Texture in Materials
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Module - 06
Microtexture measurements using EBSD technique in SEM
Lecture - 30
Kikuchi Diffraction Pattern - I

Good afternoon everyone. And today we will be continuing with module 6 which is Microtexture measurement using electron back scattered diffraction technique in a scanning electron microscope that is SEM. So, this is lecture number 30 and in which we will understand Kikuchi Diffraction Pattern and this is part 1.

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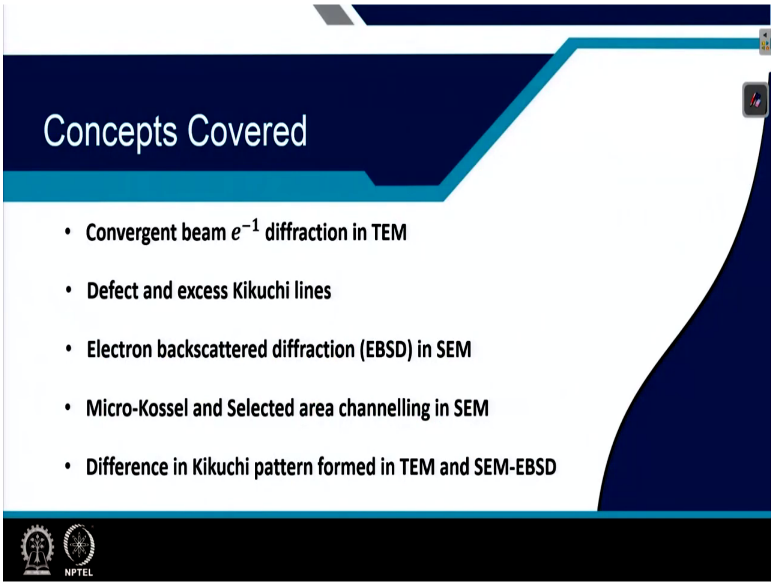

So, the concepts that will be covered in this lecture are convergent beam electron diffraction in TEM which is this one right. Just let me get the laser pointer. And then the second is defect and excess Kikuchi lines.

The third is electron back scattered diffraction in a scanning electron microscope, a brief very brief information regarding the Micro-Kossel and selected area channeling techniques in SEM and finally, the difference in Kikuchi pattern that formed in TEM and SEM-EBSD.

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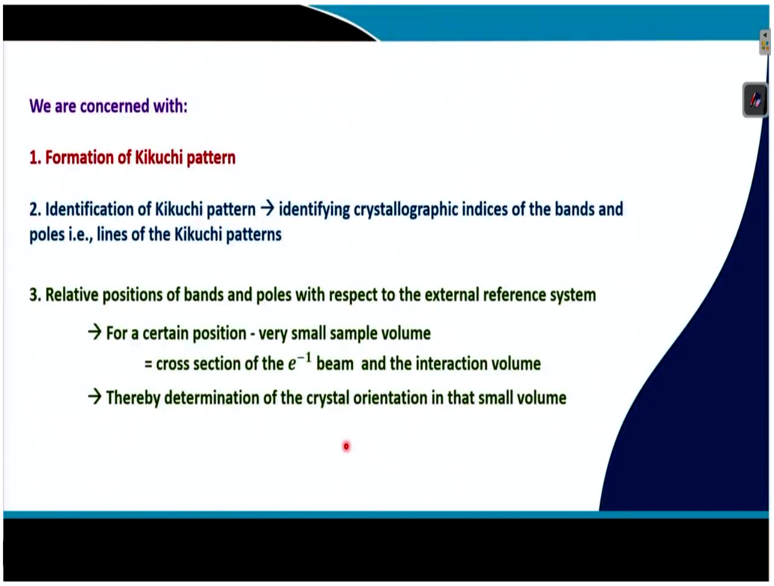

So, in order to obtain you know crystallographic information that is the information regarding the orientation that is the information regarding micro texture from a you know TEM or from a SEM that is EBSD we know and in case of TEM that is convergent beam electron diffraction technique we are mostly concerned with the formation of the Kikuchi pattern.

So, how the Kikuchi pattern can be developed and how it can be obtained properly then second is identification of this obtained Kikuchi pattern. So, we need to identify the crystallographic indices pertaining to the Kikuchi bands and you know the; you know the angle between the bands in form of zone axis right.

So, we need to understand the crystallographic indices of the bands and the poles and lines of the Kikuchi pattern, its relationship with you know the sample reference system. So, when we are putting the sample in the TEM or in the SEM what we are trying to do? We are putting the sample with knowing its specific important reference systems means, RD, TD and ND for example, for a rolled specimen.

So, if we can do that, if we can put the specimen in such a way that we know which direction is RD, which direction is TD and which direction is ND then we can get the relative position of the bands and the poles with respect to this you know external reference direction.

