Fundamentals of optical and scanning electron microscopy
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Module – 03
Unit-7 instrumental details and image formation
Lecture – 20
Electron channeling contrast imaging (ECCI)
Electron back scattered diffraction (EBSD)
- Theory and instrument demonstration

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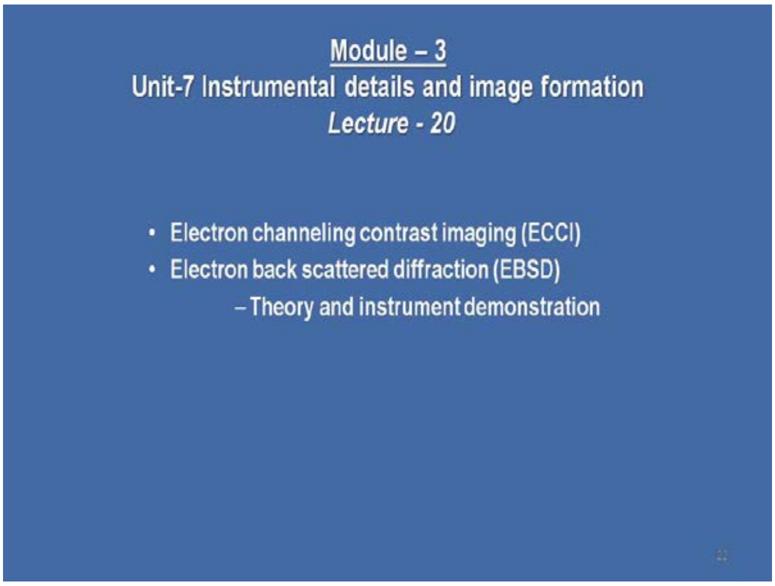

Hello everyone welcome to this material characterization course. In last class, we have just discussed about some of the special contrast mechanisms that is operating in SEM, namely the electric field contrast, in terms of voltage contrast and then magnetic field contrast. And then we also gone through a couple of examples, where these contrast mechanisms can be realized under the SEM. Today we will discuss two more some of the special contrast mechanisms or which will come under the special topics of scanning electron microscopy namely electron channeling contrast as well as electron back scattered diffraction.

This electron back scattered diffraction itself is a very popular technique these days for the characterization of microstructure as well as the quantification. The subject is quite vast and it is becoming very specialized these days, but for the sake of the completion, I will briefly discuss about the principles behind it. And also we will show some of the lab demonstration how this things are done in a much more brief manner, so that you will have some kind of idea about what is this EBSD is all about.

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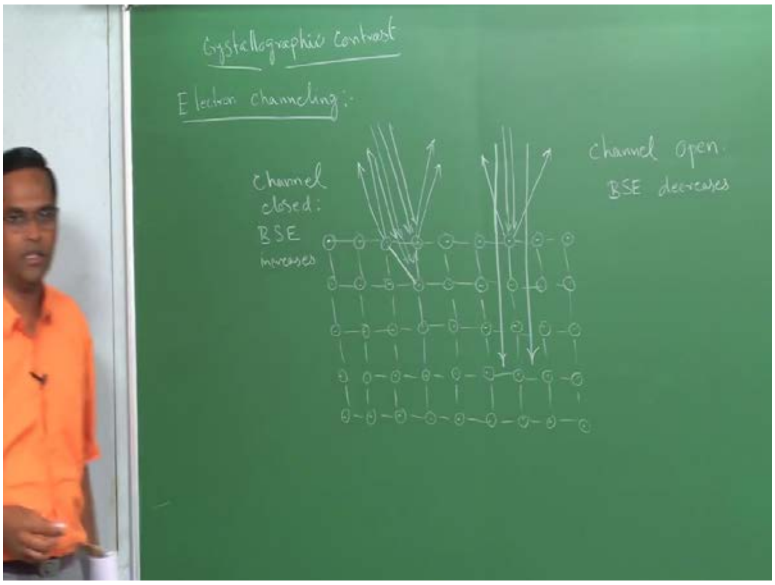

So, first we will briefly discuss about what is this electron channeling contrast. For that, I use the saw the schematic on the blackboard. It is also called a crystallographic contrast. So, what I have drawn here is a representation of a 3 D lattice in 2 D. Suppose if you have this is the electron beam, which is coming and falling on this, where the electron beam travels all the way a deeper inside the crystal. And here the electron beam is being stopped by this kind of a randomness. Suppose, if you assume that this is a kind of ordered alloy or ordered systems like this in relative to the amorphous, amorphous is always kept as a reference.

Suppose if the electron beam is able to pass through this path where you have a very least resistance; that means, the density of the atomic path is less here as compared to here. So, then the electron beam can travel all the way inside the crystal and then the chances of coming back that is the BSE electron after scattering is less. Whereas, if it is stopped in the surface itself then the probability of getting the BSE that is in terms of yield is more here. So, you have the difference in the contrast. We will write few remarks about it then we will move on to this explanation.

