Texture in Materials
Prof. Somjeet Biswas
Department of Metallurgical and Materials Engineering
Indian Institute of Technology, Kharagpur

Module - 06
Microtexture measurements using EBSD technique in SEM
Lecture - 31
Kikuchi Diffraction Pattern - II

Good afternoon everyone and we will continue today with the module 6 which is Microtexture Measurement using EBSD technique in SEM. And, in this class we will be continue discussing about Kikuchi diffraction pattern and this is part 2.

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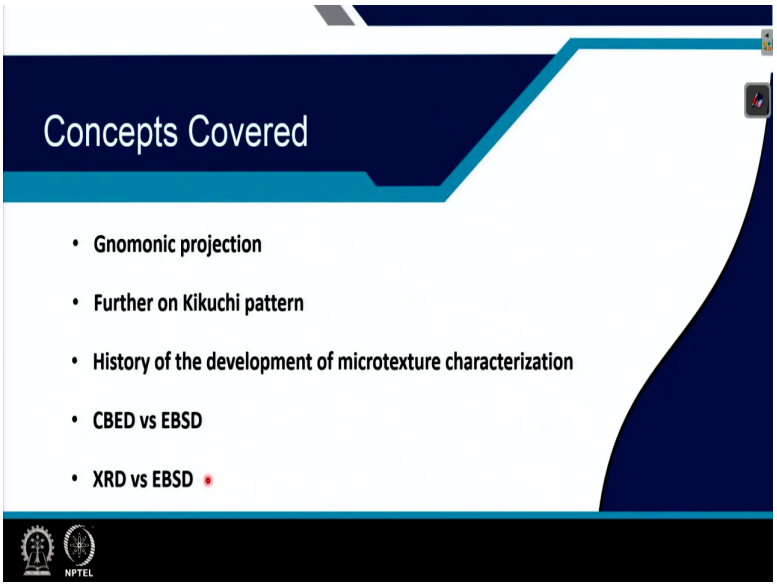

So, the concepts that will be covered in this lecture are gnomonic projection. Second, we will try to give further information regarding the Kikuchi pattern diffraction. So, little bit of brief about the history of the development of the microtexture analysis and characterization.

We will compare convergent beam electron diffraction with respect to electron backscattered diffraction in an SEM and we will compare the X-Ray diffraction technique with respect to the electron backscattered diffraction in an SEM.

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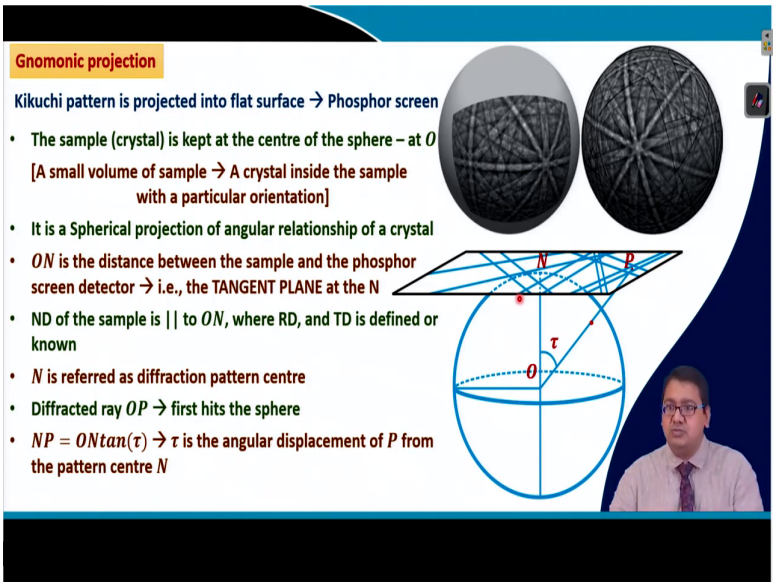

So you see, a Kikuchi pattern is basically projected on the flat surface; that means, the phosphor screen or the camera that on which the Kikuchi pattern is projected. Say for example, if a sample is inside then the Kikuchi pattern is projected on a flat phosphor screen something like this right.

But, the Kikuchi pattern is basically a gnomonic projection; that means, it has to be projected on a you see a sphere right and if we keep on projecting the sample by rotating the sample then one can get or if we can you know increase and you know calculate the whole Kikuchi pattern, it will form as kind of a sphere.

