Texture in Materials
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Module - 06
Microtexture measurements using EBSD technique in SEM
Lecture - 33
Quantitative Evaluation of Kikuchi Diffraction Pattern - II

Good afternoon everyone. And today we will be continuing with the module 6 which is Microtexture measurements using EBSD techniques in SEM; that is scanning electron microscope. So, today is lecture number 33 and we will continue with understanding the quantitative evaluation of Kikuchi diffraction pattern and this is part 2.

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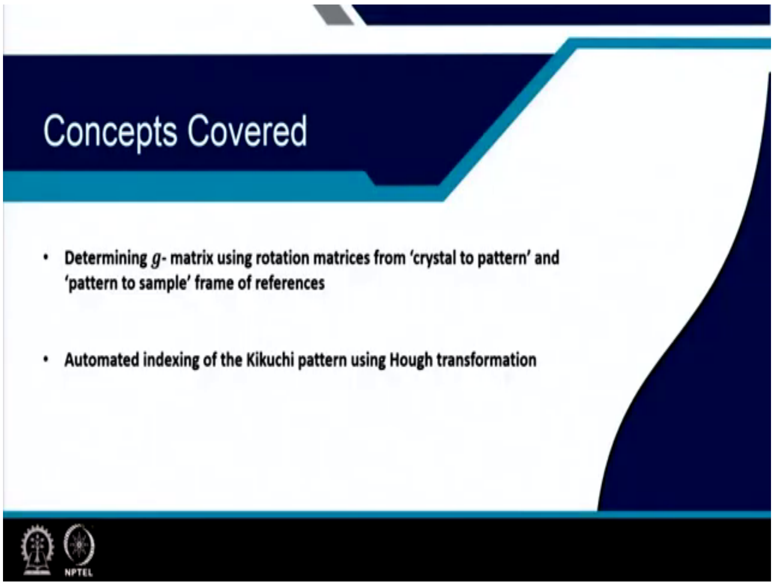

The concepts that will be covered in this lecture are; determining g matrix using rotation matrices from crystal to pattern and pattern to sample reference frame. And actually, we have done this in the previous lecture, but let us summarize this and try to understand what we left there right. And the second thing is automated indexing of the Kikuchi pattern using the Hough transformation.

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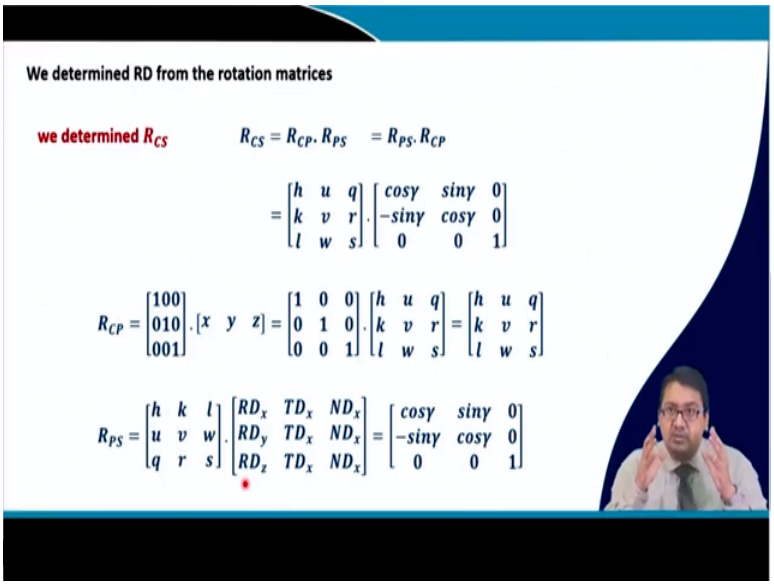

So, you see that in the last lecture class, we determined R D; that is the rolling direction; that is one of the important sample reference direction between R D T D and N D for example, for a rolled specimen right. So, the R D that we determined is in terms of the Miller indices. So, we determined this R D from the rotation matrices right. So, while we determined the R D, we determined R CS. That is the rotation matrices from the crystal frame of reference to the sample frame of reference.

