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Lecture-20
Materials Characterization
Fundamentals of Scanning Electron Microscopy

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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 ACM namely the electric field contrast in terms of voltage contrast and then magnetic field contrast. And then we also had gone through couple of examples where these contracts mechanisms can be realized under the ACM. And today we will discuss two more some of the special contrast mechanisms are which will come under the special topics of scanning electron microscopy namely electron channeling contrast as well as electron backscatter diffraction.

This electron backscatter 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 is becoming very specialized these days. But for the sake of 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. So first we will briefly discuss about what is this electron channeling contrast for that I use the some of the schematic on the blackboard it is also called a crystallographic.

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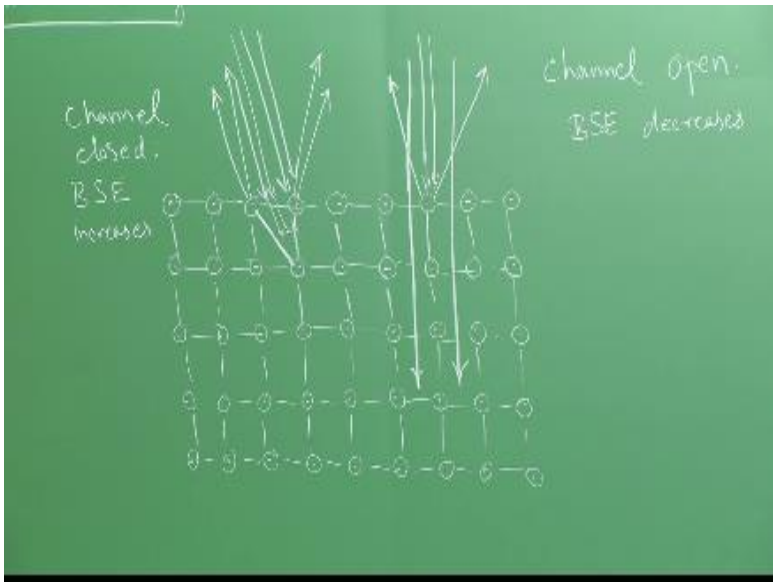

So what I have drawn here is presentation of 3d lattice in 2d. And suppose if you have this is the electron beam which is coming and falling on this. Where the electron beam travels all the way a deeper inside the crystal. And here the electron beam is being stopped by this kind of a random randomness. Suppose if you assume that this is in a kind of ordered alloy or ordered system like this in relative to the amorphous, amorphous is always skipped as a reference. Suppose if the electron beam is able to pass through this path where you have a very least resistance that means L 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 chance of coming back that is that BAC as a BAC electron after scattering is less. Whereas if, if it is stopped in the surface itself then the, the probability of probability of getting the BAC 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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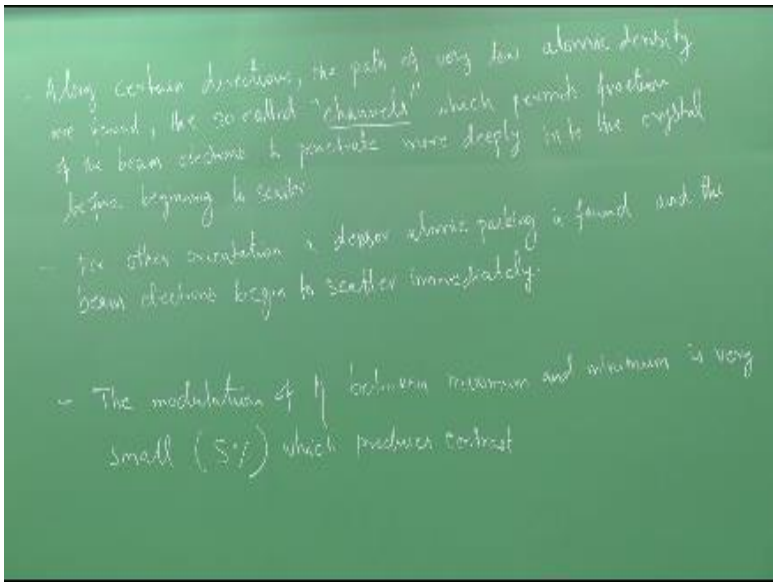