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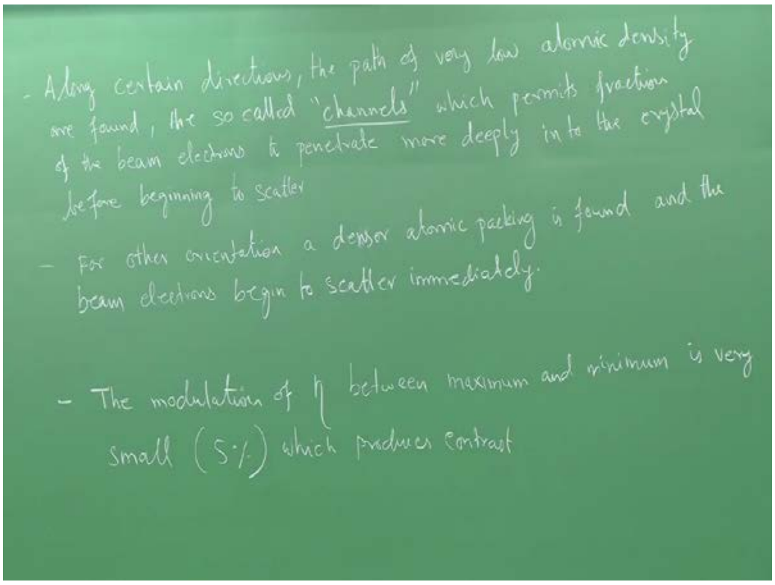

So, what I have written here is along certain directions the path of low atomic density are found something like this, the so called channels which permits the fraction of beam electron to penetrate more deeply into the crystal before beginning to scatter. That means, after reaching this point only, the scattering even starts then the BSE signal or SE signal will come out of this that kind a signal will have very low yield, that is the eta value will be lower. And on the other hand, if some other orientation where the denser atomic packing is found and the beam of electrons begin to scatter immediately something like this, where you do not have a clear channel here, the electron beam starts scattering from the surface itself which promotes the BSE yield.

So, these two produces the difference in the signal produces the contrast and if you see that, the modulation of eta that is back scatter electron yield between the maximum and minimum is very small, this not very bit number here, it is within the contrast difference is only 5 percent which produces the actual the image.

So, though it is not very powerful in terms of producing the contrast, but still it is being use sometimes and produces a electron channeling pattern something similar to back scattered pattern EBSD which we are going to discuss now. So, this is also one of the imaging contrast so called crystallographic contrast under the SEM.

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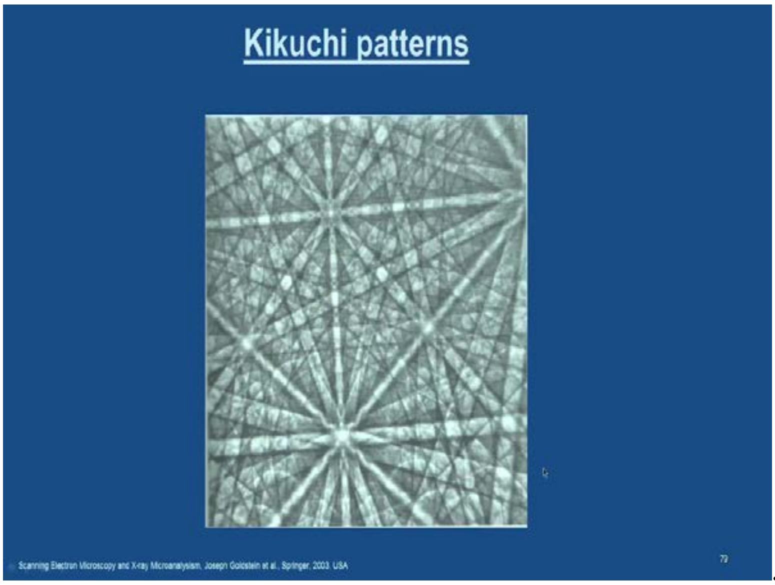

Now what we will do is, we will drive our attention to another important imaging technique called EBSD. So, this EBSD pattern will look like this. It is also called a Kikuchi pattern you will see the bands of bright and dark line pairs. And we will now see the some of the basics about this image formation. And one of the primary use of this EBSD pattern is to analyze the microstructure in terms of crystallography and grain orientation and so many other parameters are measured through this technique, and it is powerful, and it is becoming popular and popular these days. And we will just go through the basics of this technique very briefly.

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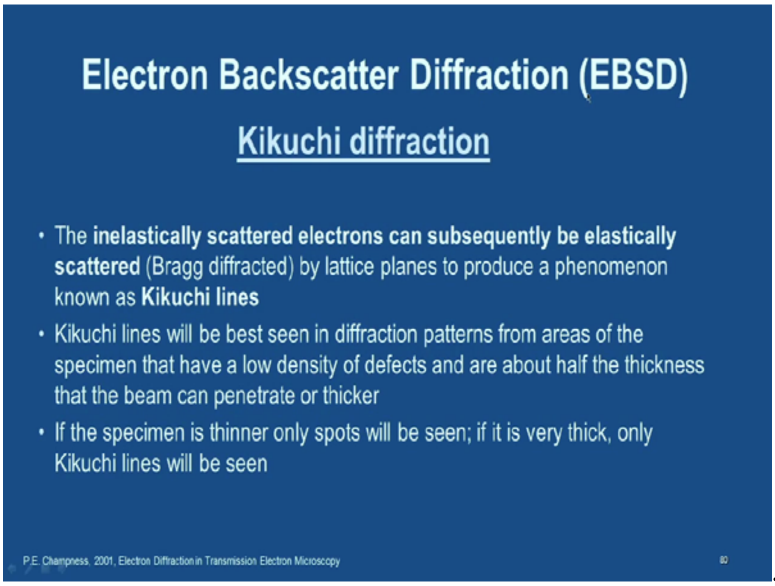

So, this electron back scattered diffraction - EBSD is also called Kikuchi diffraction. The inelastically scattered electrons can subsequently be elastically scattered that is Bragg diffracted by lattice planes to produce a phenomenon known as Kikuchi lines. So, you see all these signals whatever we get from this SEM is because of inelastic scattering, and when the inelastic scattered electrons subsequently subjected to elastically scattering or you say Bragg diffraction by the lattice planes, which produces the Kikuchi lines.

