpha is increasing in the pole figure right. U alpha is obtained by the measurement of means it is best when you calculate the mu U alpha from the random sample for an alpha beta measurement right.

So, pole figure of a randomly textured sample are measured at Bragg's angle theta as alpha is increasing and it can be integrated over the whole beta because it does not change for the beta. So, we do not have to measure from beta equal to 0 to 360 degrees and then, it has to be normalized with respect to U alpha for alpha equal to 0 degrees and it has to be made equal to 1.

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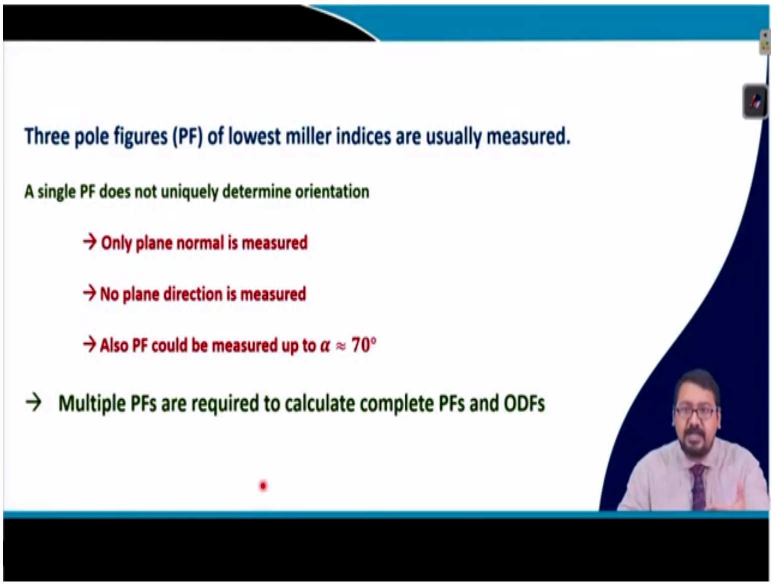

So, that for measuring the pole figure, what we found out from the discussion? We found out that there has to be three pole figures of lowest miller indices that is usually measured that has to be measured and because one pole figure will not give the total information.

The first reason is that a single pole figure only gives the plane normal and it does not give the direction of the plane. That important direction of the plane. So, the plane normal we only have. So, the plane can be rotated right on the plane the particular direction at which a certain important reference sample reference direction is there is not notified for by a single pole figure It only gives the normal to the plane.

The second thing is the pole figure could only measure up to alpha equal to 70 degree because it will get fully defocused after 70 degree. So, we have to measure multiple pole figures. All from 0 to 70 degrees and then, we have to take that data all this at least these three pole figure datas from alpha equal to 0 to 70 degree and only the data which contains the normal pole of the particular plane, hkl plane or three different h k l planes that are measured right.

And from that, the back calculation is done and the total pole figure or the calculated pole figure is obtained which is from alpha equal to 0 to 90 degrees for all the cases and from these the inverse pole figures, the important miller indices, the orientation matrix, the Euler angles or the Euler space or the orientation distribution functions could be measured or obtained ok; actually calculated.

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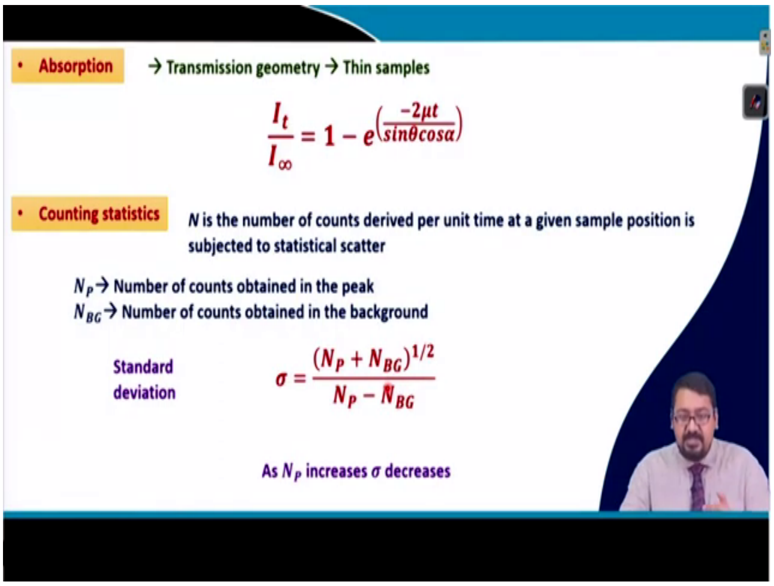

So, as I said that absorption is not very important factor in case of reflection geometry in X-ray diffraction for measuring the pole figure. But in case of transmission geometry, absorption is a very important factor. Say because when the in the transmission geometry, we use thin samples and it is the electromagnetic radiation, X-ray radiation passes through the sample and gives the diffraction in the transmission mode definitely.

So, if the intensity of the applied radiation, intensity of a sample of a certain thickness t is I t and the intensity that can obtain for an infinite thick sample is this. Then, this is given by 1 minus exponential to the power exponential to the power 2 mu t; mu is the linear absorption coefficient, t is the thickness, sin theta is the theta is the Bragg's peak and for a particular alpha cos alpha right. So, this is basically the absorption intensity that we need to know.