So, R the subscript C means the crystal frame of reference and the subscript S means the sample frame of reference right. And when we did it, we found out that R CS is equal to R CP dot product R PS. Now what is R CP? R CP is the rotation matrix between the crystal frame of reference to the you know pattern frame of reference.

And we saw that if we know a particular Kikuchi band in a Kikuchi pattern. Let us identify a particular Kikuchi band in the Kikuchi pattern and then perpendicular to it, the vector perpendicular to it say it is x has a certain you know Miller indices h k l and a vector y which is perpendicular to x; that means, it is parallel to the Kikuchi band.

And then let us say that its Miller indices is u v and w. And the Miller indices of z which is basically the beam normal, the direction at which the incident beam is falling; that is N D and let us say it is q r s. As we know the value of x and z which is h k l and q r s, we can find out the value of u v and w and we saw that, right.

So, if we look what is R CP? Then R CP can be written in terms of a matrix which contains h k l representing the x axis of the pattern frame of reference, u v w representing the y axis of the pattern frame of reference and q r s representing the z which is ND for the pattern frame of reference right. And it is dot product with R PS.

That means, the rotation matrix from pattern frame of reference to the sample frame of reference. So, as we have said in the last lecture that the sample has to be put in the TEM or in the SEM EBSD in such a way that the RD and the TD of the sample is known and is determinable by the software right.

Now, you see the rotation matrix between the pattern frame of reference and the sample frame of reference is the rotation between this x y and z representing h k l u v w q r s with respect to R D T D and N D which will have its own Miller indices.

We found out that how this rotation matrices can be calculated if we know the angular relationship between x and R D right. Say it is gamma like we said in the last lecture. So, R PS becomes equal to cos of gamma sin of gamma 0 minus sin of gamma cos of gamma 0 0 0 1. So, R CS can be also written in terms of R P S dot product R CP.

Now what is basically R CP, let us look into it in a little detail though we have seen it in the last lecture. R C P is basically the dot product between the crystal frame of reference which is what is the crystal frame of reference; say for example, for a cubic crystal. So, the x axis is 1 0 0, the y axis is 0 1 0, the z axis is 0 0 1 right. So, 1 0 0, 0 1 0, 0 0 1 are the three axes of important orthogonal axis of the crystal, right.

So, in case of cubic crystal it will be like this. Dot product with the pattern frame of reference which is x y and z. And what is x? As I said, x is h k l y is u v w and z is q r s. Now dot product between this identity matrix which is basically the you know important crystal reference frame with respect to the pattern reference frame becomes easy if you do it; it again become h k l u v w q r s. So, this value is basically the rotation matrices of between the crystal frame of reference to the sample; to the pattern frame of reference.

Now, on the other hand as I said that the rotation matrix between the pattern frame of reference and the sample frame of reference is a should be again a dot product between the pattern frame of reference which is you know this one h k l and u v w q r s dot product with respect to the Miller indices of the R D plane that is R D x, R D y, R D z.

The Miller indices of the T D plane that is T D x, T D y, T D z. Just remember that these are typographical error it should be y and z N D x, N D y, N D z right.

And we have calculated in the last lecture that if the angle between the x which is h k l and R D which is R D x, R D y, R D z is gamma, then the dot product between these two can be written in terms of cos of gamma sin of gamma 0 minus sin of gamma cos of gamma 0 0 0 1. This becomes the matrix. That is the rotation matrix for the from the crystal frame of reference, sorry from the pattern frame of reference to the sample frame of reference.