So, what basically is done is that for a certain position. So, you see the electron beam is a very thin beam it has a very small diameter. So, its it falls on a very very small area of a nanometer size. So, the beam is few nanometer diameter and it falls on that area and it irradiate it irradiates it and makes a you know interaction volume right a very small interaction volume.

So, from a very small sample volume in a poly crystalline material, the position which is definitely a single crystal because of the size of the sample volume the because of the cross section of the electron beam and the interaction volume a Kikuchi pattern is obtained from that position. And then that Kikuchi pattern is basically you know analyzed to obtain the as I said the relative positions of the bands and the poles with respect to the reference directions.

And it is basically you know from those bands and poles identification of the crystallographic planes with respect to this RD, TD and ND is obtained. And then the you know the electron beam is rastered. So, it is taken to the next step and then the same process is you know continued. So, another Kikuchi band pattern is taken and then it is shifted again and another beam falls on the side of from the other.

So, scanning of the whole sample volume you know first it is scanned like this 1, 2, 3, 4, 5, 6 and then it goes to the next step 1, 2, 3, 4 and then the whole area is scanned to obtain Kikuchi pattern from that whole area which may differ in some cases and which may differ which may not differ in some other cases. So, you see if the electron beam is basically rastered on a subjected you know small sample area to obtain the information of the microstructures like this.

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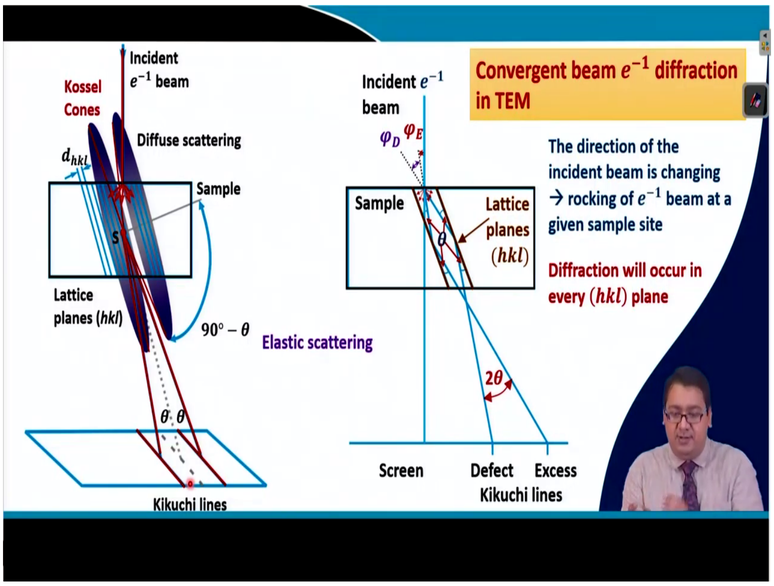

So, if we go deeper, so, how a Kikuchi band is produced? So, in case of the convergent beam electron diffraction in a transmission electron microscope when the incident electron beam falls on a sample a very thin sample and you know that the sample of a TEM is made in such a way that it is basically a round sample and it is basically thinner at the center and little thicker at the sides.

So, it is prepared in such a way that you know at the thin position one can get you know diffraction spot and one can go to the thicker and thicker position to see that whether you know Kikuchi bands can be obtained because of multiple you know scattering. So, how what is the technique that in that thin specimen a thicker a little thicker place is chosen and then the incident electron beam basically falls on the specimen. And when this electron beam falls on the specimen it starts to scatter.

So, you see it interacts the electrons interacts with the atoms and it starts to scatter and when it starts to scatter its scatterers in all possible directions right. So, you see when the scattering is one time then it is elastic scattering right as I said in the last class and when it starts to occur a multiple time it becomes inelastic scattering.

And how it is? It is such that that in every time the material the electron is scattering with when it interacts with the nucleus or the electron it loses a very little amount of energy and it is elastic scattering when the lost energy is not detectable. But when it interacts a lot of time multiple times with the electrons and the nucleus many times then the loss of energy become considerable and the scattering becomes inelastic.

We also know that when with the increase in the multiple times of interaction with the increase in the number of interaction the you know the scattering angle also increases. And as the scattering angle increases we also know that the intensity of the beam basically reduces.

So, you see that when the incident beam basically falls on the specimen and it interacts with atoms which are in periodic arrangement and so, it interacts with atoms in multiple times. And when it scatters multiple times it happens as if that the electron beam is coming from inside of the specimen and it is coming in all possible direction.

How it is that one electron beam is falling and then it is scattered one time. So, say for example, it goes in this direction and then its scattered another time and it goes like this direction, now multiple electrons are falling in the from the electron beam and they scatter multiple times using a lot of millions of atom present in the metal.

