Texture in Materials
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Module - 06
Microtexture measurements using EBSD technique in SEM
Lecture - 29
Basics of Electron Microscopy – II

Good afternoon everyone and today we will starting with module 6 again Microtexture measurement using EBSD technique in SEM and we will continue with the understanding of basics of electron microscopy. So, this is Basics of Electron Microscopy part 2.

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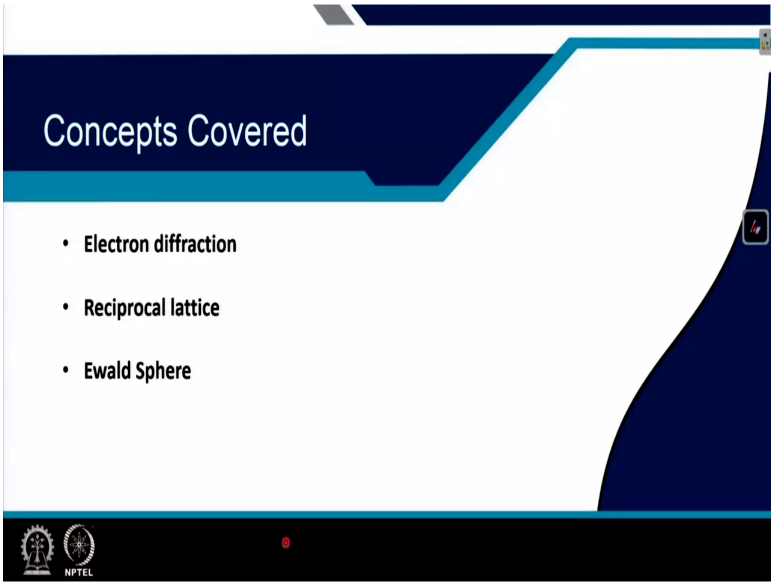

So, the concepts that are covered in this lecture are the electron diffraction techniques, the formation of reciprocal lattice says and how it looks like a little bit of information about the Ewald sphere construction.

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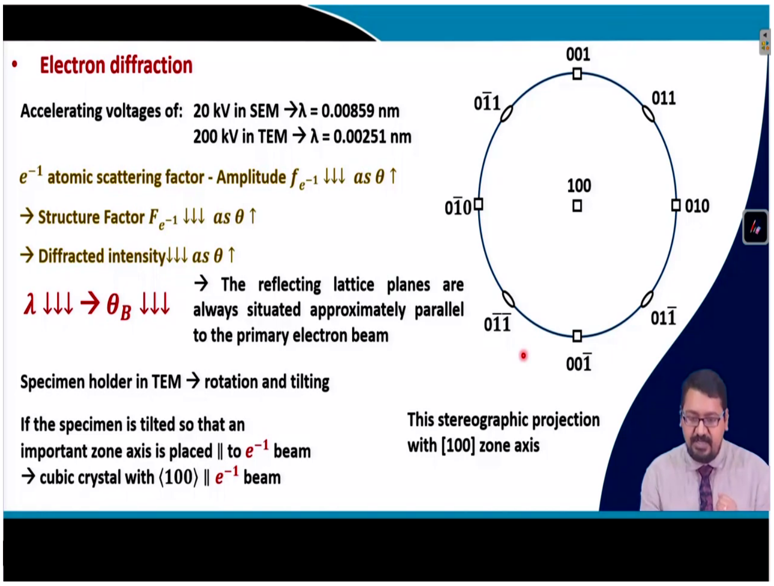

So, let us start with electron diffraction. We all know that when an electromagnetic radiation is diffracted by the presence of periodic lattice arrangement in a crystal structure what happens that, if the zone axis basically the direction at which the electromagnetic radiation is moving if it is you know 100 here is the figure.

Then the positions of the poles other important miller indices pole are positioned in such a way that they are in the surface of a sphere which is basically stereographic you know sphere which is being projected in a form of a two dimensional circle which is known as a stereographic projection and this stereographic projection we have learned in greater detailed in the previous lectures right.