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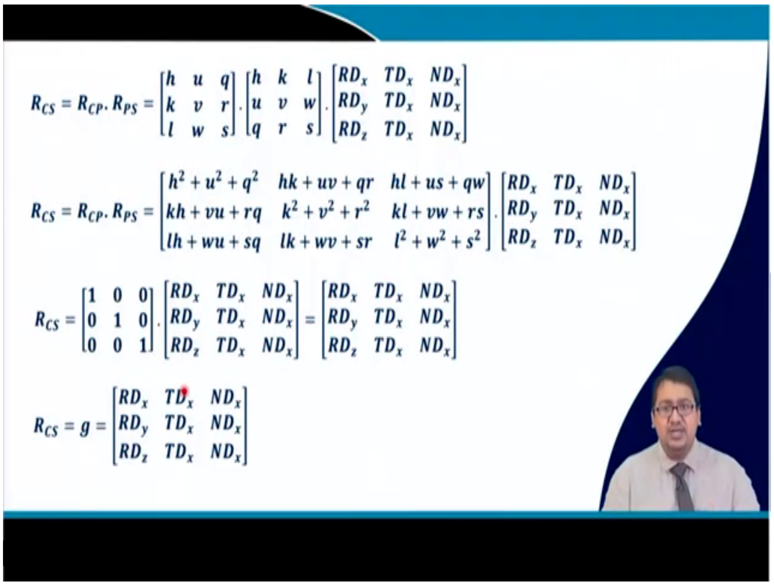

Now, if we look into this equation, we can find out that what is R CS and R CS is the dot product between R CP and R PS right. So, if we write R CP; R CP is h k l u v w q r s and dot product with respect to R PS which is h k l u v w q r s dot product with R D x R D y R D z, T D x T D y T D z, N D x N D y N D z this equation right. Now if we you know do this dot product between h k l u v w q r s h u q k v r l w s then what will happen?

Then we will get this kind of a value right this kind of a matrices. Now you see h square plus u square plus q square becomes the first you know what you call the 1 1, and then h k plus u v plus q r becomes 1 2. Then h l plus u s plus q w becomes you know 1 3. Like that you can find we can find out all the values all the 9 variables for this matrices; the dot product between this and this.

Now you will find to your surprise that even under this condition the you know the dot product between these two will be an identity matrix. That means, this, this and this will become equal to 1. Like the same if the dot product of h square plus k square plus l square is equal to 1. The h square plus u square plus q square k square plus v square plus r square and l square plus w square plus s square also becomes equal to 1.

Now, you remember that when we are talking about h k l u e w and q r s, remember that all this you know Miller indices are basically you know h when we are talking about is basically normalized to 1. That means, h is here is equal to h divided by root over h square plus k square plus l square; u is basically u divided by u square plus k u square plus v square plus w square. right. So, everything is normalized by 1 right.

So, when everything is normalized by 1 then this quadrant 1 1, 2 2 and 3 3 becomes 1, and these quadrants 1 2 1 3 2 1 2 3 3 1 3 2 all becomes equal to 0. So, when we are talking about the rotation matrix between the crystal to the sample frame of reference, then this first product becomes equal to a identity matrix which is 1 1 1 0 0 0 0 0 1.

And I hope that the students may take the example that I showed in the last lecture and use those values of h k l u v w to see whether really this thing is happening or not ok. And if not, kindly let me know in the you know in commenting by emailing me kindly let me know ok. Or you may also let me know during the live session that will occur on 28th of August.

Now you see that R C S is equal to then the identity matrix this dot of R D x R D y R D z T D x T D y T D z N D x N D y N D z, that indicates that R C S is basically equal to this value right. Because if you have a dot product with the identity matrix of certain matrix, the certain matrix that matrix remains the same.

So, you see that R C S basically gives the value of you know the orientation matrix g which is given by you know R D x R D y R D z, T D x T D y T D z N D x N D y N D z. Now that means, that as we already know the value of N D which is q r s. That is for example, in the last class it was last lecture we said we found out that it was 5 6 7 right. And by this way we also have found out in the last class that how we have calculated the value of R D in case of; in terms of Miller indices and also T D in terms of Miller indices.

So, the rotation matrix between the crystal to the sample frame of reference as usual as taught earlier is the g matrix. And this g matrix can be calculated very simply using this kind of matrix calculation, right.