And Kikuchi lines will be best seen in diffraction patterns from the areas of the specimen that have a low density of defects and are about half the thickness that the beam can penetrate or thicker. You need a thicker sample. And if the specimen is thinner, only spots will be seen; if it is very thick, only Kikuchi lines will be seen. Of course, this is with respect to some of the transmission mode. We will also discuss this when we go to the appropriate section.

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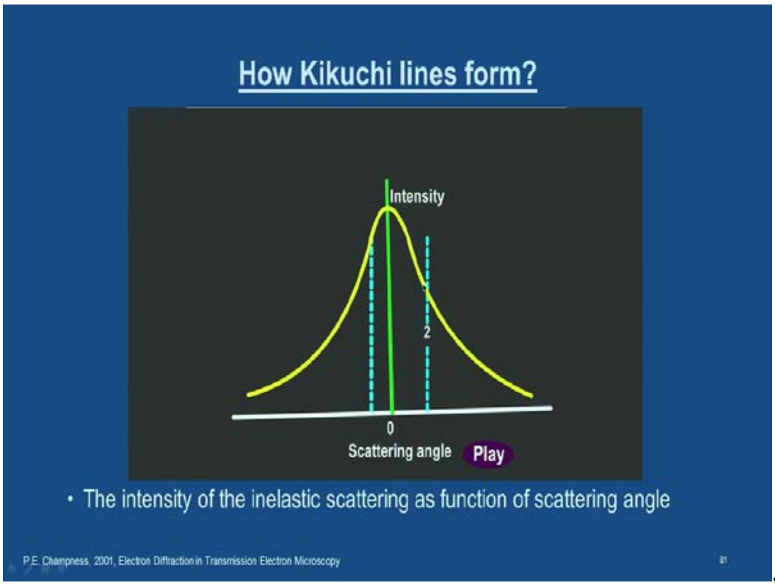

This is how it is been interpreted, how Kikuchi lines are forming. So, this is a intensity of the inelastic scattering as a function of scattering angle. So, what I have just shown here is two lines; this is one reference 1, and this is reference 2. Let us consider these two rays. So, compare to 1 and 2, the ray one has a forward scattering, in fact, you can see that the intensity of the ray one is much higher compared to the intensity of the ray two. So, you keep this is mind, then we will look at the next animation to understand this better.

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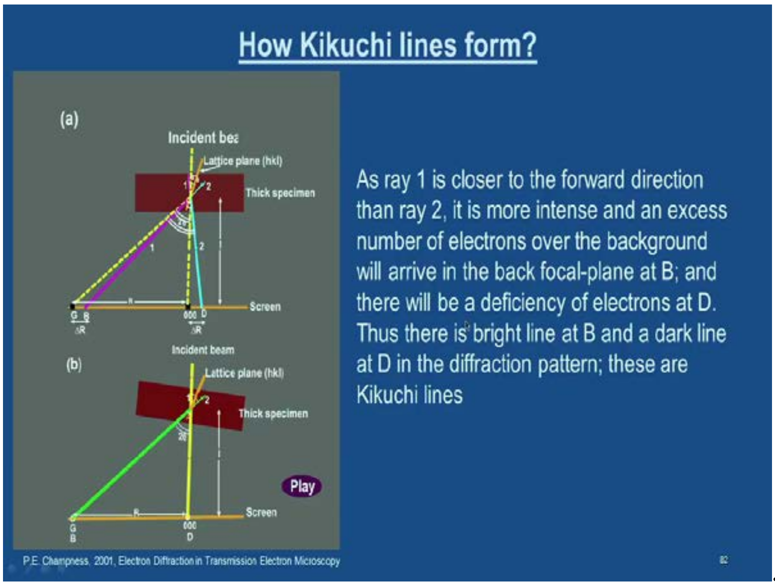

What you are now seeing in the schematic is the specimen. This is an electron beam which is falling, and this is the transmission axis. And I will just play this animation just closely observe this it is a thick specimen and this is the screen and inside the specimen we consider the lattice planes. And the ray which I mentioned as 1 and 2 are here. And as the ray 1 is closer to the forward direction then the ray 2, it is more intense and an excess number of electrons over the background will arrive in the black focal-plane at B. So, this is please understand, all this diffraction takes place in the back focal-plane in which you all you know. So, here the ray 1 which I am talking about is this ray; so obviously, compare to ray 2 this is this is more intense, because you can see that compare to this point this point will have higher intensity.

So, the excess number of electrons over the background will arrive at the back focal-plane at B here and there will be a deficiency of electrons at d. So, you are talking about an electron diffraction which is forming a kind of a cone, we will just see what is this cone which I am talking about. And what you have to understand is one ray with excess electron are high intensity falls in the black focal-point B and the deficient line will fall here. And there is a bright line at B, and a dark line at D in the diffraction pattern, and these are all Kikuchi lines. So, you can see that go back and look at this pattern again, a bright and a dark line which is coming a parallel line is because of this diffraction effect.

We will understand this little more now. So, once the crystal is rotated little bit then everything falls in the ray 2 falls within the optic axis the ray one falls with the diffraction spot.