And so, the scattering basically takes place in all possible direction at 360 degrees in the space and therefore, the electron seems to come at all possible angles from inside the sample. And now when this kind of multiple scattering takes place this scattering becomes inelastic and the angle at which it is scattering it decides the intensity of the beam. So, higher the angle of scattering larger multiple times the scattering has taken place and therefore, the angle is higher and the you know intensity of the beam is lower.

Now, when such inelastic scattering takes place and the beam seems to come out in all possible direction from inside the sample then if a final a scattering takes place that scattering because it is a single scattering will be an elastic scattering. And when this scattering takes place and because of the periodic arrangement of the you know, various of the atoms the various hkl planes can lead to you know reinforced scattering which is basically diffraction.

And this diffraction occurs as if in a kind of you know locus of point like you know like a Kossel cone. So, the diffraction say for example, from a certain hkl plane may occur in all possible way in order to form a cone something like that. And it happens in all possible direction followings the Braggs law. Now, as you know that because the electron beam has a very small you know lambda.

Now, because it has a very small lambda the Braggs law is relaxed and because of which the a certain hkl plane can diffract from both the direction right. And as the electron beams seems to be coming from inside the specimen and in all possible direction the diffraction following the reinforced scattering following Braggs law that is the diffraction occurs from both the side of a single hkl plane.

And it forms a locus of you know point which makes a cone a cone like that in both the direction. This cones are basically known as Kossel cones right and like in this example we have shown it for a single hkl plane which is diffracting to form a Kossel cone all possible hkl planes basically diffracts.

Now, under such situation what happens that, these Kossel cones which are basically diffraction at you know Braggs angle theta for a certain hkl plane. So, theta b for a certain hkl planes the when this Kossel cones you see if it size increases because it is a ray right it will spread out right. So, when it spreads out and it spreads out and falls on the phosphor screen which is just placed below the incident beam and the sample.

So, when it falls on the phosphor screen it forms two you know lines and this lines are basically you see the angle between these two lines are basically 2 theta. So, if we make an bisector of this line which is basically parallel to the hkl plane that it becomes you know theta and this one also theta right. So, theta plus theta is 2 theta.

Now, these Kossel cones when falls on the phosphor screen because of the you see because the angle of diffraction is so small as the lambda is so, small for an electron beam for each and every hkl plane the Kossel cones you know the when it falls on the phosphor screen it forms straight lines like this. So, you see that in this way you know Kikuchi lines can be obtained from TEM using convergent beam electron diffraction.

Now, when the incident electron beam falls on the specimen, the incident beam is you know slightly you know tilted you know, it is called rocking of the incident beam. The rocking actually takes place at a very small theta so that the electrons that are generated in the inside the material by the method of you know multiple scattering or inelastic scattering seems to come from all possible direction.

And such that a proper you know Kikuchi pattern or a proper Kossel cone can be obtained from which the Kikuchi pattern can be formed. Now, you see that here is a little detail of how the Kikuchi pattern basically develops in case of the convergent beam electron diffraction inside the TEM. You see when the electron beam falls on the specimen and as I said that the direction of the electron beam is you know it is falling. So, it is slightly rocked.

So, it changes. This is known as the rocking of electron beam at a given sample site. So, as the electron beam is falling on the specimen, it is slightly rocked and you see multiple scattering takes place, this red arrows are shown which shows that multiple scattering takes place in the specimen.

And as I said that as the number of scattering increases the angle of scattering basically increases. So, as for a small number of scattering that is phi E, the angle is less right and the intensity of this beam which seems to be come from inside the specimen is basically you see higher right. And when for a certain hkl plane this electron beam basically comes and diffracts you know finally, final elastic scattering which is reinforced scattering following the Braggs law happens.

Then this incident beam falls here and its diffracts and goes to the outer position of the Kikuchi band. Now, on the other hand when the number of scattering that is increased then the in inelastic you know scattered in a in inelastically scattered you know electrons has lower energy and it has an angle that is phi D which is greater than phi E.

So, when it falls on the same hkl plane it falls like this and it scatters a inside that is reinforced scattering following Braggs law that is diffraction following a particular theta be for a certain hkl plane for the same hkl plane in this case, this beam has a lower intensity when it falls here.

So, what happens? That while the incident electron beam also falls straight on the screen because the specimen is so thin, it is almost electron transparent and so, it falls on the screen it the intensity of this electron beam which is diffracted and falling here will be lower and the incident and the electron beam which is diffracting and fall falling here will be little higher leading to form a Kikuchi band.

You see Kikuchis are basically two lines which are forming one line is inside near to the incident beam and another line is outside. So, the outside line becomes you know excess it is called the excess Kikuchi line because it is brighter and the inside line is called the defect Kikuchi line because it is basically you know darker because its intensity is lower.

So, you see like that we can in convergent beam electron diffraction. In a TEM we can we will obtain each Kikuchi bands with a excess Kikuchi line and a defect Kikuchi line. So, the excess Kikuchi line will be outside and the defect Kikuchi line will be inside for each and every hkl plane right.