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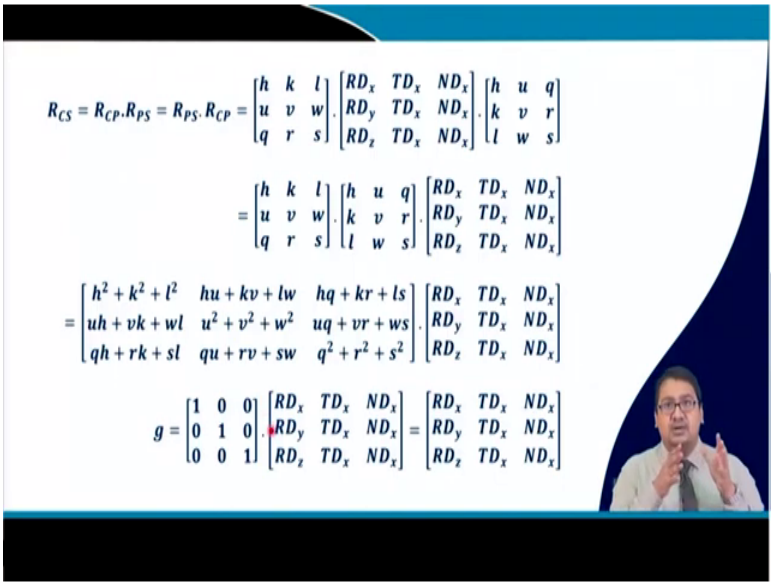

Now, as I said that you see R C S can be written also in terms of you see R PS dot R CP rather than R CP dot R P S. Now in this case what will happen then this R P S which corresponds to h k l u v w q r s R D x T D x N D x R D y T D y N D y R D z T D z N D z is dot product with R C P which is this value right h k l u v w r q r s.

Now if this happens, we can write the same equation like this. And I am telling you this because if there is a confusion because of the you know dot product between these two matrix is coming out to be a little different than what we actually perceive. Now, if we do the dot product here then it comes out to be you see h square plus k square plus l square, u u square plus v square plus w square, q square plus r square s square for the 1 1 1 2 2 and 3 3 and for 1 2 1 3 2 1 2 3 3 1 3 2 it comes as a mix right h u plus k v plus l w and so and so right.

Now as I said earlier that all h k l u v w q r s are basically normalized to 1. That means, that h is basically h divided by h square plus q square plus k square plus l square right; u is basically u divided by a basically equal to u divided by u square root over u square plus k square plus l square. Sorry u square plus v square plus w square right.

Now, so, it is normalized by 1. So, if we calculate this also you will find out that the 1 1 2 2 and 3 3 of this you know 3 cross 3 matrix becomes equal to 1 and the rest will become equal to 0. So, the matrices again will give the value of g, the same it comes to the same calculation basically. So, this becomes an identity matrix as I showed earlier and then this becomes again the values related to R D T D and N D.

Thus the rotation matrix between the crystal and the sample frame of reference gives the value of orientation matrix g. Thereby it gives the value of you know the Miller indices for R D T D, and definitely the ND which was which is basically priory calculated using you know three Kikuchi bands. And you have seen the calculation that how it is used to calculate the zone axis and from the zone axis the angle between the zone axis and the N D is used to calculate the value of N D and then R D was calculated.

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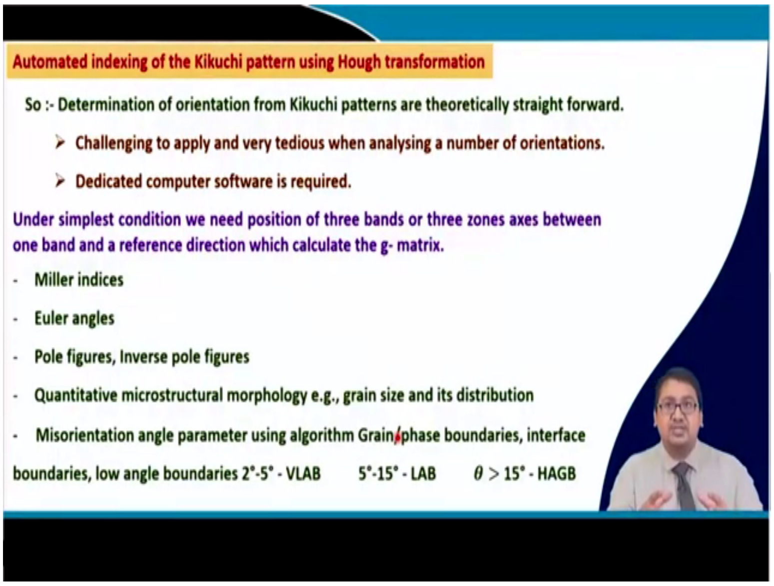