And this is the 100 stereographic projection because it has 100 zone axis and we can see that how the positions of various other 01 bar, 001 0001 planes are represented in poles in the circumference of this you know stereographic projection. But when we talk about electron diffraction, the electron diffractions you know the electron beam the energy of the electron beam depends upon the voltage.

Now, in case of SEM the usual voltage is around 15 kilo volt to 30 kilo volts and then as the voltage between the cathode and the anode; that means, the electron gun and the you know sample which is anode is increased the lambda that is the wavelength basically decreases.

So, if you look into the voltage range of the SEM the lambda is in the range of 0.00859 nanometer you can see that it is so, less right and when we look into high resolution transmission electron microscope, where the voltage can increase up to 200 kilovolts we can have lambda wavelength of the electron beam to be equal to you see 0.00251 for example, for 200 kilovolts.

So, you see that on the other hand we have also learned that you see the electrons atomic scattering factor the amplitude of atomic scattering factor given by small f right always decreases largely as the theta increases. On the other hand, the structure factor which depends upon the atomic scattering factor capital F also will decrease then as the theta that is the you know scattering angle increases.

And basically in this case it will be the Braggs principle followed scattering angle that is the diffraction angle theta b. So, the diffraction angle if increases then the diffracted you know intensity of the electron beam basically decreases largely. So, as the lambda is decreased the theta B that is the Braggs angle theta basically keeps on decreasing.

That means, that particular h k l plane will now diffract at a smaller Bragg angle because the lambda has been increased n lambda equal to 2 d sin theta right. So, the reflecting lattice planes are actually in case of electron beam are situated almost approximately parallel to that of the you know incidenting electron beam the primary incidenting electron beam.

So, you see what we can do that as the sample which is a very thin sample in case of TEM and which is on the holder put in a copper grid and that sample holder or the specimen holder of the TEM is such that that one can you know rotate and tilt that holder you see and when you can do that, you can bring that sample a certain important axis say for example, 100 axis you know parallel to the electron beam.

That means, we can place the 100 plane in such a way so, that its normal is basically parallel to the electron beam and then what do you mean? That axis becomes the zone axis.

So, now, 100 becomes the zone axis and we are talking about a cubic crystal where we are trying to put 100 parallel to the electron beam and making it a zone axis. And so, that we can compare it with this stereographic projection here where if 100 is the zone axis then the positions of the you know smaller miller indices 100 110’s are situated at the you know circumference of this sphere or the circle right.

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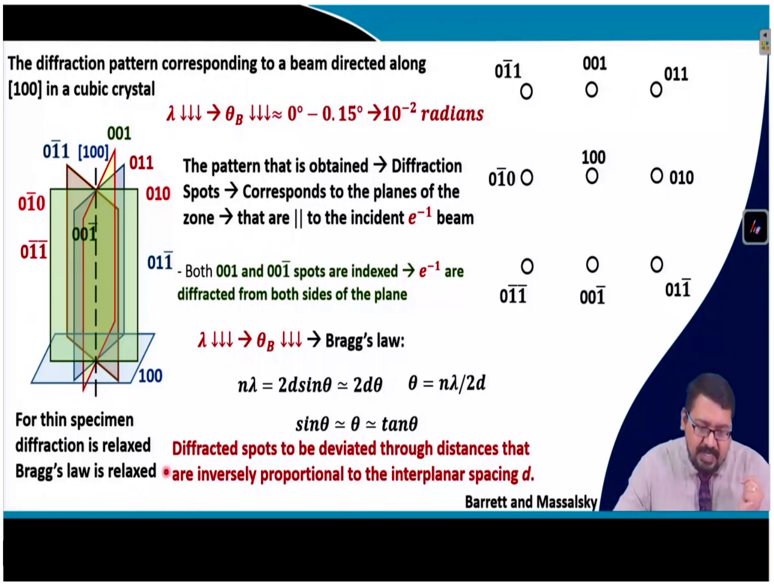

So, if we look ahead the diffraction pattern that can be obtained in case of you know electron beam striking this cubic crystal with the say you know sample holder being rotated such that the zone axis is 100. That means that the plane as I said 100 the perpendicular to the plane is parallel to the electron beam direction right.