So what I have written here is along certain directions the path of low atomic density are found something like this. These so called channels which permit the fraction of beam electrons to penetrate more deeply into the crystal before beginning to scatter. That means after reaching this point only the scattering even starts then the BSC signal or AC signal will come out of this that kind of 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 start scattering from the surface itself which promotes the BSC yield. So these two produces the, the difference in the signal produces a contrast and if you see that the modulation of ETA that is a backscatter electron ETA between the maximum and minimum is very small it is not very big number here. It is within the contrast differences only five percent which produces the actual the image so, so though it is not very powerful in terms of producing the contrast but still it is being used 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 by imaging contrast so called a crystal graphic contrast under the specimen. Now what we will do is we will, will drive our attention to another important imaging technique called EBSD.

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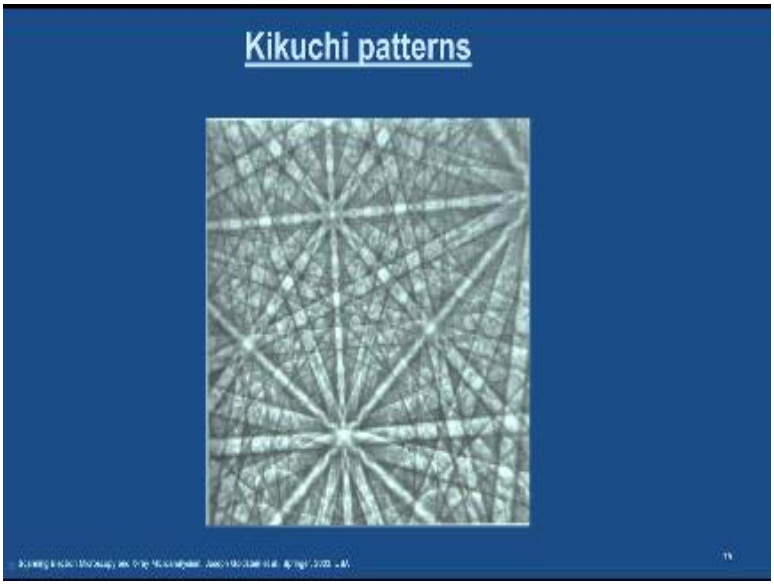

So this EBSD pattern will look like this it is also called a Kikuchi pattern. You will see the, the bands of bright and dark line pairs. And we will now see the some of the basics about this image formation and one of the primary uses of this EBSD pattern is to analyze the microstructure in terms of crystallography and grain orientation and so many other parameters are measured through this technique. And it is very powerful and is becoming popular and popular these days and we will just go through the basics of this technique very briefly if not.

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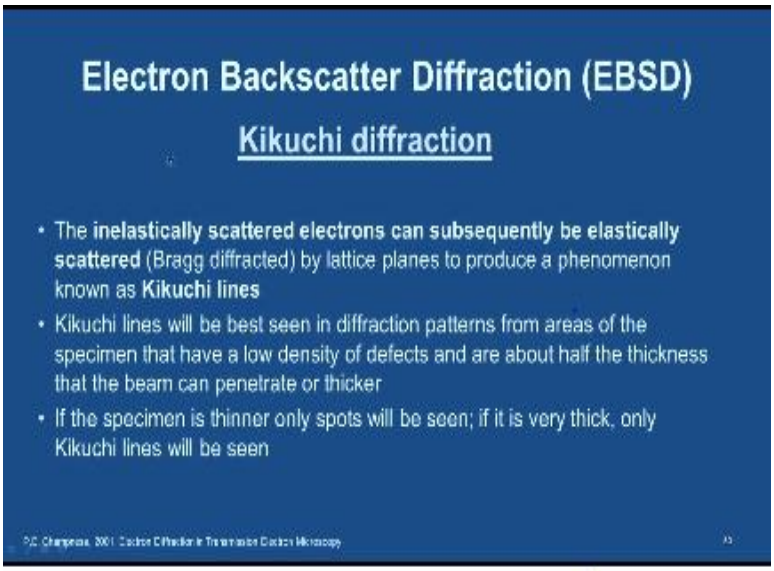