So, you see you know that determination of orientation from Kikuchi pattern is you know theoretically very straightforward. So, its mathematically possible and it is there is no issue if there is a Kikuchi pattern is given and one can just calculate it using a piece of pen and paper, right.

So, the problem is that in an EBSD scan what happens that the electron beam basically falls on the sample at a very small area, that area is in nanometer scale, and then there is an interaction volume. And if it is tem then you know convergent beam diffraction takes place and Kossel cone forms and then that Kossel forms it falls on the phosphor screen. On the other hand, in EBSD there is a 70-degree tilt and again the because of you know multiplely scattered electron seems to come from inside the sample, and then you know it is finally, scattered and reinforced scattering because of the periodic nature of the you know crystal structure. It gives a Kossel cone at an following the Braggs law again forming you know Kikuchi pattern in a phosphorous screen which is now vertically kept ok, away from the you know incident beam direction.

Now in both the cases you see you get the Kikuchi pattern and calculation of the Kikuchi pattern is so easy. But, it is very important to remember that this is a single scan on a poly crystalline material where maybe a 100-1000 scans will like that 100-1000 Kikuchi patterns like that will be obtained right.

So, you see basically what happens that the electron beam falls and there is an interaction volume and then we get the Kikuchi pattern and the beam basically shifts and falls on the side of it. And then again it shifts it falls another at another point and like that. It scans the whole sample one-by-one, one-by-one and rest of the whole sampled area. Like that to obtain a particular microstructure in an EBSD or even in a convergent beam electron diffraction in a TEM a full rastering of say 100-1000 points; 100-1000 Kikuchi patterns are obtained.

So, it is very challenging to apply this calculation directly to you know for this 100s 1000 Kikuchi pattern. So, because now; therefore, in order to do so dedicated computer software's are required. Now under simplest condition we just need basically three bands right and the which can calculate three zone axis's and between two bands right and thereby we can calculate the reference matrices and g matrices. And from the g matrix one can calculate the Miller indices of RD TD and ND.

Then we can calculate the Euler angles phi 1 phi phi 2. Then we can calculate the pole figures right any pole figure any h k l pole figure as needed. And from that we can also; from the you know Euler angles we can also find out the inverse pole figure. And also, you see we can do complex calculation and find out you know any important information about the microstructural morphology like the grain size or its distribution right or we can find out that misorientation angle parameter using some you know little complex algorithm.

And now you see grain phase boundaries, interface boundaries can be calculated. One can find out low angle boundaries like 5 to 15 degrees or very low angle boundaries like 2 to 5 degrees which are basically geometrically necessary boundaries. One can determine the you know fraction of or the length of the grain boundaries like high angle grain boundaries, like which are 15 degrees or more.

So, there are many things one can do in order to obtain you know information of quantitative microscopy using EBSD and this Kikuchi pattern technique. But yes, for this a dedicated computer software will be definitely required.