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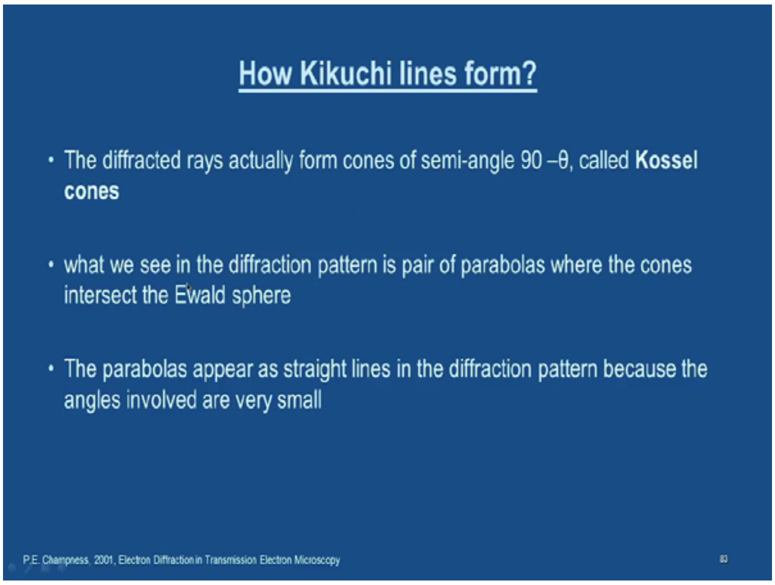

The diffracted rays actually from cones of semi angle 90 minus theta called Kossel cones. The cone which I am talking about this in a 3D, it will appear as a cone, I will show you one more schematic you will appreciate that. What we see in the diffraction pattern is pair of parabolas where the cones intersect the Ewald sphere. The parabolas appear as straight lines in the diffraction pattern because the angles are very small. You see in an electron microscopy, we just discussed in the fundamentals that you can within the increase acceleration voltage your alpha can be reduced or controlled to very small value, and because of that you can see this.

One of the primary difference between an x ray diffraction and electron diffraction, if you recall, if you probably if we go and go back and discuss about these fundamental principles on Ewald sphere, you will appreciate this. And if you are not able to pick up this at this. So, the plus or minus g pair of lines and the region between them is known as Kikuchi band. Their angular separation of the pair of lines is 2 theta their spatial separation in the diffraction pattern in the black focal-plane is g and lines are perpendicular to the g-vector. Each reflection has an associated pair of Kikuchi lines attached to it.

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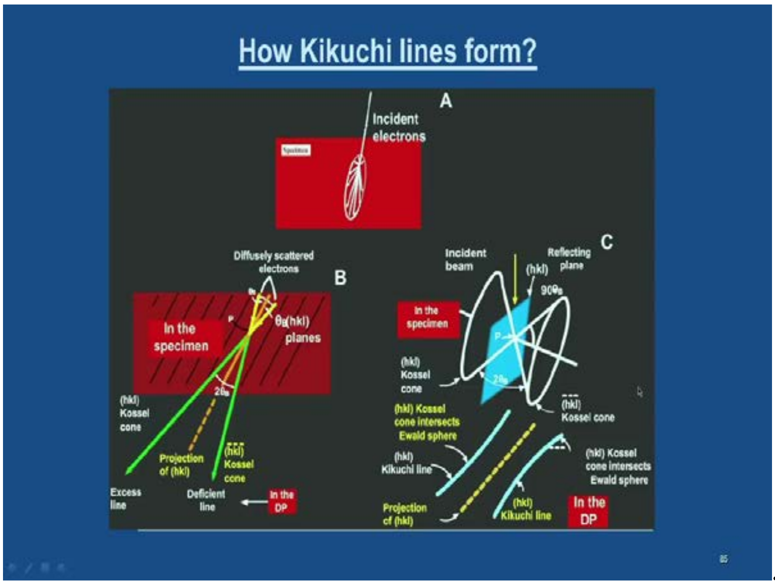

So, this is a schematic you can look at it, and you can appreciate what we are now talked about. So, you have the specimen here. The incident electrons come and interacts and they are subjected to diffraction. Suppose if you consider this sample is so thin, and then if you look at the three dimensionally the electron beam which falls it produces a cone like this. It is a projection here; it is actually a three-dimensional cone.

For each plane if the cone is produced on both sides, so one these are called Kossel cones. And when these cones are intersects the Ewald sphere or what actually we are looking at is only this parabola, because it is a only the intersection of this cone on a two dimensions is appears which appear like this. You can see that this is the Kossel come intersects Ewald sphere here. And this side is also the other cone will intersect all these pattern is appearing in the diffraction pattern that is why we discuss it is called DP.

See, if you look at, if you assume this and then come back to this diagram what we have just discussed, for the convenience we can imagine it like this in a 2D, this is the a specimen you have this hkl planes where the electron beam comes, and then it produces the cone here. The one we talked about in excess line; another it is deficient line intensity and the angular measure between these two line is 2 theta b, 2 theta b because of the Bragg diffraction and then you can see that the deficient line will appear dark and the excess line will appear bright. And again you may wonder that since it is a very flat cone and the theta is so small here for the same reason; actually the parabola in all practical purpose it appears a straight line in the electron diffraction pattern that is EBSD pattern that is because of the very, very small alpha, which you experience in the electron microscope.