So, a sample which is kept at the centre of the sphere something like this is diffracted. Say for example, the electron gun is here and the electron beam is falling and a diffraction is coming out from a very small part of the sample or very small volume of the sample which is basically part where the sample is single crystal in a polycrystalline sample right. Even the that polycrystalline area which is basically rusted or covered is actually very small in say few micrometers right.

Now, if the sample is kept at the centre O and if N is the you know point where the incident beam is falling on the sample on the phosphor screen that is the centre of the you know projection pattern. Then any diffraction pattern which is forming at this position basically is should be forming at these this particular projection on the point on the sphere.

So, whatever we has to project in the sphere, we are basically you know projecting it on the flat surface. So, it is the Spherical projection on which the angular and it depends upon the angular relationship of that crystal ok.

So, if ON is the distance between the sample and the you know phosphor screen detector right so you see the phosphor screen detector is basically kept tangential at N where it is touching the sphere and it is tangential to the sphere right. And, if you see that if the electron beam is incidenting like this then the beam normal BN or ND is parallel to ON right, where for a sample which is kept here the RD and the TD has to be defined right or known.

So, you see for example, if this direction in this sample this direction is RD and this particular direction if it is kept TD, then the Kikuchi pattern that can be that has been obtained can be related to the you know sample reference system in order to get the information of the orientation.

Now, as I said earlier that N is basically the diffraction pattern centre. And, say for example, as I was mentioning earlier that if a diffraction from a particular hkl plane are two diffraction right, two Kikuchi lines can be obtained and if it is at an angle tau it should be forming on the sphere here, but it forms here on the phosphor screen.

So, the diffraction pattern that is N P. NP is basically related to ON, as ON equal to you know sorry NP equal to tan of ON tan of tau right. So, NP is equal to ON tan of tau. So, instead of because it is a gnomonic projection it has to be this arc which should be equal to ON times tau, tau should be in radian the angular relationship is now ON times tan of tau.

Now, so N so you see, in case of the electron beam the theta is so small and the lambda is the lambda is so small that the you know diffraction angles thetas are also so small that this tau basically is also very small. So the difference where NP becomes equal to ON times tan tau in most of the cases becomes equal to ON times tau ok.

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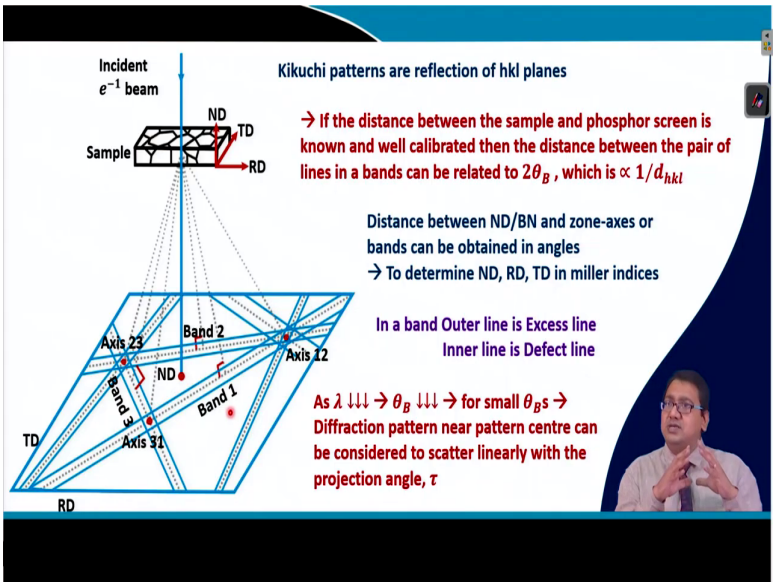

So, if we look further into how the Kikuchi patterns are developed for various hkl planes, we should look into this schematic. This is a schematic where the incident electron beam is falling on a polycrystalline material. And, you see as I said the incident electron beam say for example, is falling first at this point and then a Kikuchi pattern is obtained and then it falls in the next point and then another Kikuchi pattern is obtained and then another next point and then another next.