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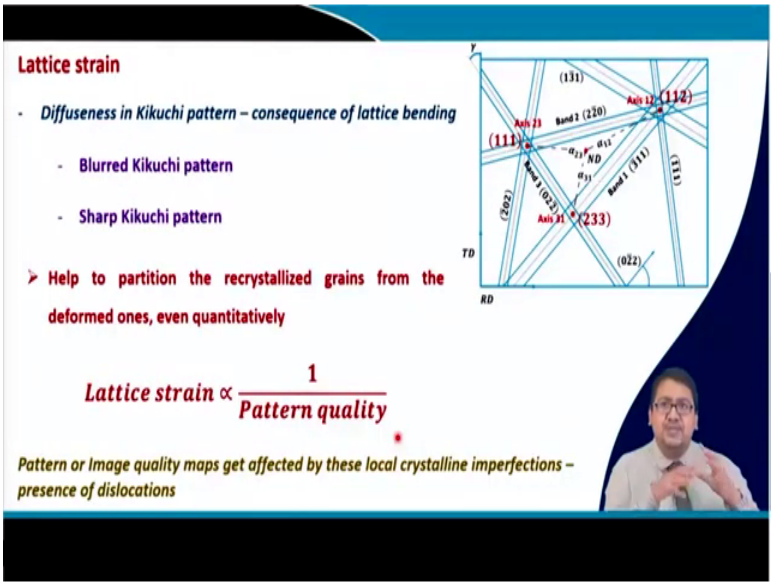

So, other than this you see there are you know not only there are softwares. There are many softwares here like for example, I will say that the one software that I miss the three softwares that we usually use, is first is that you know the Atex software that is you know it is a freeware and is developed by two of my very close friends. They are you know Jean Jacques Fundenberger and Benoit Beausir. Both of them are professors in University of Florence Metz France.

And you can you know just type their name and download Atex from there where we can do you know analysis of these Kikuchi bands, Kikuchi pattern and these quantitative microscopy microstructure obtained from EBSD TEM and even using XRD.

The second software that we usually use is the TSL OIM software which is basically made by you know edX Mi Tech Company. The third is the HKL software which is made by you know Oxford instruments. And all these softwares are equally beneficial and they will have you know their own some you know pros and cons in it, but we use all of them separately and to find out information about these quantitative micro structures, right.

So, there are development in the you know understanding of this Kikuchi band throughout this years. And later it was found that in the Kikuchi bands can be you know very sharp and sometimes very blurred. And it was understand that it depends on the strain of the sample the Kikuchi bands are sharp or you know blur.

Now the blurring or the diffusiveness of this Kikuchi pattern is definitely because of the lattice strain which can be considered like a bending in the lattice right, which is you know making the pattern diffused.

Now it is the same thing like you see the as we have said that in XRD when there is a lattice strain present in the sample, and this lattice strain is you know not is a heterogeneous strain then you see there is a peak broadening right. And the sharpness of the peak basically decreases, it is the same thing right. In case of electron backscatter diffraction or in case of convergent beam electron diffraction.

So, this information of the Kikuchi band can be you know correlated using image correlation softwares. And then others and the softwares can be developed, mathematical formulations can be developed which can lead to you know separate out regions of the microstructure, which are highly diffused or you know sharp into regions, like you know recrystallized part and the deform part.

For example, if we take a material which we have done say for example, of large plastic deformation at relatively warm temperatures. So, certain parts. So, there is you know deformation and then there is you know recovery and recrystallization. And there are certain parts in the microstructure which is recovered and recrystallized and there are certain part of the microstructures which are severely deformed.

So, the severely deformed part of the microstructure will be having a diffused Kikuchi pattern right. And the you know the recrystallized part of the microstructure will be having a sharp pattern. So, you see the pattern quality of the map can decide that whether that part of the microstructure is highly strained or it is in a relaxed condition, a recrystallized condition or not.

So, one can use this technique to partition the microstructure into deformed part and recrystallized part. So, lattice strain is basically proportional to you know inversely proportional; sorry to the pattern quality. And thereby the pattern or image quality may get affected by you know the local crystalline imperfections like you know the presence of dislocation structure, because of because of whatever I discussed in this slide, right.