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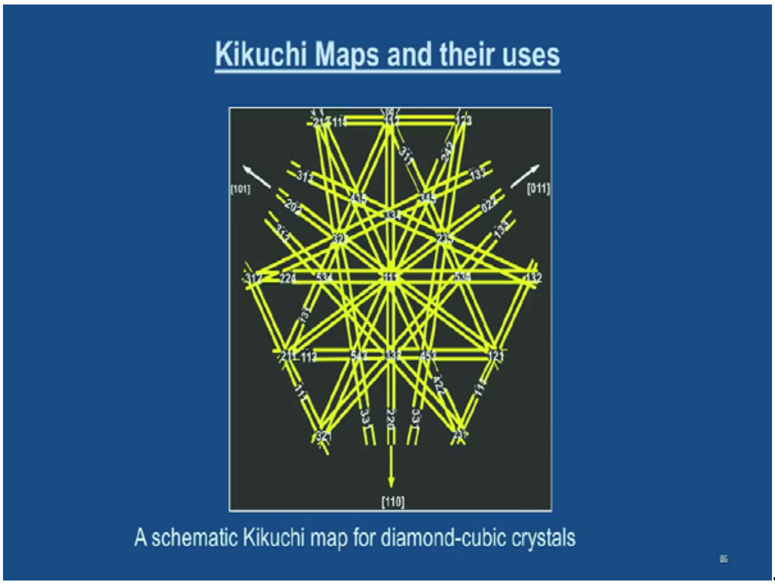

So, this is the typical a schematic of Kikuchi map for a diamond cubic crystal. So, we will just see that some of the applications of this. As I mentioned that you can map the grain orientations and orientation mapping and then you can identify the phases and you can quantify all the micro structural parameters. We will just show you some glimpses of all this, if not in detail.

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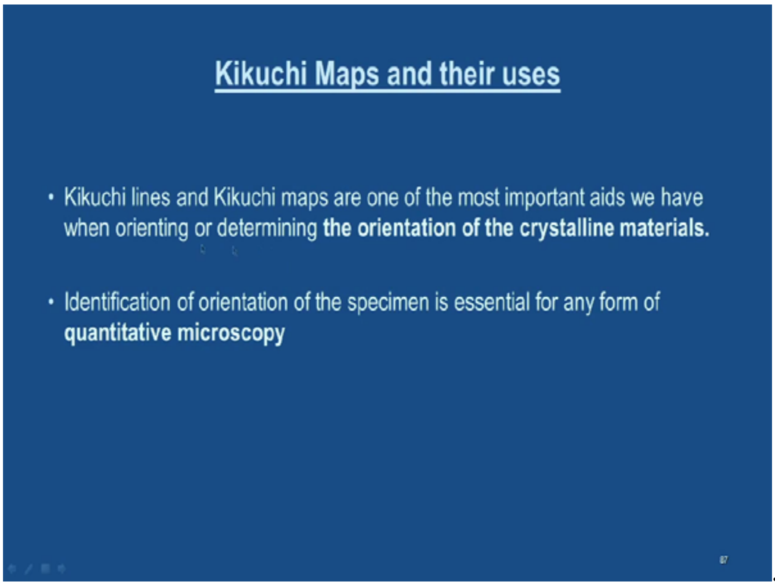

So, the Kikuchi lines and Kikuchi maps are one of the most important aids we have when the orienting and are determining the orientation of the crystalline materials. Identification of orientation of the specimen is essential for any form of quantitative microscopy. See this is one major application here quantification.

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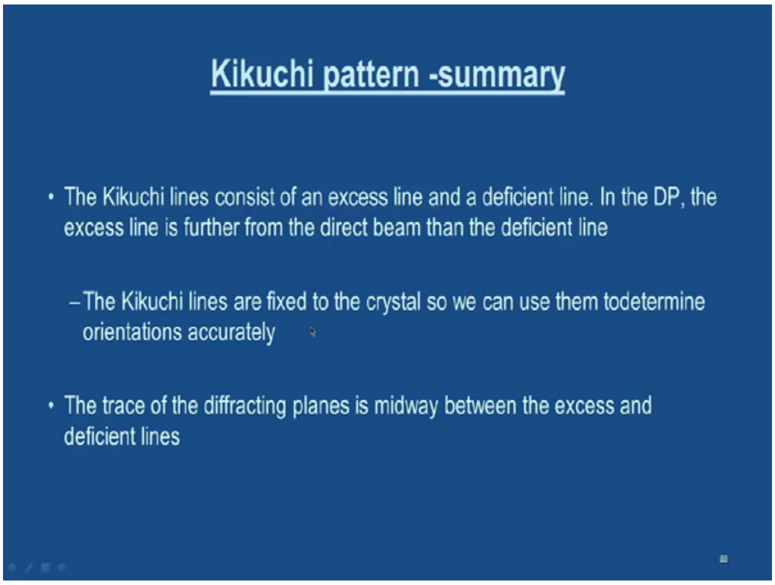

If you can summarize this Kikuchi lines consist of an excess line and then a deficient line in a diffraction pattern. In the DP, the excess line is further from the direct beam than the deficient line. The Kikuchi lines are fixed to the crystal so we can use them to determine orientations accurately. The trace of the diffracting planes is midway between the excess and their deficient lines. So, for time being, you just try to understand this with a simple diffraction phenomenon by looking at this schematic. Now we will just go to the laboratory demonstrations where we will actually look at some of the samples which is being loaded in the SEM.

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So, this is a sample which is loaded in the specimen stage, and then you can see that the specimen stage is tilted to about 70 degree, so then only you can produce that very flat cone and then alpha can be very small. And you can see that the camera just came that EBSD camera just came, and this is your pole piece what you are just seeing is a pole piece, and this is the sample which is kept at angle of 70 degree, and the camera is come very close now. Now we will see how the Kikuchi map is generated with this sample. What you have to do is the one of the primary requirement of producing EBSD sample is the very fine polish, which is very difficult which is done by this electrolytic polishing. And you first generate a secondary electron image of the sample.