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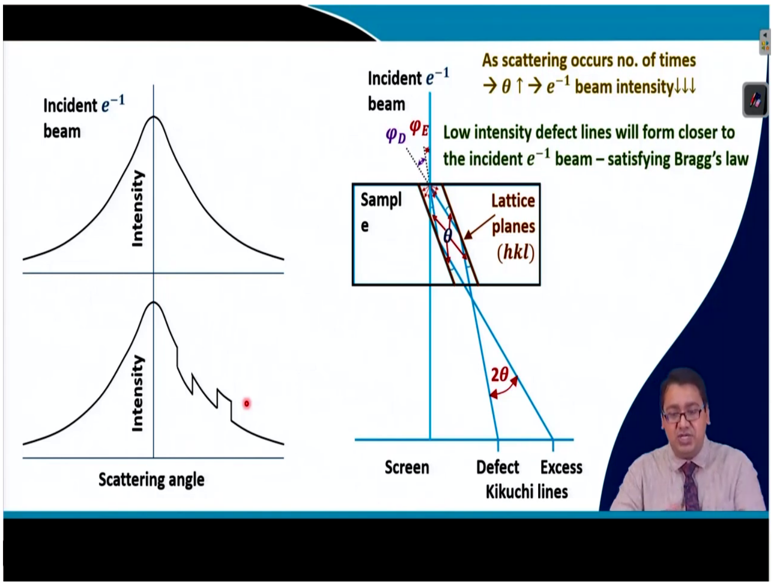

And therefore, you see that when such a situation arises when such a situation arises then what happens that the if I explain in a little detail that as I said that this electron beam which is diffracted and coming inside forms a low intensity line which is defect line. And the beam this beam which has an high intensity diffracted outside to form a high intensity excess line both basically satisfies the Braggs law.

So, if we look here in the figures on the left side if there is no sample present or the sample is present in the thinner position where there is no Kikuchi formation, then the intensity of the incident electron beam falling on the you know the surface of the phosphor screen will look something like this.

But, you see that once this you know Kikuchi bands starts to form say for example, for a certain hkl plane if we show and then the excess line and the defect line generates. Then what is basically happening is that the intensity which is present here which has to be here now, because of the diffraction is going and falling here whereas, the intensity which usually will fall here without the Kikuchi diffraction will go and fall somewhere here form somewhere here.

So, you see that because of such a phenomena the intensity of the direct incident beam reduces in one case and increases in the other case for you know one Kikuchi line and another Kikuchi line in for a Kikuchi band to form such kind of excess defect Kikuchi lines for in each and every hkl plane.

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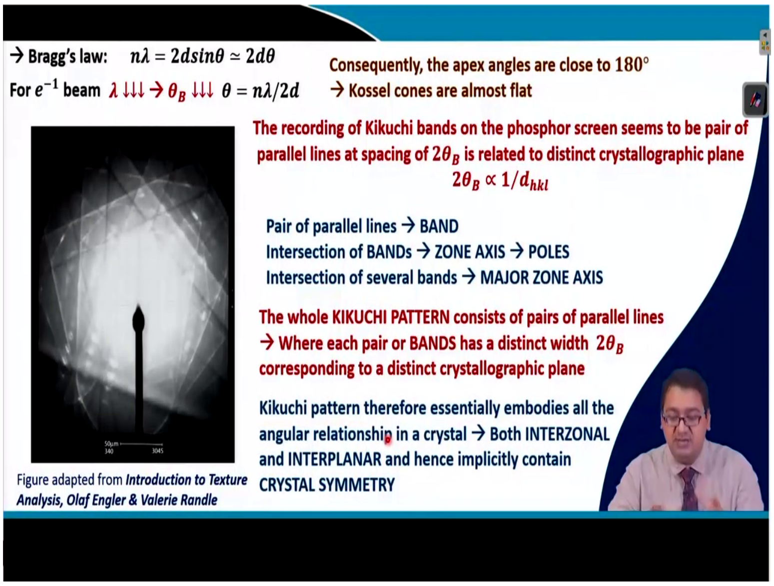

So, here we are showing the figure. This figure has been adapted from the figure present in Introduction of Texture Analysis book of Olaf Engler and Valerie Randle. And you can see what I was trying to explain you is that here is for example, a band right, this is a Kikuchi band out of many Kikuchi bands. So, this is another Kikuchi band this as another Kikuchi band that has formed.

And if you look the inside band inside line of the band is darker right and you see for this band also the inside line is darker you see for every band the inside line is darker the inside line is darker. And if you look at the outside line the outside line is basically brighter. The outside line you see for this Kikuchi band also and for this Kikuchi band also is basically brighter.

So, when the incident electron beam falls on a very thin specimen from where we can obtain Kikuchi band means from the thicker part of the thin specimen and then the direct incident beam falls making a very bright area on the phosphor screen. However, when excess and defect lines arises because of the diffraction forming strength of the Kossel cones and formation of Kikuchi pattern the diffraction pattern basically looks like this.