Now, another important thing is the counting statistics. that the larger the counting or the larger the statistics for the certain peak, then the standard deviation of the peak basically reduces. So, if ; if that N is the number of counts; N is the number of counts derived for unit time at a given sample position and this is subjected to a statistical scatter.

Now, if N P is the number of counts at the certain Bragg's peak and N BG is the number of counts for the background. Now, the standard deviation sigma is basically given by N P plus N BG to the power half that is square root of N P plus N BG by N P minus N BG. Now, here that as the N P increases, the sigma that is the standard deviation decreases.

So, counting statistics are very important. So, once we could correct all these errors or the required corrections could be obtained, then, the samples intensity, the texture intensity of the sample is basically normalized to multiples of random orientation distribution that is MRD.

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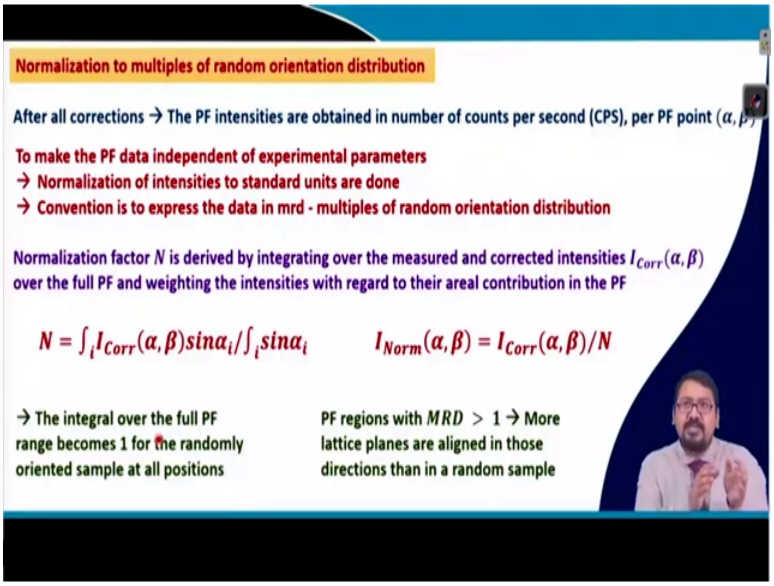

So, normalization to MRD is essential in order to remove the effect of experimental error or experimental the effect of experiment or sample size or experiment size from the sample. Now, how we do it? So, as I said after all the collections, the pole figure intensities are obtained in terms of number of counts per second that is CPS, per pole figure points that is per pole figure alpha beta points.

So, what we do? To make the pole figure data independent of any experimental parameters as I was saying. Normalization of intensity has to be done to get a standard unit right. So, the convention is to express that in multiples of random orientation distribution which is known as MRD; multiples of random orientation distribution.

So, the normalization factor basically N is given by integration of the intensity of corrected alpha beta times sin alpha by the integration of sin alpha right. So, it is basically the integration over the measured and corrected intensity over the full pole figure and the weight the weighting of the intensities with regard to their areal contribution in the pole figure.

So, the intensity after normalization is equal to intensity character divided by this factor N. So, this normalization factor is such a factor that for the randomly oriented sample, the I normal will become equal to 1 right. For pole figures with certain texture, MRD will become greater than 1, for those positions of for which a particular hkl l plane has is aligned more than that of the random sample ok.

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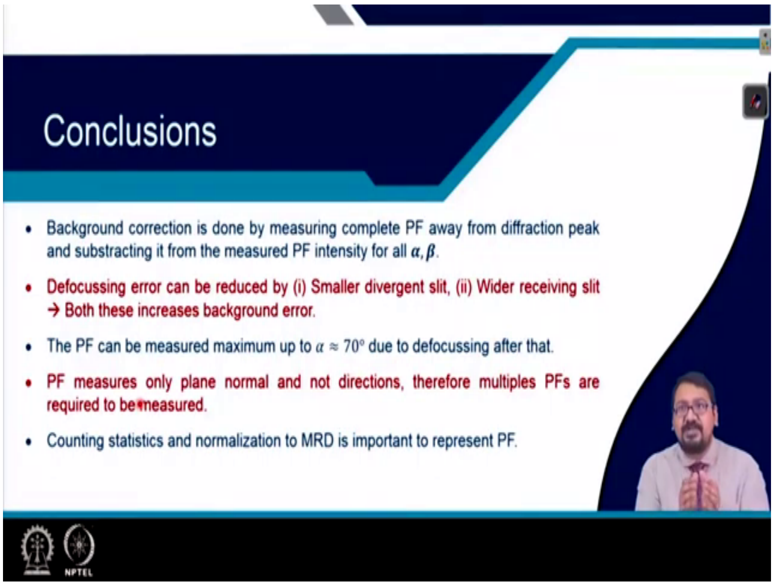

So, finally, the conclusions. Background correction is done to measure by measuring complete pole figure away from the diffraction peak and subtracting it from the measured pole figure intensity for all alpha and beta. Defocusing error can be reduced by using a smaller divergent slit and a wider receiving slit, but it will increase the background error. The pole figure can be measured up to alpha equal to or less than 70 degrees due to large defocusing after that angle.

The pole figure measures only plane normal and it does not measure even the direction and therefore, multiple pole figures are required to measure the whole texture. Also, the reason is which can measure up to alpha equal to 70 degrees. The counting statistics and normalization to MRD is important to relieve the information of the pole figure from the experimental parameters and to represent it independently in terms of MRD in the pole figure.

Thank you very much.