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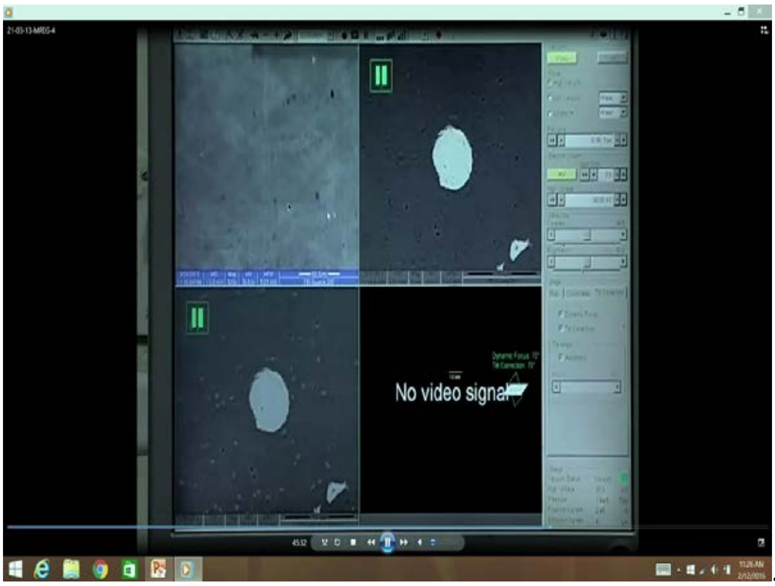

So, now the secondary electron image is getting focused. So, you can seen that some of the feature start appearing. This sample is being investigated by one of our scholar for his PhD thesis, Mr. Devender. Now we will demonstrate that EBSD patterns which is obtained from this sample. Normally what happens is once you obtained in a secondary electron you just grab it on another screen where the orientation microscopy software called TSL, which handles this EBSD analysis.

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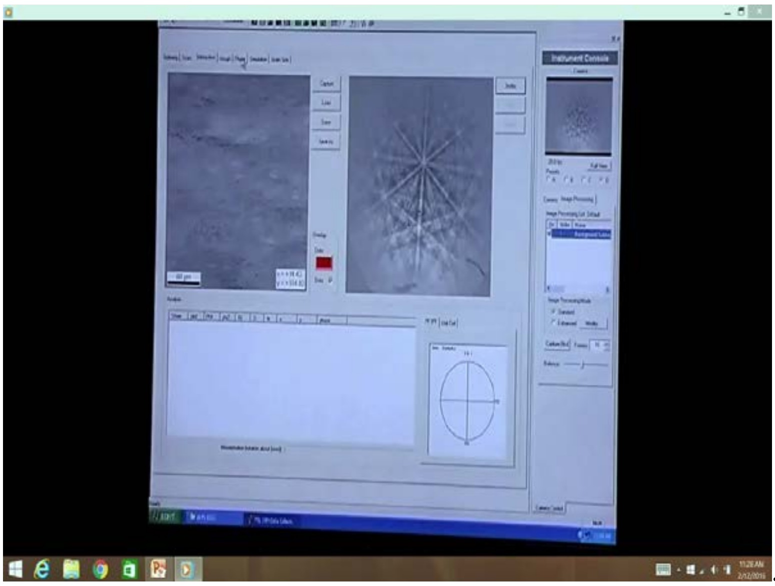

So, the now what happens is the beam is connected to directly connected to I mean synchronized with your mouse. So, wherever you put the cursor on this sample, and then click, then the corresponding Kikuchi lines are generated here at each point. These information is coming from the sample about 20 nanometer thickness. So, you have to be very careful about this aspect when you talk about representation of the bulk texture or bulk orientation and so on.

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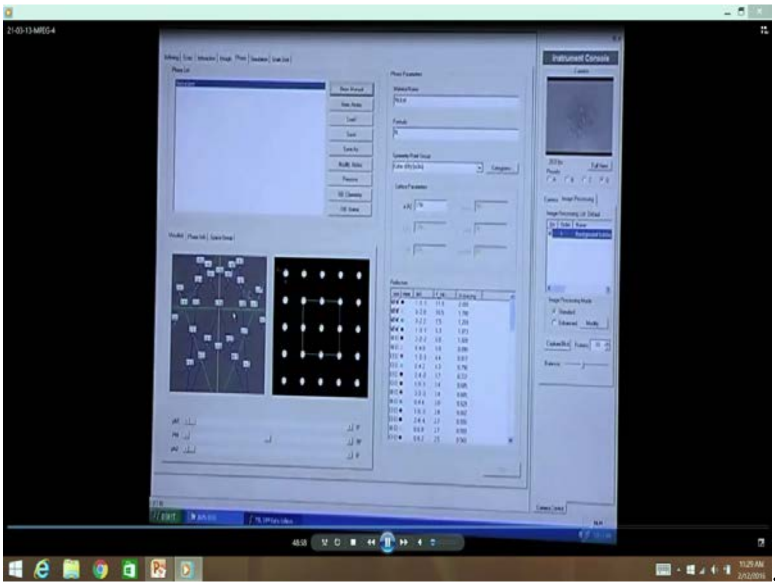

And normally what happens is I will just briefly tell you how this is the analysis done by the software. So, the electron beam just goes and then you can just click the mouse and then it produces the Kikuchi line. If you know the crystal system of the specimen, in this case, it is nickel. So a database belong to this nickel is selected and then the software will generate a orientation which is similar to what is being generated in your sample, and these two patterns are overlapped because this is for example, this is a orientation now the software will super impose this pattern which is very close to this. Because this is already a well known pattern we just already index. So, this will get super imposed and then your actual specimen EBSD data also will be indexed. So, like that each, yeah.