And when if we know that how we have put the sample inside the TEM and if we know the RD, TD and ND then one can relate these you know lines or bands with respect to you know the RD, TD and ND in order to obtain the information of the orientation of that particular position of the sample right. So, as I said that if we look into the Braggs law and we have learned this in the last class also that n lambda is equal to 2 d sin theta.

But, as the sin theta as the theta is so small because the lambda is also so, small for the electron beam, we can say that n lambda is equal to 2 d theta. So, theta is basically you know n lambda by 2 d which is you know so small. And therefore, what happens that because of this small theta which is in 10 to the power minus 2 radians or something the apex angles of this Kossel cones you see we form this Kossel cones right cones like this it comes like this from a single hkl plane.

And the apex angle is almost close to 180 degrees. So, the Kossel cones are almost flat and. So, that the recorded Kikuchi pattern you see Kikuchi pattern on the phosphor screen. So, each band of the Kikuchi pattern on the phosphor screen seems to be a pair of parallel line.

And you know if the distance of the sample and the phosphor screen is properly calibrated then the thickness of this these bands each of these bands could be related to you know 2 theta B right because, but a proper calibration is needed and once it is calibrated we know that we can relate each thickness to 2 theta B.

So, this is 2 theta B for a certain hkl plane and this is another 2 theta B for another hkl plane. So, from the thickness of the band one can find out which hkl plane it is coming from; because from this formulation 2 theta B basically is inversely proportional to d hkl right. So, it is inversely proportional to the lattice spacing of the planes right.

So, we have discussed, but to brief it; the pair of parallel lines are basically known as bands the intersection of bands are basically zone axis. And in case when in certain case when there are several bands intercepting at the same point it could be a major zone axis right.

So, the whole Kikuchi pattern basically consists of pairs of parallel lines right where each pairs of lines has a distinctive width 2 theta B corresponding to this crystallographic you know plane. So, is proportional to 1 by d hkl then the Kikuchi pattern therefore, essentially embodies the angular relationship in the crystals.

So, both interzonal and interplanar and hence the it can implicitly contain the crystal symmetry of the material. So, this gives this has the crystal symmetry of the material and can give the information of the crystallographic texture of the material if RD, TD and ND of this sample which is put in the TEM is known.

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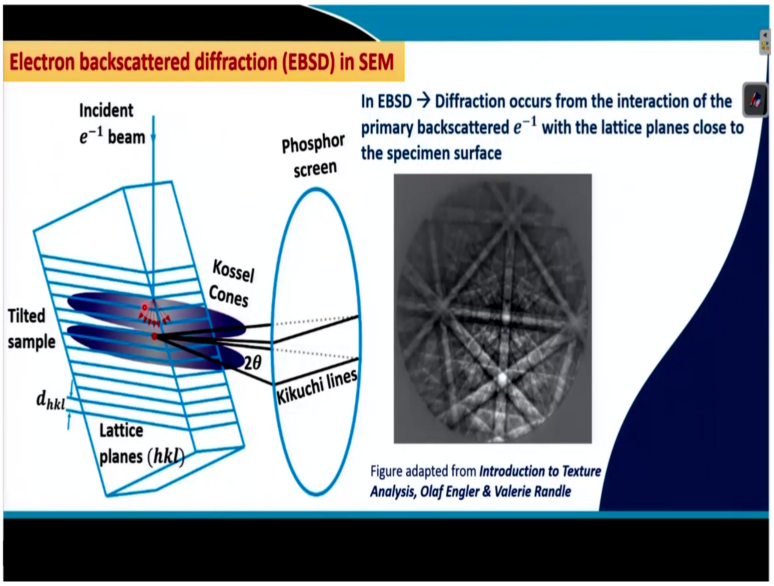

In case of scanning electron microscope the process of doing or observing or obtaining this Kikuchi pattern is known as electron backscattered diffraction. Now, as I have said in the previous lectures that there are you know elastically back scattered electron and elastically scattered in the front side in case of TEM right.

So, say for example, there could be inelastically back scattering and then finally, it could be diffracting. So, you see that when the electrons basically interact with the nucleus or if it can we can get elastically back scattered electron, but if we can tilt the sample and if you can tilt the sample at say for example, at a certain degree say 60 degrees to 80 degrees something like this.

So, when the electron beam basically falls on the sample and once it falls on the sample by the same phenomena as I explained in case of convergent beam electron diffraction in the TEM, when the electron beam falls on the sample elastic scattering takes place if the scattering is less number of times right when it is the loss of energy is not detectable.

And elastic in an inelastic scattering takes place when it you know scatters multiple amount of time and so, there is a loss of energy of the electron. So, while multiple scattering takes place in the sample near the surface at the interaction volume for a certain small part where the electron beam is falling, you know as I said that electron beam has a diameter of few nanometers.