And like that if the whole sampled area is scanned, at a certain time when the Kikuchi pattern is falling sorry when the incident electron beam is falling on the sample somewhere here on this particular grain. Then, a certain Kikuchi band will be obtained. So the schematic of the Kikuchi band can be shown here.

And, by the if the distance of the sample with the phosphor screen is known and the and this is properly calibrated, then the thickness of the bands can be related to 2 theta B and which is related to the you know interplanar distance of the particular hkl plane.

So, you see the bands have different thickness therefore, they are of different 2 theta bs and therefore, they belong to different and specific hkl planes right. So once, the you know if this is band 1, this is band 2 and this is band 3 and then from this 2 theta B, if we can find out the you know the hkl plane or hkl value of miller indices of band 1, the miller indices of band 2 and the miller indices of band 3.

Then this is the first step where we can get we got this information and then what we can do that we can obtain the information of the miller indices of this zone axeses right. So, the axis 1 2, axis 2 3 and axis 3 1 we can get the information from. So, how we can get?

If we you know do a cross product of band 1 and band 2 we can get the miller indices of axis 1 2, if we do a cross product of you know band 2 and band 3 we can get a miller indices of axis 2 and 3 if we do a cross product of band 3 and band 1 we can get the miller indices of axis 3 1.

And, now as I said that if the distance between the sample and the phosphor screen is well calibrated, then the angle between this axis 1 2 and ND, axis 2 3 and ND and axis 3 1 and ND is basically known. And, if we know this angle as say for example, alpha 1 2, alpha 2 3, alpha 3 1 then you know by using you know a dot product between that.

So, the dot product between the beam normal, that is the direction of the incident electron beam and which is parallel to ND. Say for example, it is some u v w plane. So, one can obtain, so the relationship between this NDs miller indices dot product with the miller indices of this three axes will give you the value of cos of the angular difference between them and thereby we can obtain ND of the specimen.

Similarly, we can obtain RD and TD of the specimen by simpler mathematical calculations and that we will show you in the next you know lecture. So, this is the way by which for each Kikuchi pattern obtained out of the millions of Kikuchi patterns that are obtained for a certain sampled area the ND RD TD can be determined in terms of you know the miller indices.

So, yes of course, if we have the TEM convergent beam electron diffraction, we can get axes and defect line. If we do the same thing in EBSD where the calculations to calibrate the sample with the phosphor screen, the distance between that and the angular relationship makes the mathematics little bit complicated, but we can get the same information in that case also.

So, now as the lambda for the electron beam as I said earlier is so small that the theta b is are extremely small. So, even if we are projecting on a parallel phosphor screen, it is very those every diffraction pattern or bands that are forming are very near to the you know diffraction pattern centre right. So, we can most of the time can consider that the scatter is linearly with the projection angle that tau that we were trying to explain the last this slide.

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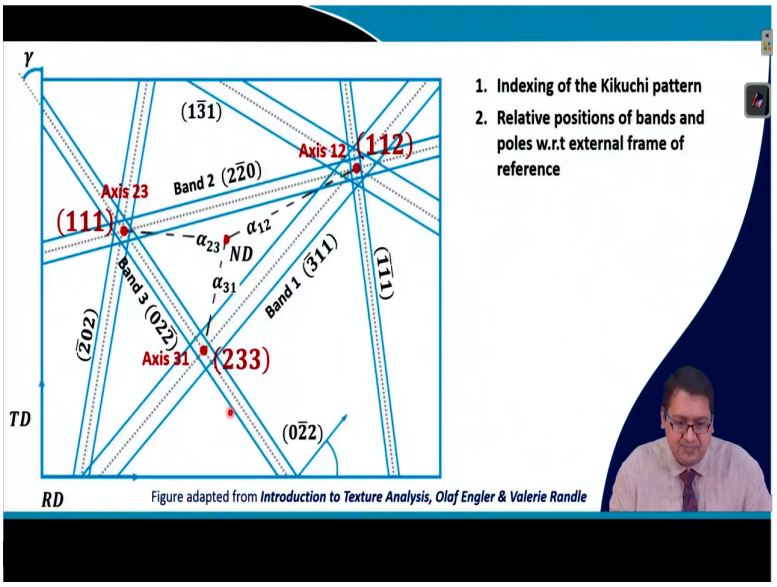

So, the typical band that formed like that may look something like this and this is an example that I will be using. And I will be using this example and it is given in you know introduction to texture analysis by Olaf Engler and Valerie Randle.