So this electron backscatter diffraction EBSD is also called Kikuchi diffraction the, the in elastically scattered electrons can subsequently be elastically scattered. That is black diffracted by the lattice planes to produce a phenomenon known as Kikuchi lines. So you see all these signals whatever we get from this specimen. This because of inelastic scattering and when the inelastic scattering started electrons subsequently subjected to elastically elastic scattering or you say Bragg diffraction by the lattice planes which produce the Kikuchi lines. And Kikuchi lines will be best seen in the diffraction patterns from the areas of 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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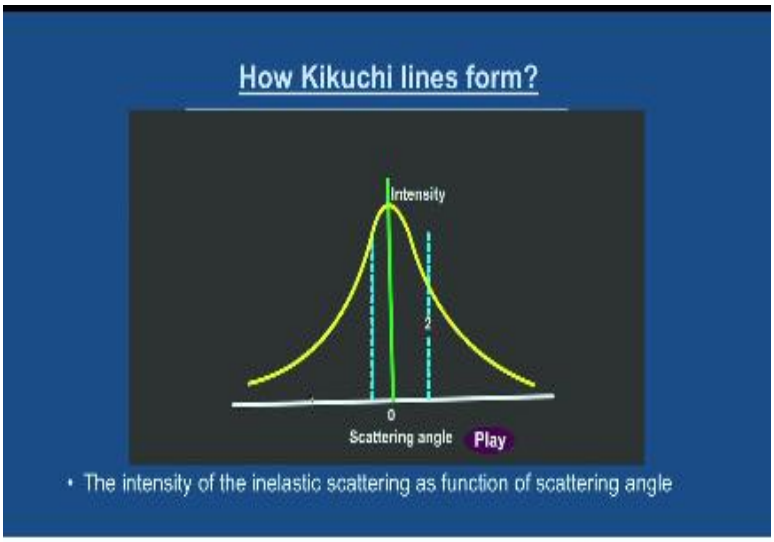

And this is how it has been interpreted how quick you g lines are forming. So this is a intensity of the inelastic scattering as a function of scattering angle. So what I have just shown here is two lines this is one reference 1 and this is reference to let us consider these two rays. So compared to 1 and 2 the, the ray one has as 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 2. So you keep this in mind then we will look at the next animation.

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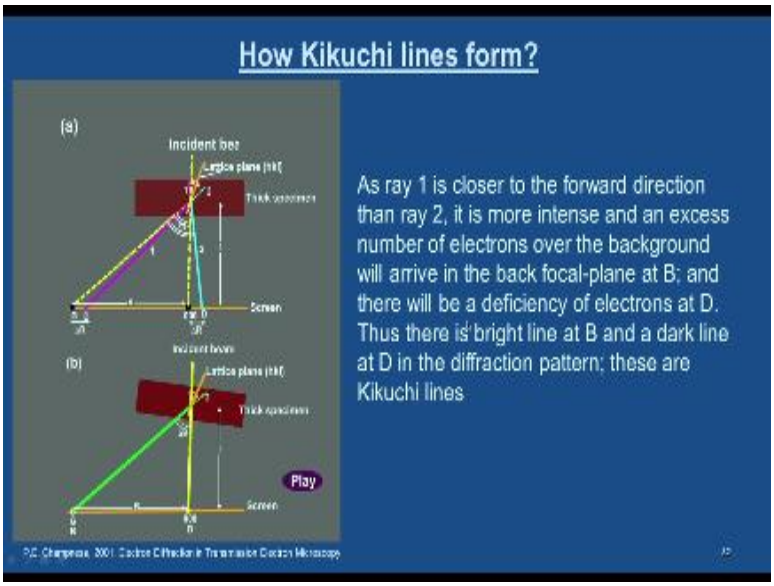