Now, you see when multiple time scattering occurs while the sample is tilted like this it seems that the scattering is making the electron to come out in all possible direction in the space as if the electron source is inside the sample. So, it seems that the electron source is somewhere here from where the electron is basically coming out in all the direction like this.

And when such a situation occurs then what happens that the; if the electron comes out after the final scattering and if it is a reinforced scattering because of the presence of you know periodic lattice arrangement of the crystal of the atoms. Now, what happens that in this case also say for example, many electrons are coming on the above direction and few are coming in the lower direction and it is throughout.

And then each of this inelastically scattered electron basically finally, diffract to form in a locus of a cone right a locus of a cone same like the one which we observed in the TEM, but you see in this case also for a different geometry right. And this locus of the cone for a certain hkl plane can be observed on in a phosphor screen which is not present below the incident beam, but present at 90 degree away from the electron beam right.

So, it is present at a different position from the electron beam. So, in this case also the Kossel cones have a very small you know apex angle. And thereby what happens that when it the electron which is finally, diffracted falls on the phosphor screen forms you know Kikuchi lines. And here also a band contains two lines and the angle between the bands could be related to 2 theta right and it forms like this.

So, this is a figure which is obtained from the book of Olaf Engler and Valerie Randle, Introduction to Texture Analysis. And you can see that for different hkl planes one can obtain different bands right and these bands are forming each band has a different thickness. And therefore, it can be related to the you know 2 theta B means the 2 times of the Bragg angle and thereby it could be related to a certain hkl plane.

So, if the sample is placed in such a way that the RD, TD and ND that is the important sample reference direction is known then one can relate and find out these crystallographic planes and then relate it to the sample reference direction to find out the orientation. So, in

EBSD diffraction occurs from the interaction of primary back scattered electrons with the lattice planes which are close to the specimen surface right.

So, in case of TEM you see it is a transmitted electron which gives the Kikuchi pattern. And in case of you know the electron backscattered diffraction that is EBSD, the it is in the reflection geometry right to obtain the Kikuchi pattern. And you see in this case the planes which are somewhat like this will be irradiated.

And therefore, you see each and every plane will be irradiated which are you know in parallel to this direction almost parallel to this direction. Each and every plane will produce an Kikuchi line or Kikuchi bands.

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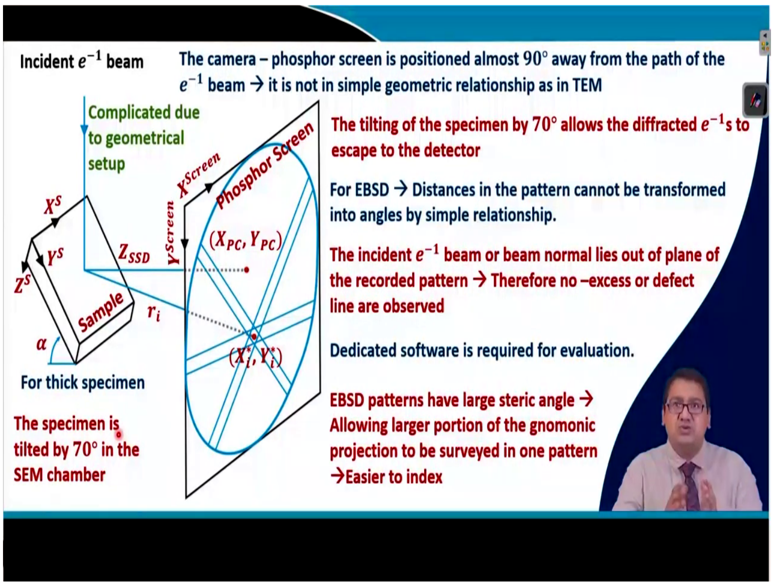

So, you see that in case of EBSD why it is important to observe EBSD than the TEM? Because you see in case of TEM the sample preparation is extremely tough. In case of TEM to you know raster the sample in a large amount is also difficult and texture is also related to you know statistics.

Even if it is micro texture a large number of grains needed to be scanned to get sufficient information to for the confidence of the you know researcher or the scientist who is working that the information generated is quite representative. So, in case of EBSD what happens? Thicker samples can be used to do EBSD. So, the sample preparation becomes much easier and therefore, EBSD is a more you know known and understood technique though its geometry is complicated.

Because you see its geometry is complicated because the sample is rotated at a certain angle and one can rotate at various you know alpha. 70 degree is mostly preferred because at this angle the electron beams Kikuchi pattern can be obtained in a phosphor screen which is present now vertical with the sample.

You can see that if this is the incident beam and perpendicular to the incident beam is not somewhere which is you know parallel to the one which is representing a major you know zone axis of the specimen, but it is some. And it is not even at the center of the phosphor screen, but it is relatively at certainly at the top position somewhere at the top position right.