And I will be using the same example so that students can basically see this understand what is happening and then also can go to that book and read it to you know understand in more detail if required, right. So, that they use the same example because you know there should not be any deviation in the understanding it to make it easier for the students ok.

So, you see as I was saying that in this example we have this band 1 and band 2 and band 3. And we know we can find out 2 theta B if it is calibrated for each of these bands and from which if we can find out you know the h k l is 3 bar 1 1 and for the band 2 if it is 2 2 bar 0 and for the band 3 it is 0 2 2 bar then one can find out the zone axeses which is 1 1 2 and 1 1 1 and 2 3 3.

And, because it is well calibrated then we also know alpha 1 2, alpha 2 3 and alpha 3 1. So, one can calculate ND. So, what I mean to say here is that we will give this example in the next lecture class and we will show how the Kikuchi pattern is basically indexed. And then how the relative positions of the bands and the poles with respect to this external frame of reference RD TD and the ND one determines the orientation of the material right.

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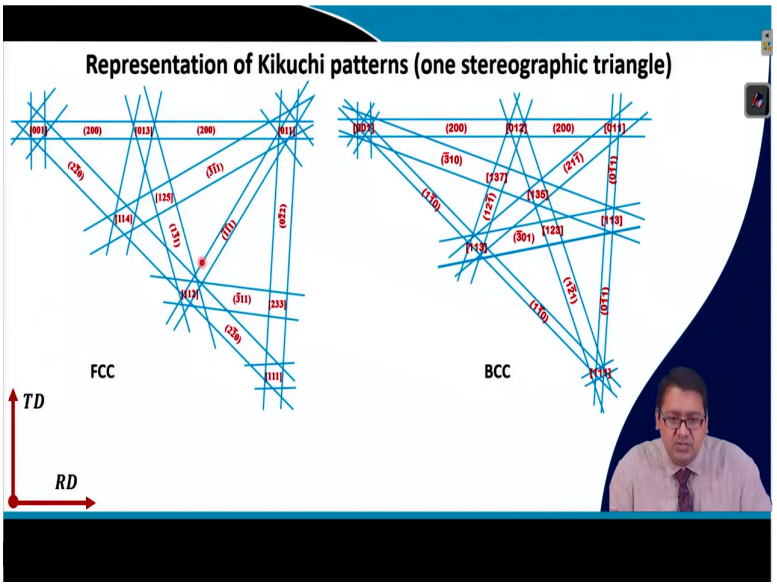

So, while we observe Kikuchi patterns for various materials of different crystal symmetry. Most of the time in case of metallic materials we utilize either it will be an we know that it will be either face centered cubic material, body centered cubic material or hexagonal close packed material.

Most of the time it is either face centered cubic like aluminum, copper and you see austenitic stainless steels or it could be BCC like you see ferritic steels. Most of the time if you work with ferritic steel say for example, then it is BCC definitely.

So, we often specific types of pattern which are representative of either it is coming from FCC or either it is coming from BCC. And you see, that usually an experienced person with an experienced eye can tell the difference between the pattern which is obtained which is coming from FCC or it is coming from BCC by just looking into it.

And just for the sake of demonstrating I will tell you that in case of FCC you see the zone axes's which are forming are 0 0 1 0 1 1 and 1 1 1 which is exactly same than the zone axes's which is coming from the BCC 0 0 1 0 1 1 and 1 1 1.

But you see, in case of FCC the other zone axes's are 1 1 4, 1 1 2 whereas, in case of BCC the zone axes's basically 1 1 3. So, there are these kind of subtle differences that occurs while we observe the Kikuchi pattern in case of FCC and in case of BCC and the software basically analyzes it.

But, the pattern type where that how this bands basically are intercepting to form you know major or minor zone axes's, determines that it is coming from an FCC material or on BCC material and an experienced eye can determine this just by manually looking at it.