To understand this better what you are now skiing in the schematic is the specimen this is an electron beam which is falling. And this is the transmission axis and I will just play this animation just closely observe this. It is a thick specimen and this is the screen and inside the specimen we consider the lattice planes and the array which I mentioned as one and two are here and as the ray one is closer to the forward direction than the ray two. It is more intense and an excess number of electrons over the background will arrive in the back focal plane at B. So this is please understand all this diffraction takes place in the back focal plane which all you know.

So here the array one which I am talking about is this ray so obviously compared to ray two this is this is more intense because you can see that compared to this point this point will have higher density. So the excess number of electrons over the background will arrive at the back focal plane and be 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 this cone which I am talking about is and what you have to understand is one ray with excess electron or higher intensity falls in the back focal point B and the deficient line will fall here.

And there is a bright line at B at the dark line at D in the diffraction pattern. And these are all Pikachu 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, the ray two falls within the optic axis the ray one falls through the diffraction spot.

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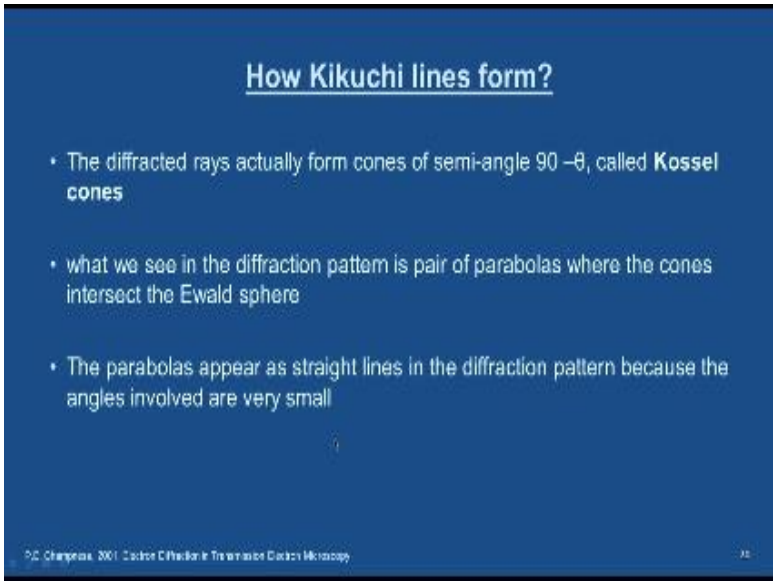

The diffracted raise actually forms codes of semi angle 90-θ called Kossel cones. The code which I am talking about this in a 3d it will appear as a cone where I will show you one more schematic you will appreciate that. What we see in a diffraction pattern is a pair of parabola where the cones intersect the Ewald sphere. The parabola appears as straight lines in the diffraction pattern because the angles involved are very small you see in an electron microscopy. We just discussed in the fundamentals that you can with the increase in the acceleration voltage your α can be reduced or controlled to very small value.

And because of that you can see this and one of the primary differences between an x-ray diffraction and electron diffraction if you recall. The if you probably if we go and go back and

discuss about these fundamental principles on a Ewald sphere you will appreciate this. And if you are not able to pick up this at this.