So, what I mean to say is in this case the camera or the phosphor screen is presented at 90 degree away from the path of the electron beam. And this is not a simple geometric relationship as in the TEM. So, you know complicated mathematical expressions will be needed to solve the you know Kikuchi pattern to obtain the hkl planes and to relate it to the RD, TD and ND of the sample. Of course, this will be, but it can be done using a software.

Now what happens that why we do the tilting? If we do the tilting at 70 degree it helps to you know ah the electron beam to diffract and it goes directly to the detector which is placed at vertical in this case. Now, in case of EBSD the distances of the pattern cannot be transformed into angles by simple relationship and that is what I said earlier because of the complex geometry.

The incident electron beam or the beam normal lies you see the incident electron beam or the beam normal and this is the same thing B n actually lies out of plane of the you know the phosphor screen. So, the phosphor screen is here and in case of TEM it used to be here. So, therefore, you see because the phosphor screen is 90 degree away it is vertical now, it is out of the line of the electron beam.

Now, in case of EBSD there would be no excess or defect lines right. So, in this case you will have no excess or defect lines and that is what we should I should have said in the last slides also. Now, in order to you know evaluate this Kikuchi pattern and to raster the whole microstructure a part of the microstructure sampled area and to obtain multiple millions of

Kikuchi pattern for a you know small area of the sample and then to obtain the texture a dedicated software is required right.

So, what is the advantage here? The EBSD pattern that is obtained in obtained in SEM has a much larger steric angle nearly 5 times larger than what is obtained in case of you know convergent beam electron diffraction in a TEM. So, what happens is that is it allows a large portion of the go gnomonic projection to be surveyed in one pattern and therefore, it becomes easier for indexing.

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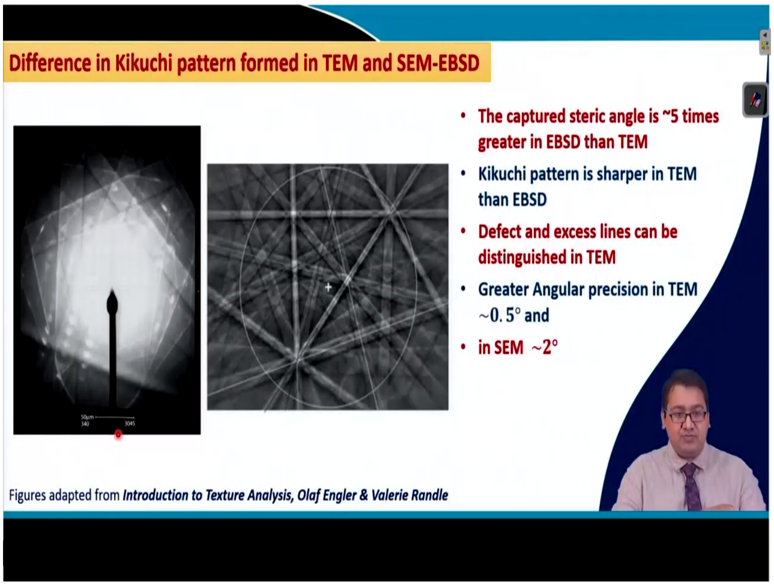

So, you see what is the difference between the Kikuchi pattern that is obtained from the transmission electron microscope and what is it different from the scanning electron microscope in EBSD. So, you see. In case of EBSD a large steric angle nearly 5 times can be obtained as compared to the TEM case right the. However, in case of TEM the Kikuchi patterns are sharper and more prominent than that of the EBSD.

In case of TEM there is a presence of you see defect lines and excess lines. In case of EBSD there is no defect lines or excess line they have equally dark intensities in both the lines right. Now, the angular precision of TEM is very high that is 0.1 degree to 0.5 degree. In case of SEM the angular precision is low that is nearly 2 degrees in most of the cases.

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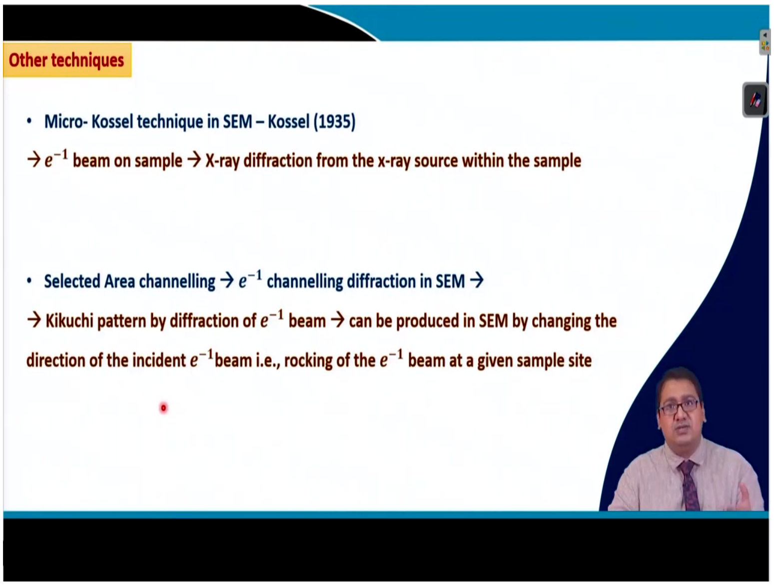