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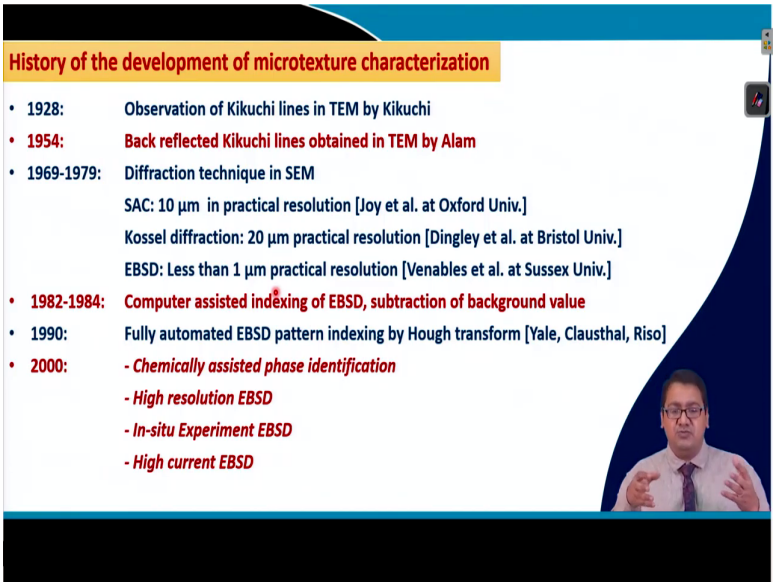

So, let us go and to the next this thing and discuss a little bit about the history of the development of the microtexture characterization. So, in 1928, it was Kikuchi who observed the Kikuchi lines in TEM by, he observed that when the samples are thick. Then instead of spots he was getting lines. And those lines are also in pairs and therefore, it was called Kikuchi lines and later it was Kikuchi bands and then Kikuchi patterns.

In 1954 back reflected Kikuchi lines are obtained in TEM and it was done by Alam. In 1969 to 1979 the diffraction technique using of in TE in SEM scanning electron microscope was developed slowly slowly including Kikuchi pattern.

So, you see selected area channeling diffraction was initially used and I gave a very brief information regarding that in the last lecture. And you see that the diffraction is basically back scattered diffraction and without a tilting and it was done by Joy et al at Oxford University.

So, in this case the practical resolution of this procedure was only 10 micrometer. The special resolution of the you know Kikuchi diffraction pattern was also very small. The steric angle

was very less. So, this was not further utilized. But yes, it was a step to you know develop this Kikuchi pattern analysis and understanding and obtaining orientation information.

Then as I said that Kossel diffraction using you know X-Ray diffraction technique. The X-Rays that are coming out from the sample are used by Dingley et al at Bristols University. And the practical resolution of this technique was only 20 microns. So, that is a technique which is their historically present, but it is also an advancement in the technique of understanding this kind of methods right.

So, during this 1969 to 79 you know EBSD electron backscattered diffraction technique by keeping the samples at 70 degrees was obtained by Venables at Sussex University; so Venables and co workers. So, here the resolution basically reduced to 1 micron, which was a much advantage in obtaining microtexture analysis.

So, in 1982 to 1984, now computer assisted indexing of EBSD started to develop and subtraction of background which comes during the you know Kikuchi diffraction patterns were utilized and to obtain proper Kikuchi pattern and information of the you know microtexture of the material.

So, in 1990s, slowly slowly fully automated techniques hardware integrated with softwares are slowly slowly were developing. And EBSD patterns are now you know indexed using the hough transformation technique. And, then there were many universities and you know national labs like Yale Clausthal University and Riso National Labs were involved in developing this separately.

So, in 2000, you know chemically assisted phase identifications can be done. Now, nowadays you see you can do EBSD of single phase and also of two phase samples, where at the same time while the EBSD is going on the you know electron backscattering techniques can be used to chemically, to understand the chemical composition or the variation of the chemical compositions of certain of the of those specific small volume areas of the samples where the Kikuchi pattern has been taken.

Now, the EBSD is slowly slowly becoming more and more high resolution. And, we have now nanometer say 20 nanometer, 30 nanometer you know electron beam size where the interaction volume may not be greater than 50 nanometer can be obtained for various metallic materials to obtain you know EBSD pattern from such small volume right.

Later developments are like, In-situ experiments in EBSD like for example, you see SEM integrated with a heating stage. A sample can be kept and it could be you know heated while the EBSD is being taken. So, an EBSD of an initial sample is taken to observe that particular area how the grain size, how deformed it is for example, it is a rolled sample keeping the RD TD intact.