So, once it is such then you see if we have a plane which is parallel to the beam direction; that means, this plane. So, the electron beam will is falling along this plane and then it will get diffracted as because the lambda of the electron beam is so, small that the theta B is so, small.

See in a range of 0 to 0.15 degrees and therefore, in a range of 10 to the power minus 2 radians, then you see the n lambda equal to 2 d sin theta could be n lambda equal to 2 d theta and this theta and lambda is so, small then the diffraction on this plane can occur in both the direction.

So, it can occur in this direction and it can occur in this direction in order to get both 010 pole and the 01 bar 0 pole and these poles will be represented here in form of reciprocal lattice right. On the other hand, you see if we look into the other plane say for example, this plane which is you know 001 plane if you look away and then if you look towards then it is 001 bar plane and this single plane will be diffracted in both the directions ok.

And then it will give a pole 001 and 001 bar. Like that the diffraction can be obtained from the 01 bar one plane or the 011 bar plane which is the same plane with this direction as 01 bar 1 and this direction as 011 bar and could be represented in these positions like that. The other plane right the other plane this one the red colored plane with the direction this 011 and this direction 01 bar 1 bar could be represented here in form of a reciprocal lattice.

So, the diffraction that is obtained from an electron gun or the electron beam looks something like this in case of a you know simple cubic crystal structure and you see the diffraction spots basically corresponds to the planes of the zones that are basically these planes are basically almost parallel are at fully parallel actually with respect to the incident electron beam.

And you see both you know for example, as I wrote here 001 001 bar or you can say 01 bar 1, 011 bar spots could be indexed from the same electron beam traveling in the same direction and basically we can do that because the electron beam is slightly you know convergent very slightly it is convergent or it is rocked and so, that both the diffraction could be obtained.

And this convergence angle is need the need of that convergence angles is very low you know like 0.15 or less than that even and you can see that diffraction from a single plane can take place in both the directions. So, as I was say that lambda is so, low that the theta is very low and the Braggs law is basically you know deviated because sin theta is basically equal to theta. So, n lambda equal to 2 d theta.

So, theta is equal to n lambda by 2 d here. So, one can say that sin theta is equivalent to theta equal to tan theta in this case right. So, for very thin specimen for very thin specimen and we know that for TEM we need very thin specimen the diffraction also becomes relaxed because you see the reciprocal lattice becomes elongated while the sample thins down.

So, that is we will come to the next slide. But you see the diffracted spot is basically deviated through the distance which is inversely proportional to the actual distance of that interplanar spacing what do we mean by this? We will show you in the next slide.

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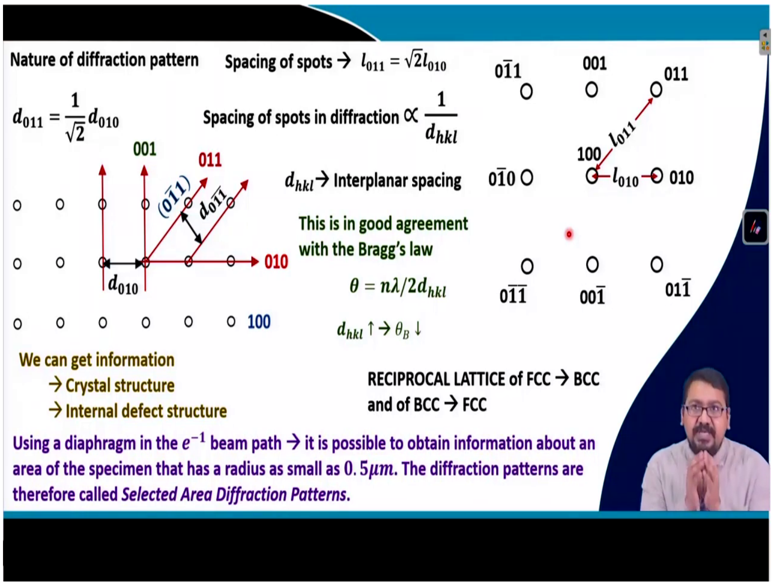

If we look into the nature of the diffraction pattern there are spots. So, this is 100 and this is a direct beam which is parallel to the you know the zone axis which was kept 100 right. And this is the distance from the 010 plane which is like that parallel to the electron beam and the 011 plane which is parallel to the electron beam.