So, there are other techniques in SEM that can be used to obtain you know Kikuchi patterns or to obtain orientation relationship with respect to the specimen. And one of the technique that I would like to mention in a very for very brief that it is Micro-Kossel technique and this is Kossel in 1935 who basically showed this and you see in this case electron beams actually falls on the sample.

And the X-ray diffraction is basically taken because you see electrons falls on the sample produces you know diffracted X-rays also. And this diffracted X-rays are basically used to ah understand the orientation relationship of the material. And this diffracted election diffracted X-ray beams comes from inside the sample. On the other hand, another technique another important technique, selected area channeling is one of the technique where the sample in the SEM is not tilted, it is kept flat like that of the TEM.

In this case what happens that the electron channeling diffraction takes place in the SEM and the Kikuchi pattern and the electron beam falls and the back scattered diffraction takes place like this. And there is a detector on the top along with the electron you know gun and the Kikuchi patterns are obtained on that phosphor screen. And, but you see it the Kikuchi pattern that is obtained has a very very small steric angle. So, in this case also the incident beam has to be rocked.

So, rocking of electron beam at the sample site is done so that the electron comes out in all possible directions in order to get the final you know reinforced scattering. So, these are two techniques which are basically not used for nowadays to do this orientation or micro texture measurement.

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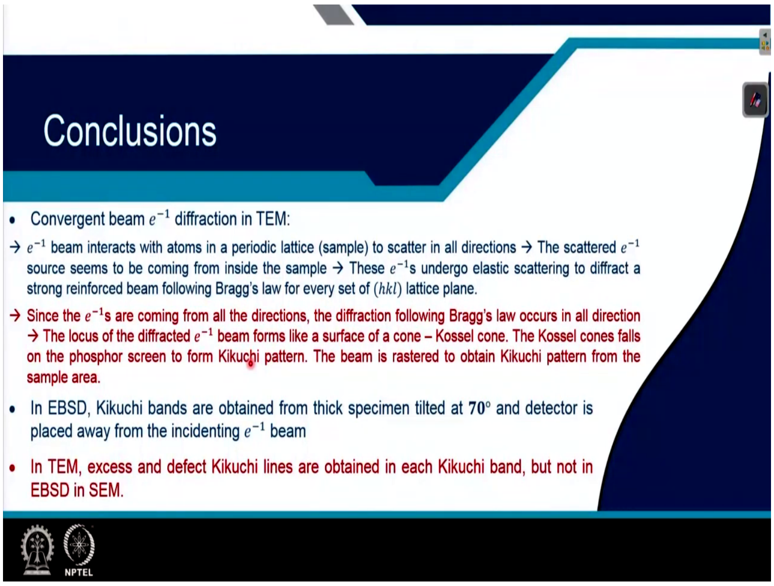

So, what we can conclude from this today's lecture is you see convergent electron diffraction in TEM is used in such a way that the electron beam is basically you know rocked while it falls on the sample. And it interacts with the atom in the periodic lattice arrangement of the sample and it scatters in all possible direction.

So, the scattered electron source seems to be coming from inside the sample right. And these electrons undergo a final elastic scattering and this elastic scattering actually will be reinforced scattering. That means, it will follow the Braggs law and it will diffract because of the periodic arrangement of the atoms. And this strong reinforced scattering will occur for every set of hkl planes, right.

Now, since the electrons are coming from all possible direction the diffraction following the Braggs law will also occur in all possible direction, but following Braggs law right. So, the diffraction will form a locus of diffracted electron beams which is like a surface of a cone and it is called a Kossel cone, right. The Kossel cones basically falls on the phosphor screen to form the Kikuchi pattern.

Now, the beam is basically rastered one after another. So, it scanned the whole small sample area to obtain the Kikuchi pattern of the sampled area that we have decided. Now, in case of

EBSD, Kikuchi pattern is obtained from a thick specimen which is easier to you know the sample preparation of thick specimen is much easier as compared to the TEM sample preparation.

Well, thick specimen is basically tilted and the detector is now vertical instead of horizontal detector which is now away from the electron beam. So, you see in this case also Kikuchi bands can be obtained, but with a lower angular precision. Now, because of this difference in the arrangement in case of TEM excess and defect patterns are observed. In case of EBSD no such excess or defect lines are observed. That is all for today's class.

Thank you very much.