And then it is annealed a little bit. Say for example, it is an aluminium material, so it is annealed at 150 degrees for half an hour and we and the observation of how the grains have you know increasing could be observed by doing you know in-situ EBSD. And then again the annealing can be done at little higher temperature and how the grains are evolving during the annealing process could be observed.

So, such kind of In-situ experiments. On the other hand you see tensile stages or you know fracture stages could be integrated with the SEM and in which you see one can use a tensile specimen and inside that SEM chamber and the EBSD is taken initially and then after a strain of say 5 percent and then again EBSD is taken at 10 percent and 20 percent like that.

And it can be done for you know compression test or tension test or you know some kind of flexural test or even for fracture toughness experiment looking into the plastic zone. So, various kinds of In-situ experiments can be done in EBSD in order to observe the variation in the microstructure and the texture altogether using this quantitative microstructural technique nowadays. And everything is available.

So, sometimes some materials are you know difficult to obtain Kikuchi pattern and therefore, the EBSD current can be increased or decreased. And for some specific use also, the current can be vary, that is the electron beam current can be varied to obtain Kikuchi patterns.

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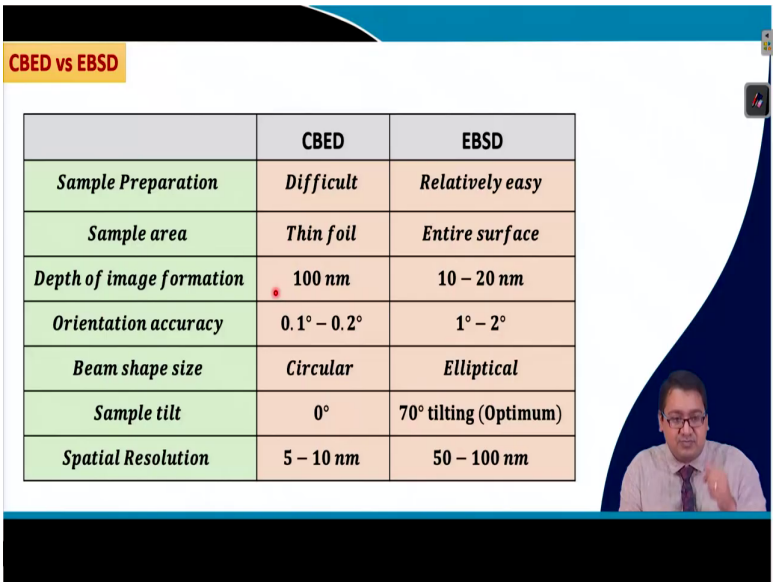

So, coming to the you know comparison between the convergent beam electron diffraction technique in transmission electron microscopy and the electron backscattered diffraction in the scanning electron microscopy. In case of CBED in abbreviation, the sample preparation is very difficult right.

We have to make thin you know TEM samples which are extremely difficult to prepare. In case of EBSD the sample preparation is comparatively much easier. So, it is a thick sample and we always will have some you know manual electropolishing or microprocessor controlled electropolishing techniques where the electropolishing can give a very nice you know strain free surface to do an EBSD.

Yes of course, the sample preparation for EBSD technique is also difficult in case of certain material and definitely it is difficult than that of for doing a simple microstructure you know analysis in an SEM or an optical microscopy, but it is relatively easier as compared to you know the CBED technique in TEM.

The sampled area is a thin foil because TEM is a transmission technique and EBSD, we can get the entire surface. So large area can be scanned using EBSD right. So, depth of image formation is in case of TEM, it will be higher 100 nanometer whereas, in EBSD because it is a backscattered electron which is diffracted the you know depth of image formation is only 10 to 20 nanometer which is also good in terms of microtexture analysis.

In case of TEM, convergent beam electron diffraction technique, the orientation accuracy is very high, 0.1 to 0.2 degrees right. In case of EBSD with the more advancement of the techniques, the orientation accuracy is reducing and reducing sorry increasing and increasing, so the angle is reducing and reducing, so it is now 1 degree to 2 degree in most advanced you know SEMs.