And if you look into the spacing of the spots between 100 and 011 and 100 and 010 which is given by you see the spacing of spot between 100 and 011 is given by 011 is given by l 011 and between 100 and 010 is given by l 010. So, l 011 is basically equal to root 2 l 010 and we can see it from the figure.

But if we look into the actual crystal arrangement for this cubic unit cell you will find out that the crystal arrangement and you are looking around you see again in the axis 100 where the plane this plane along with the screen is 100 and on which the atoms are arranged like this in you know periodic fashion at equal distance.

So, if these planes are 010 planes and the perpendicular to this plane the direction is 010 right. So, the distance between these planes that is d 010 is given and this is this much. We look in this in the same plane if we call it a plane this plane 010 the direction along the vertical is 001 right. On the other hand if we are looking into this particular plane which is 01 bar 1 this is also 01 bar 1 and the direction to this is 011 the same direction.

Then the distance between this 01 bar 1 plane is given by this particular d right. Now if we compare that this distance with this distance then d 1 sorry d 011 bar becomes equal to 1 by root 2 d 010 right whereas, this is in the real lattice space and what happens that l 011 equal to root 2 l 010 in the reciprocal lattice space.

So, the distance of the spots of the reciprocal lattice space is inversely proportional to the distance of the planes in the real lattice space right and the spots in the reciprocal lattice space are basically the planes only. So, you can see that the spacing of the spot in the diffraction is inversely proportional to the lattice interplanar spacing right not the lattice spacing interplanar spacing.

So, actually this is in very good agreement with the Braggs law why it is so? Because you see theta is equal to n lambda by 2 d for that h k l plane and what is theta? Theta is basically the distance between the two spots from the center spot to this spot right. So, as the d h k l is increasing then the theta is decreasing or the d h k l is decreasing then the theta is increasing vice versa.

So, this reciprocal lattice spacing actually you know is in good agreement with the Braggs law with respect to the you know interplanar spacing for the real lattice case. So, we can tell here that reciprocal lattice of face centered cubic material looks like you know BCC whereas, the reciprocal lattice of body centered cubic material looks like FCC Face Centered Cubic and this is basically the way while crystallographers understand that what is the basic lattice structure by observing the reciprocal lattice of a certain unknown element.

So, we get the information about the crystal structure, we can get the information of the internal you know defect structure of the material, at the same time using this technique. So, what we do in electron transmission electron microscopy in order to get this kind of diffraction is that, we use a diaphragm in the you know electron beam path.

And the diaphragm has a you know very small radius about 0.5 micrometer and it helps us it makes it possible to obtain information from a very small area of the specimen a particular position where it is a single crystal in the poly crystalline specimen and therefore, this kind of you know diffraction pattern are called as selected area diffraction pattern ok.

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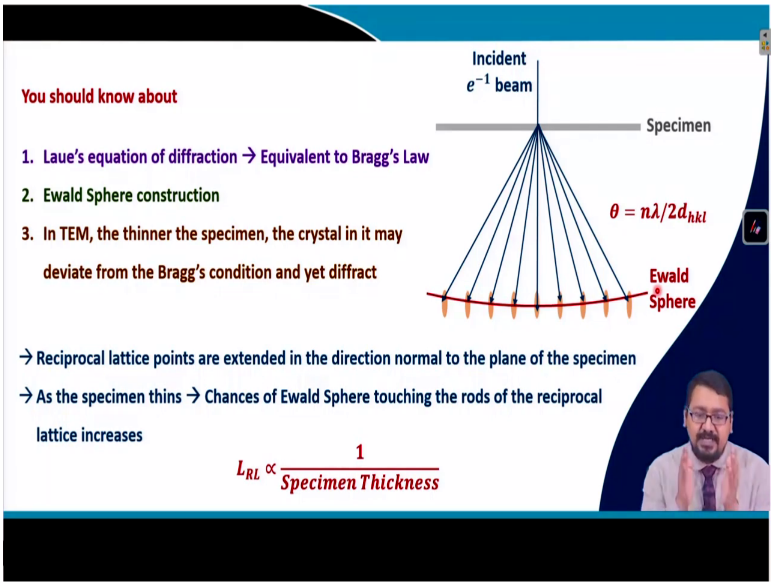

So, thinking that what you should know about while we are doing this course. So, we should have an understanding of the Laue's equation of diffraction which is basically equivalent to the Bragg's law because it is important for electron diffraction.