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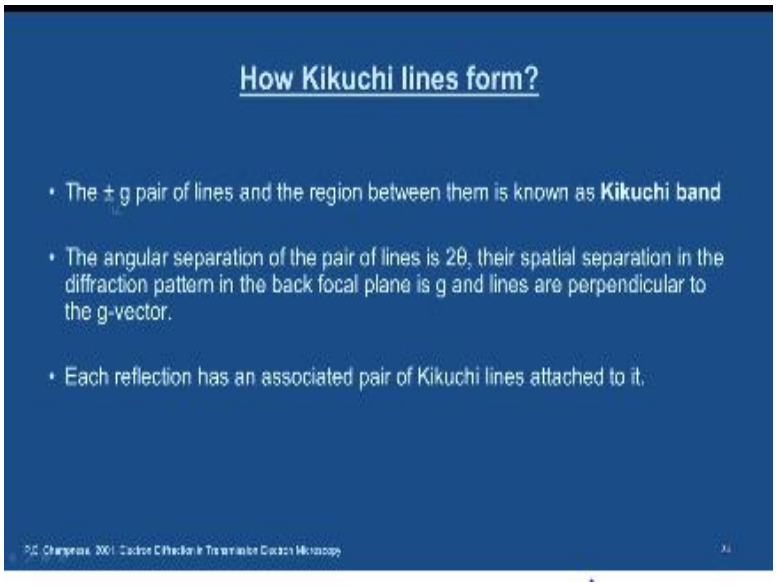

So the plus or minus G pair of lines and the region between them is known as Kikuchi band the angular separation of the pair of lines is 2θ. They are spatial separation in the diffraction pattern in the back focal plane is G and the lines are perpendicular to the G vector. Each reflection has an associated pair of Kikuchi lines attached to it.

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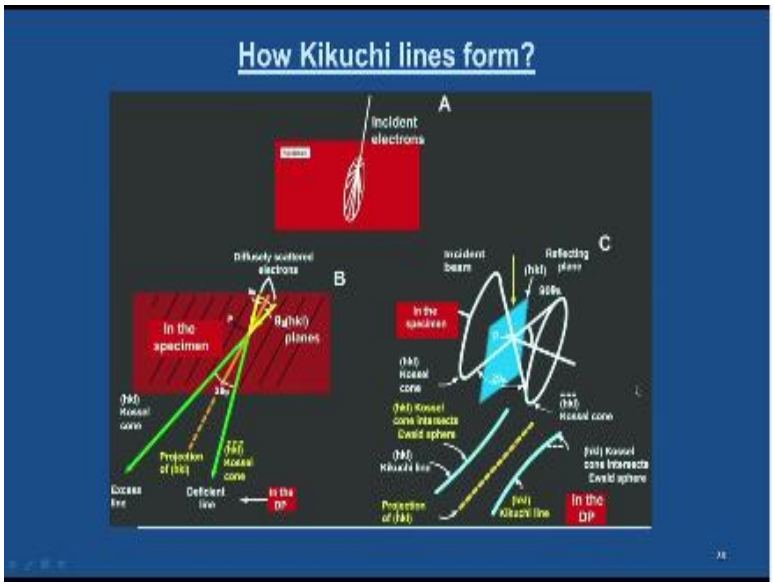

So this is a key metric you can look at it and you can appreciate what we are now talked about. So you have the specimen here the incident electron comes and interacts and they are subjected to diffraction suppose if you consider this sample is so thin and then if you look at the three dimensionally the electron beam which falls it produces a cone like this. It is a projection here it is actually a three dimensional cone on for each plane if the cone is produced on both sides. So one these are all 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 the only the intersection of this cone on a two dimension is appears with which will appear like this.

And you can see that this is a coastal cone in intersects a Ewald sphere here. And this side is also the other cone will intersect all this pattern is appearing in the diffraction pattern that is widely described leaders in BP. 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 specimen you have this HKL planes. Where the electron beam comes and then it produces the cone here the one we talked about nexus line another is a deficient line in intensity and the angular measure between these two line is 2θB, 2θB 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, a very flat code 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 in the electron diffraction pattern that is EBHD pattern. That is because of the very, very small α which, which you experience in the electron microscope.