Now, in case of TEM the beam shape is exactly circular which is an added advantage because of which the angular accuracy and the spatial resolution will be always higher in case of TEM. In case of electron backscattered diffraction the beam is elliptical right. The sample is tilted at 0 degrees in case of TEM CBED, and it is tilted at 70 degrees to get the optimum EBSD pattern in the phosphor screen which is almost vertical with respect to the sample and it is away from the electron beam path right.

The spatial resolution in case of you know CBED is you know very high, 5 to 10 nanometer, in case of EBSD it is usually in a range of 50 to 100 nanometer. However, you see in case of CBED the steric angle is lower and in case of EBSD the steric angle is 5 times than that of the you know TEM.

So, you see, another important thing is that the calculation to obtain the information of the crystallographic texture from the EBSD is somewhat calculated be sorry complicated because of the you know geometric tilting of 70 degrees of the sample and the placement of the phosphor stream which is vertical. In case of CBED it is easy right.

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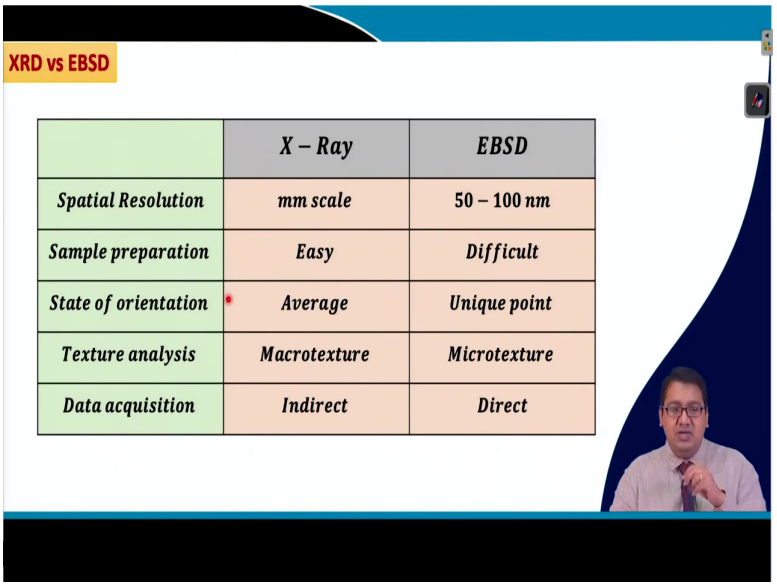

And finally, let us discuss a little bit about what is the difference between X-Ray diffraction technique and electron backscattered diffraction technique, because both of these techniques are used to carry out you know texture determination and it is most largely carried out.

So, you see X-Ray diffraction is basically a macrotexture determination technique, EBSD is a microtexture determination technique. We know what is the difference between the macrotexture and the microtexture. So, in case of X-Ray diffraction the spatial resolution will be millimeter scale. In case of EBSD the spatial resolution is nanometer scale.

So, in case of EBSD, one can obtain the microtexture information that involves the microstructural morphology. So, if you see, that in case of EBSD one can obtain phi 1 phi, phi 2 of the Euler angles, as well as x y and z that is the position from which it is coming from and that we can use a software to create the whole quantitative microstructure.

In case of X-Ray it is not possible to determine from where the X-Ray beam is coming from the diffracted X-Ray beam is coming from the information is from the total part of the sample because you see we when we studied is the divergent X-Ray beam and it is the incidental X-Ray beam and it is a convergent diffracted beam right.

So, in case of X-Ray the sample preparation is easier, in case of EBSD sample preparation is comparatively difficult. Because, in case of X-Ray the sample preparation we just have to polish the sample make it you know surface should be polished and there is no need to do you know electropolishing to make the surface strain free or there is no need to do you know etching or chemical polishing.

We just need to do get a high surface finish by doing a you know sample per you know polishing of the sample and maybe a little bit more like a diamond paste polishing or something. So, what is the state of orientation? So, in case of X-Ray diffraction technique in a goniometer that we get an average texture.

So, the texture information is the average texture information from the whole sample. So it is a sample property. In case of EBSD, the texture is from the unique point; that means, from the sampled area and which is unique point means it is the EBSD beam is also very small and so it is raster to obtain a certain sample area.