The 2nd thing and the most important thing is to understand the Ewald Sphere construction and you see that what is an Ewald Sphere. An Ewald sphere is a sphere where the lattice you know reciprocal lattice spots or the poles should interact in order to get the diffraction.

And you see when the while doing transmission electron microscopy, the if the specimen is made thinner and thinner the crystal in it may deviate the Bragg's condition and it will yet diffract and why it is so, because you know the as it is becoming thinner and thinner the reciprocal points or the reciprocal spots or the reciprocal poles of those h k l planes can be it gets extended in the direction normal to the plane of the specimen or it gets extended along the direction parallel to the electron beam.

And then it more likely to get you know intersected by the Ewald Sphere. So, as the specimen gets thin the chances that the Ewald Sphere touches these you know reciprocal lattice poles which are now looking like rods which long rods and therefore, it gives the diffraction right in a thin specimen in a more proper manner.

So, you see the LR is the length of those rods or the lengthened reciprocal lattice spots lengthening is along elongation is along you know perpendicular to the plane of the specimen or along the electron beam is basically inversely proportional to the specimen thickness.

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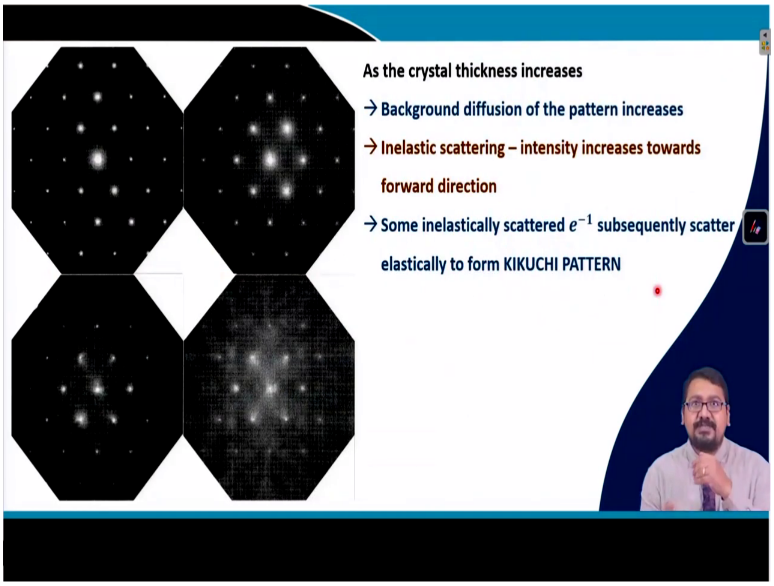

So, on the other hand. So, we can get a spot which looks like this and we can get a large amount of spot which from which we can you know observe that what crystal structure it is, at what zone axis we can rotate the specimen holder and put it in a certain zone axis and observe the diffraction spot and find out information about it like whether the material is nano crystalline or it is poly it is larger grains or it has some deviation in the crystal structure like that.

But you see as the on the other hand as the crystal thickness is increased. So, while the crystal thickness is increased, the background diffusion of the pattern starts to increase. So, you see this is for the thinnest specimen and then the specimen thickness is slightly more and you see there is a background diffusion. You see the it has become you know the beams have become slightly spread out right and there are slight diffusion also in between them.

Now, with the further increase in the you know thickness of the more you know this kind of diffusion will take place. Now why this kind of diffusion in the spot patterns are taking place? Because you see the thicker the specimen the number of times the scattering is taking place between the incident electron beam and the electrons and then and the you know electron cloud present in this specimen will keeps on increasing.