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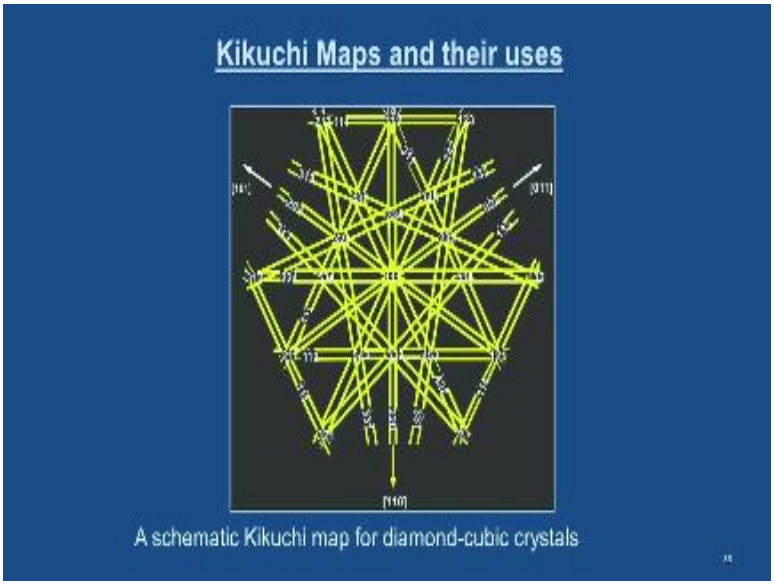

So this is the typical schematic of Kikuchi map for a diamond cubic crystal. So we will just see that some of the applications of this you can as I mentioned that you can map the grain orientations and orientation mapping and then you can identify the faces and you can quantify all the micro structural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

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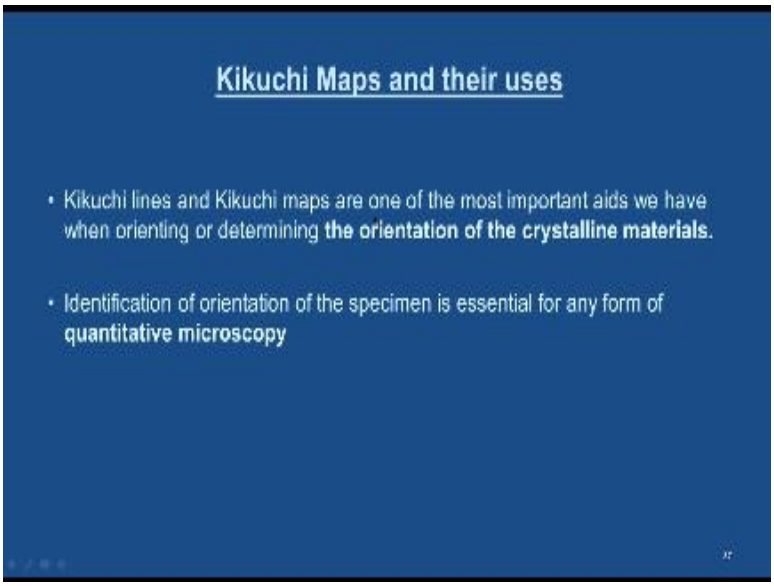

And the Kikuchi maps are one of the most important aids we have when the orienting and are determining the orientation of the crystalline materials identification of orientation of the specimen is essential for any form of quantitative microscopy see. If this is one major application here quantification and if you can summarize,

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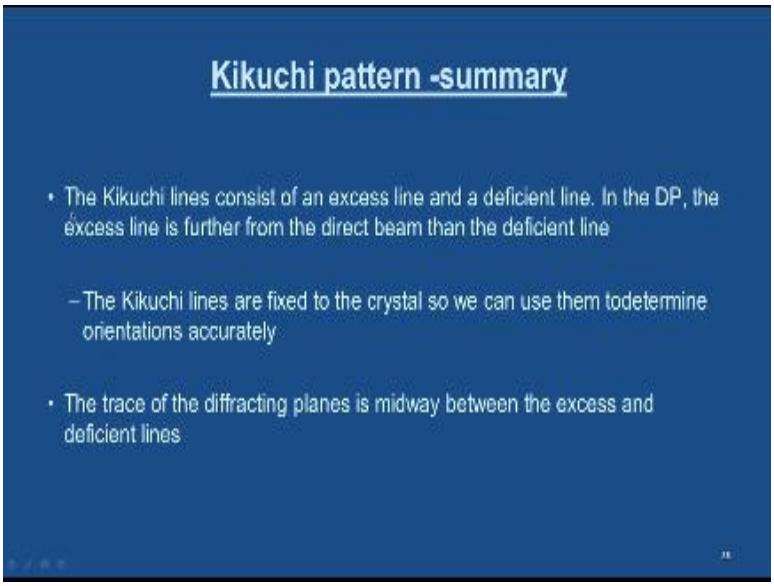