So maybe, if the sample is heterogeneous the texture which is obtained from EBSD may not be representative of the sample or may not be giving the exact property of the sample, but in case of XRD the large sample area along with a greater penetration depth, we get an average information of the texture which is basically the property of the sample.

Data acquisition technique in case of XRD, the data acquisition technique is indirect. Why it is indirect? Because, we measure the alpha beta by rotating the sample while the you know divergent X-Ray beam is falling over the sample and the convergent diffracted beam is going above. Now, during the time the sample is rotated in such a way so that for the whole pole figure you know the alpha and beta can be scanned.

So, it directly measures the pole figure and now the pole figure can be measured only up to 70 degrees. So, more than one pole figure at least two or three pole figures has to be measured calculated back to obtain the you know texture information in terms of you know orientation matrices and then in terms of phi 1, phi, phi 2 that is the Euler angles.

In case of EBSD, it is a direct measurement because the electron beams falls on the sample the Kikuchi bands are forming. The Kikuchi bands can be directly calculated to obtain the you know information of the RD TD and ND in terms of you know miller indices and thereby one can obtain directly the orientation matrices and thereby from the orientation matrices one can calculate the pole figure inverse pole figure orientation distribution functions of an Euler space and that is it.

So, these are the difference between you know CBED with the EBSD and XRD with the EBSD.

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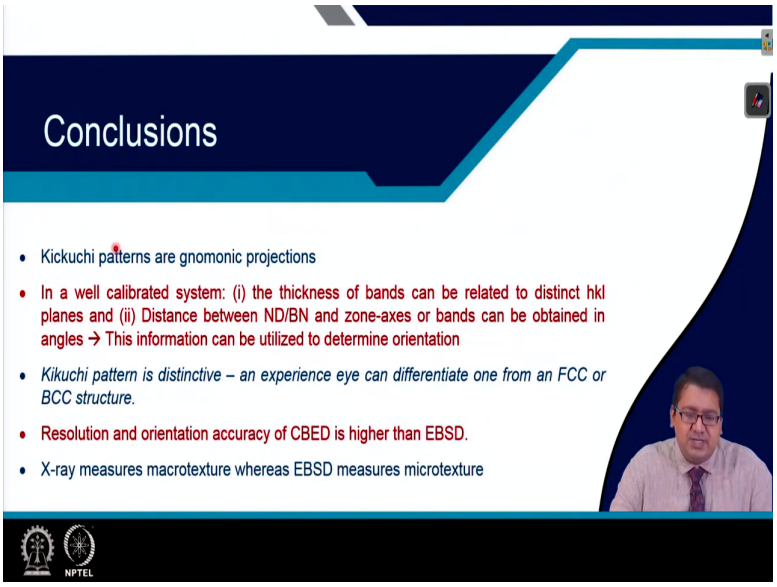

So, finally, what we can conclude from this lecture course lecture, that you see Kikuchi patterns are basically gnomonic projections, but we project it on the flat phosphor screen and because the lambda of the electron beam is so small the you know the diffraction patterns are not very away from the diffraction centre.

So, you see the distance of the diffraction pattern from the diffraction centre can be you know instead of tan tau it can be related to tau even if it is a gnomonic projection in most of the cases. So, if the system is well calibrated then the; that means, the distance between the sample and the phosphor screen is well known and well calibrated, then the thickness of the band which is in length basically in a phosphor screen can be you know converted into the angular thickness.

And then that angular thickness can be equal to is equal to 2 theta B can be related to a distinct hkl plane because 2 theta B is proportional inversely proportional to d hkl right. So, the distance between you know NB or the beam normal that is the incident beam normal and the zone axes or the bands can be obtained in terms of angles, as I described in the some slides before.

So, this information is basically used to you know determine the orientation of that particular point right. So, see finally, Kikuchi patterns are basically distinctive and experience I can basically distinguish between you know it is coming from either a FCC material or a BCC

material BCC structure. So, resolution and you know orientation accuracy of CBED in a TEM is much higher than that of EBSD in an SEM.

Finally, X-Ray gives information of macrotexture and we have give the given the difference between what is microtexture. EBSD gives the information of microtexture and that is the difference between the X-Ray texture goniometer and the EBSD measurement technique X-Ray gives indirect way of measuring texture. EBSD is a direct way of measuring texture and that is all for today's class.

Thank you very much.