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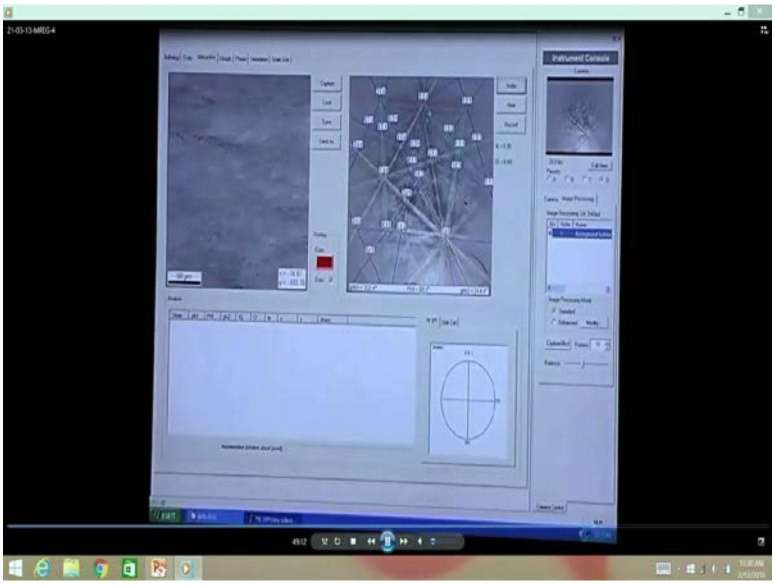

Now, you can see that it is a super imposed with the specimen data. So, now, we can identify some of the zone access like this. And each point, your probe will generate an EBSD pattern like this; and it will record the orientation data, and then you have to select the area under which you want to do this mapping. So, the area is being selected and also the spot I mean the step size, there is something called a step size; that means, under the what are the minimum distance a electron beam has to travel after it a scans one spot or one location, it is just step size.

Here it is a 1 micron is selected; that means, the electron will beam will move 1 micrometer, after it collects one signal that is one data crystallographic data to the another region. So that means, you have to be very careful about this step size; if the step size is on comparison with your grain size, then you will not be able to get the a meaningful crystallographic data. Because at least you are suppose to scan within a grain 2, 3, 4 orientation information should be obtained in order to get a meaningful data. So, your step size is very crucial here.

So, in this particular example, this region is being selected and now te the beam will scan this sample like this line by line. And as I said it will index automatically, and then record it, and it will go back again it will record. So, a typical scan of this range in a normal EBSD, a conventional camera takes about 6 to 7 hours. So, it is a very time consuming process, but today you have a modern recording media where very high speed camera is employed if you have that kind of facility you can reduce this time by one-third. So, this is how the indexing is done. What now you are looking at is in the beam is scanning and it is getting automatically indexed; and finally, it will get recorded.

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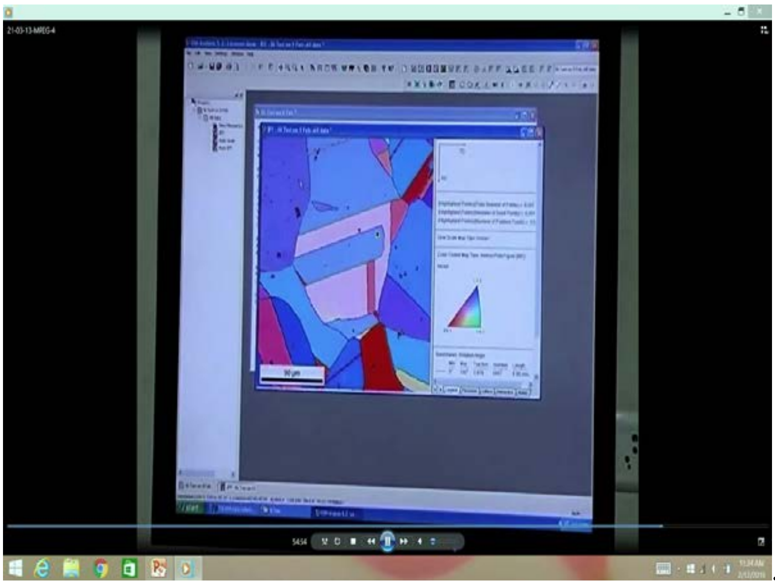

So, what I will do now is since it is going to take long time, I will go to the final result. For example, typically you get this is an inverse pole figure map. See you see a very nice colorful picture like this. So, you have to be very careful in understanding this; each color indicates, it is a orientation mapping. So, be very careful about it; this is not a microstructure this is a orientation map. What is the orientation map. You look at this key here. So, this particular color - blue color belongs to 1 1 1 orientation. This green color belong to 1 0 1 orientation, and red color belong to 0 0 1 orientation. So, the each color indicates the whole grain orientation belong to this particular number, so that is what it means.

And another important thing we can do is see what this scholar is trying to do is to look at the mis orientation between these two grains. See what he has done is he has just taken the cursor and then drawn this a line here between these two lines these, two boundaries you can see that the mis orientation angle between these two is about 60, so that he confirmed this as a twin. So, you can ready readily understand the mis orientation between the two boundaries. So, these boundaries are characterized as twin boundaries. And on the other hand, if you do a scan here and then this is only about 30 degree; so definitely, it is not a twin boundaries.