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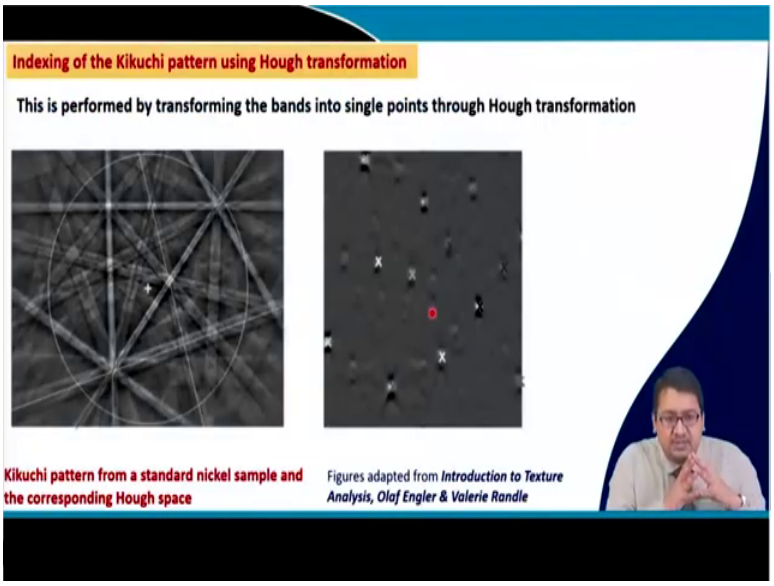

So, if we look into the indexing of the Kikuchi pattern, the softwares uses you know Hough transformation technique to index them right. So, indexing of this Kikuchi patterns, using this Hough transformation technique basically involved in transforming these bands. You see in this Kikuchi pattern there are so many bands right.

So, it is basically related to transforming each and every of these bands which are basically brighter than the rest of the you know areas of this pattern right into single point points. So, each band is transformed into a single point in the Hough space and this process is known as Hough transformation.

You see I have given two figures one is the Kikuchi pattern and this Kikuchi pattern is for the standard nickel sample which has been taken from the book of you know Olaf Engler and Valerie Randle; that is Introduction to Texture Analysis. And each of these Kikuchi band ok could be transformed into points here as shown. So, this is the Hough space and this is the Kikuchi pattern space right, so into you see spots. So, each spot represented here represents a band in the Kikuchi pattern. Now, let us look that how mathematically a band can be converted into point using Hough transformation.

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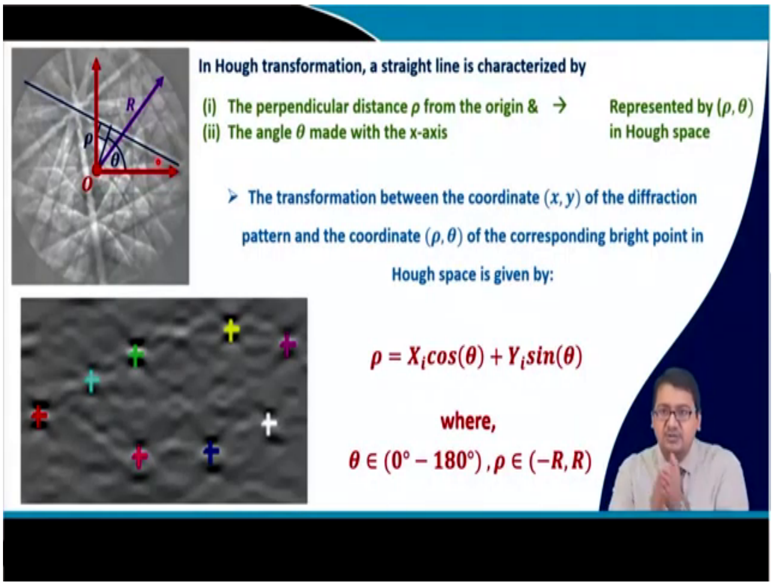

First of all the band is basically taken, captured and then as you saw it is a rectangular you know image and from which a rounded part of the image is considered. And this is very important so that the image correlation technique becomes proper. Now if this is the center of this you know rounded you know image of the pattern, then the and say for example, this is R D and this is T D. As I said that this has to be known.