This the Kikuchi lines consist of an excess line and an efficient line in a diffraction pattern in the DP the excess line is for further from the direct beam than the deficient line. The Kikuchi lines are fixed to the crystal so we can use them to determine orientations accurately the trace of the diffracting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

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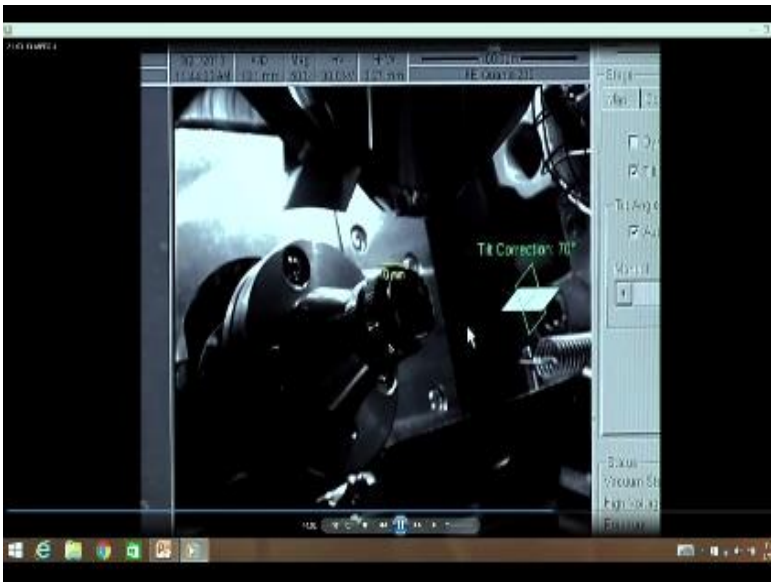

So this is a sample which is loaded in the specimen stage and then you can see that the specimen stage is tilted to about 70°. So then only you can produce that very flat cone and then α can be very small and you can see that that camera just came that EBHD camera just came. And this is your pole piece what you are just seeing is a pole piece and this is the sample which is kept at angle of 70°. And the Tamara has come very close now. And 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.

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Which is very difficult which is done by this electrolytic, electrolytic polishing and you first generate in secondary electron image of the sample? So now the second electron image is getting focused so you can see that some of the features start appearing. This sample is being investigated by one of our scholar for his PhD thesis MR. Devanedar. And now we will demonstrate that EBSD pattern which is obtained from this sample. Normally what happens is once you obtain in a second electron you just grab it on the another screen where the orientation microscopy software called TSL.

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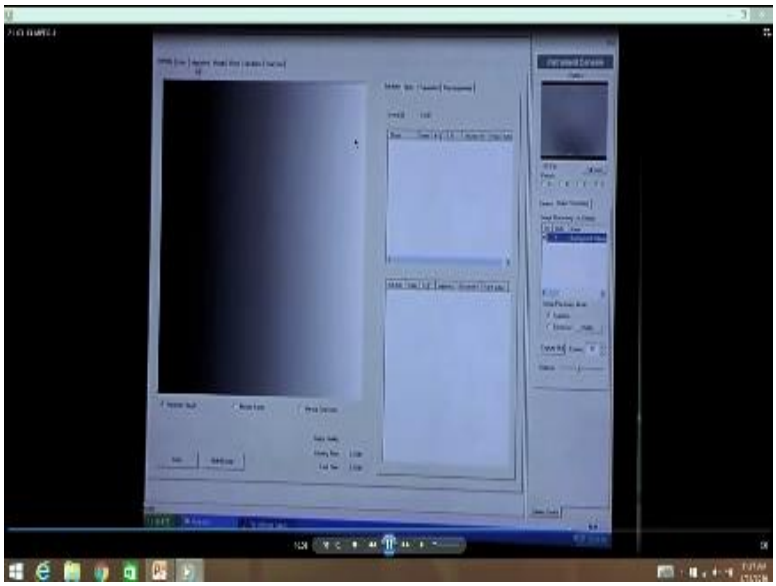