So, these are the very, very powerful tool to determine the grain orientation instantaneously, and you can do a lot more calculations like you have the orientation spread and then you have the mis orientation distribution, you can mis orientation distribution also we can see from this sample. So, like that you have all this a very useful quantitative information can be obtained from this technique.

And another very important aspect is like you can also look at the surface textured information. Yeah, so this is a pole figure which also shows the texture within this top and 20-nanometer layer of the sample. And it shows kind of random texture here; it is not showing any a particular texture. And we will show you some of the sample where it exclusively shows a very nice texture. And you can also look at the quality maps like this, and some of this iq maps that is quality maps also widely used in some of the a breaker sized grains and deform grains and so on. I am just giving you a very glimpses of it; I am not getting into the details.

So, just basically, I am just highlighting the usefulness of this EBSD technique. And finally, I would like to show some of the a sample which is exhibiting a very strong texture which I want to give you one example. So, let me go back and take one more shot. So, look at this map, where it shows mostly a 0 0 1 orientation that means, most of the grains are oriented towards 0 0 1 orientation. So, if you take a pole figure, then it will clearly show you the cube texture - 0 0 1 texture. This is the one classical example you can see how the a cube texture is shown.

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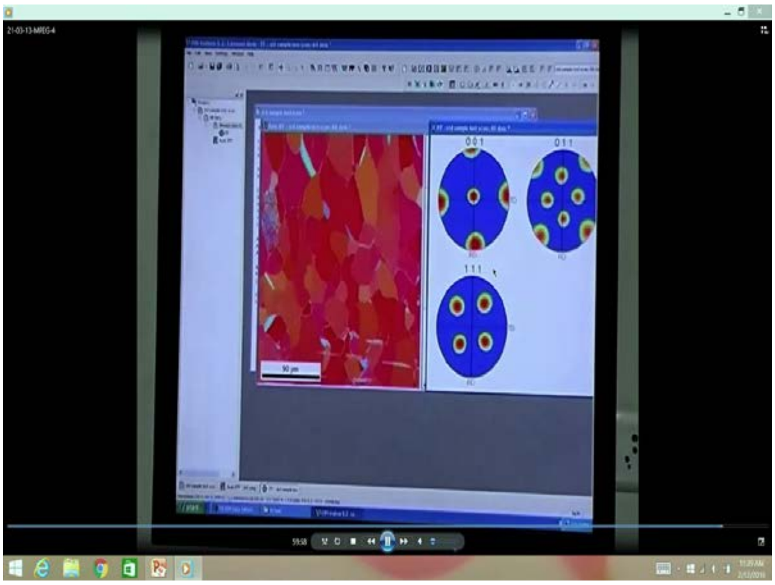

So, this is very nice a pole figure shows a cube texture 0 0 1. Any material, which exhibits cube texture will show the pole figure of this kind of three different orientation here. So, in this case, this is a nickel sample, where a student processed it to obtain this cube texture. And again I am telling you this is this information is coming from the top 20-nanometer the surface layer. So, if you really want to do it or prove it as a material property, you may have to do it in a x-ray texture, EBSD is not a characterizing the bulk behavior in the sample so that point you have to be very careful; other than that it is very useful to characterize this.

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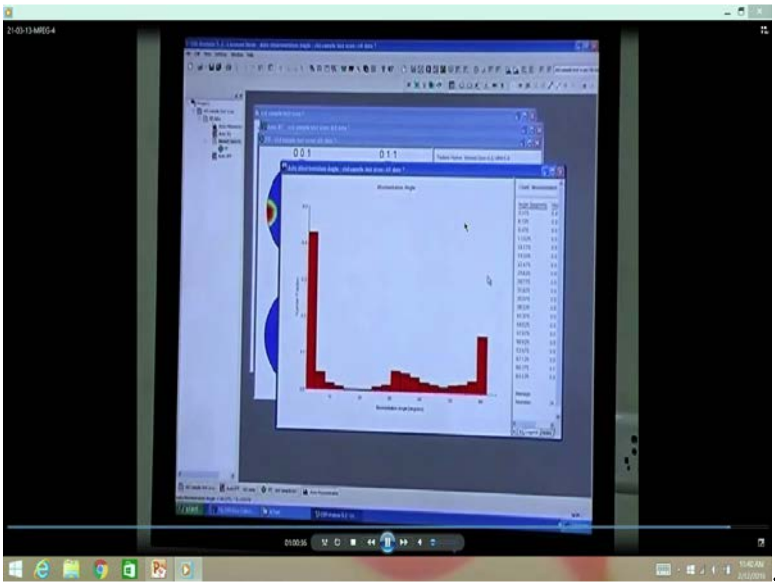

Again this you can see that mis orientation angle. You can see that a low angle and high angle boundary distribution, which is readily available using this a software interface.

So, I think, we will stop here. What I would like to say is so as a whole we have now gone through a number of concepts involving on SEM; apart from the conventional imaging technique like scanning I mean secondary electron imaging or back scatter electron imaging, and we have also very briefly introduce the special contrast mechanisms. And at this particular technique, just we have just shown, I have not gone into the details for the lack of time constraint, but then EBSD itself a separate course one can go through to get into all the details.

But as a part of this on SEM course, I think whatever I have just shown is I hope it is useful to realize that is one of the powerful tool which gives about a crystallographic information and so on. And with that, I will finish all this discussion on the scanning electron microscopy. And in the next class, I would like to do some more tutorial problems, and you can just go through those tutorial problems, and you get back to me whether you have any doubts.

Thank you.