So, if the specimen is thin then the scattering is mostly elastic with no detectable loss of energy while the specimen is getting thicker and thicker, the you know the electron beam is scattering several times and then it will get I mean there will be a loss of energy a detectable amount of energy is lost and then it becomes you know multiple scattering or inelastic scattering.

So, finally, you see when the thickness is large this type of you know spots they starts to form like you know slight you know diffused kind of bands could be observed. You see in this case also diffuse kind of bands could be observed and this happens because when multiple scattered electron multiple times scattered electron or in a inelastically scattered electron.

Finally, scattered one single time before leaving the specimen and that scatter should follow the Braggs law or the Laue's equation so, that it diffracts at that particular angle as per the Laue's equation or the Braggs law so, that it can be said diffraction, then it can it will form you know subsequently it will form a Kikuchi pattern.

A Kikuchi pattern which is like a you know band or a line instead of you know spots. So, you see these bands that are forming they are basically formation of Kikuchi pattern which can be observed when the samples gets thicken and thicken in a TEM in a TEM.

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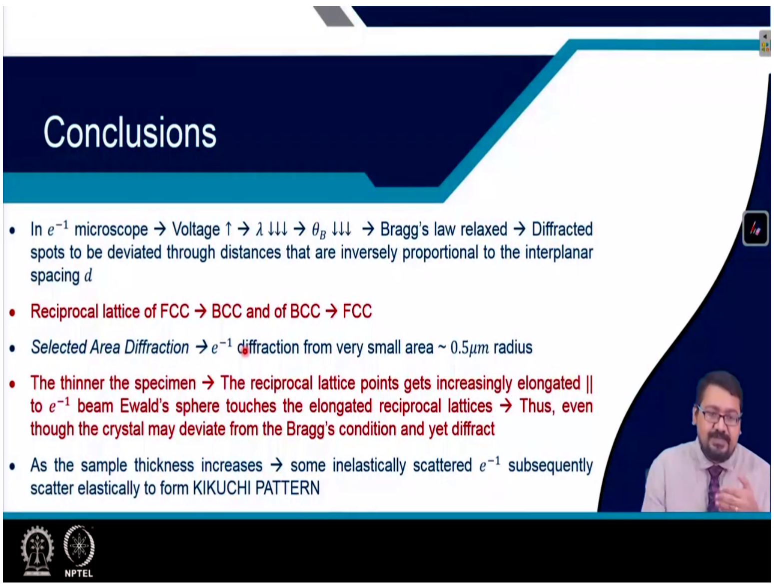

So, what we can conclude? So, the conclusions are in the electron microscope as the voltage is increasing, the lambda is decreasing right and as the lambda is decreasing the theta following Laue's equation or the Braggs law is decreasing. Simple as it is the Bragg's law becomes relaxed the diffracted spots will be deviated through the distance that are inversely proportional to the interplanar spacing of that particular you know crystal structure.

So, reciprocal lattice of you know face centered cubic is basically body centered cubic and body centered cubic looks like face centered cubic this is also a thumb rule. So, a diaphragm is kept in between the specimen and the electron beam so, that it can irradiate only a small area say 0.5 micron and then one can get a selected area diffraction and the diffraction spot.

So, thinner the specimen you see the reciprocals as I said the Braggs law is relaxed and the reciprocal spot becomes increasingly elongated parallel to the electron beam or perpendicular to sphere you know touches very easily that elongated reciprocals poles and thus even though the crystal may deviate from the Braggs condition or the Laue's equation condition it will yet diffract.

Lastly, as the sample thickness increases some you see multiple scattering or inelastic scattering will takes place, subsequently there will be final elastic scattering where the scattering takes place following Bragg's law and it comes out of the sample forming band type of structure instead of spot which is known as Kikuchi bands or Kikuchi patterns.

We should remember here that when Kikuchi pattern we are talking about the electron beam is falling it is not only diffracting from the single h k l plane to form Kikuchi pattern, it will be diffracting from a multiple planes ok to form bands which are intersecting each other we will talk about this later; that means, several h k l planes are being diffracted to form several Kikuchi bands leading to form multiple Kikuchi bands in a phosphor screen and that is what we call it a Kikuchi pattern.

Thank you very much.