And then if this is the radius of this pattern, so this is the radius R R from the center of this rounded circular image taken. And then say for example, if we consider this band this Kikuchi band and we can consider either both the bands right, both the lines of the band, the lower line or the upper line or we can consider the center of the band, the average of this band right. So, let us for easy of our calculation, let us calculate the average of the band say this one, right.

Now, this is a line corresponding to the Kikuchi pattern image right. Now how it will be transformed using Hough transformation technique in Hough space in form of a point. So, let us draw a line, this line which is perpendicular to this band right perpendicular right.

So, if from origin which is determined to be the center of this space, the distance perpendicular distance from the origin to the Kikuchi band or the average Kikuchi line is rho and the angle of this you know perpendicular line or vector is theta, then this Kikuchi line can be you know shown in Hough space in terms of you know a point which is at a distance rho and theta from the origin right.

So, you see that say for example, with respect to this Kikuchi band this is the point that is obtained. So, this point is such a way that it is at rho and theta with respect to the origin of the Hough space right. So, in this way all the Kikuchi bands are calculated, each one will have a certain rho and theta and thereby the you know Kikuchi pattern space is transferred into Hough space.

So, the Hough transformation is a process, is a mathematical process in which a straight line can be transformed into a Hough space with respect to the perpendicular distance rho from the origin and the angle theta with respect to the x axis right. Sorry the x axis that is R D right. The space that forms in form of rho and theta rho as an x and theta as in the y coordinate is known as the Hough space right.

So, the transformation between the coordinates x y of the diffraction pattern space or the Kikuchi pattern space and the coordinate rho theta corresponding to the Hough space, basically corresponds to the you know bright line transforming into bright point in the Hough space when done using image correlation technique.

Now, therefore, what is rho? Rho comes out to be equal to X i cos theta plus Y i sin theta and i is the you know indication of that particular Kikuchi band, the i-th Kikuchi band, where theta can be between 0 to 180 degrees and R or rho can be between minus R to plus R.

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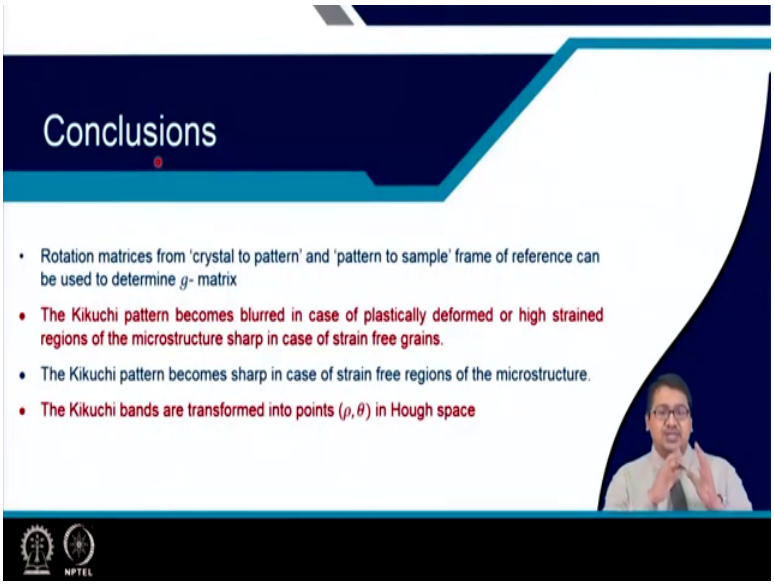

So, what we can conclude from this lecture class? We can conclude that the rotation matrix from the crystal to the pattern frame of reference and pattern to the sample frame of reference; can be determined using, can be determined can be used to determine the g matrix right. Second, the Kikuchi pattern can become blurred in case of plastically deformed or highly strained sample areas; that is regions of the microstructure, and it can become sharp in regions which are strain free or recrystallized right.

The Kikuchi pattern; yes, becomes sharp when it is becomes recrystallized right in strain free regions of the microstructure. The Kikuchi band are transformed into this lines are transformed into points of Hough space, which is a space made up of rho and theta and we explained what is rho and theta in this lecture, right.

Thank you very much for this class.