Which handles this II BST analysis so there now what happens is the, the beam is connected to directly connected to I mean synchronized with your mouse. So wherever you put the cursor on the sample and then click then the corresponding Kikuchi lines are generated here at each point. And this information is coming from the sample about 20 nanometer thicknesses. 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 and normally what happens is I will just briefly tell you how this is the analysis is done by this software.

So the electron beam just goes and then you can just click the mouse and then it produces the Kikuchi light and if you know the crystal system of the specimen in this case it is nickel so a database belongs to this nickel is selected. And then the software will generate a orientation which similar to what, what is being generated in your sample and these two patterns are overlapped because this is a for example this is the orientation. Now the software will superimpose this pattern which is very close to this because this is already a well-known pattern. Which is already index so this will get superimposed.

And then your actual specimen EBSD data also will be indexed. So like that the each yeah now you can see that it is a superimposed with the specimen data. So now you can identify some of this zone axis like this. And on each point you are your probe will generate an EBSD pattern like this and it will record the orientation data and then you can 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 what are the minimum distance a electron beam has to travel after it scans one spot or one location.

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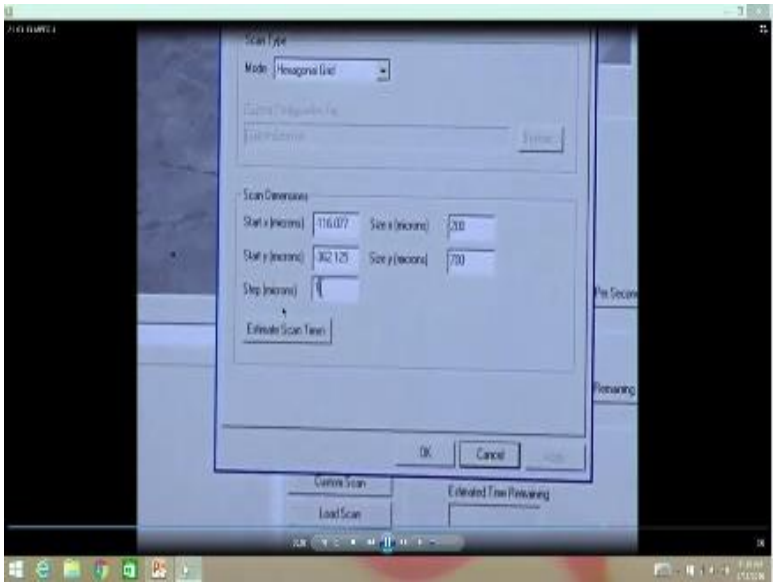

That is just step size here it is one Micronics selected that means the electron bill beam will move one micrometer after it collects one signal. That is one data crystallographic data to another region that means you have to be very careful about this step size if the step size is on comparison with your grain size. Then you will not be able to get the meaningful a crystallographic data because at least your you are supposed to scan a grain within, within the grain 2, 3, 4 orientation information should be attained. 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 the, the beam will scan this sample like this line by line and as I said it will index automatically and then record it and then you go back again it will record. So a typical scan of this range in a normal EBSD a conventional camera takes about six to seven 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, so this is how the, the indexing is done. What now you are looking at is this is not the beam scanning and it is getting automatically indexed.

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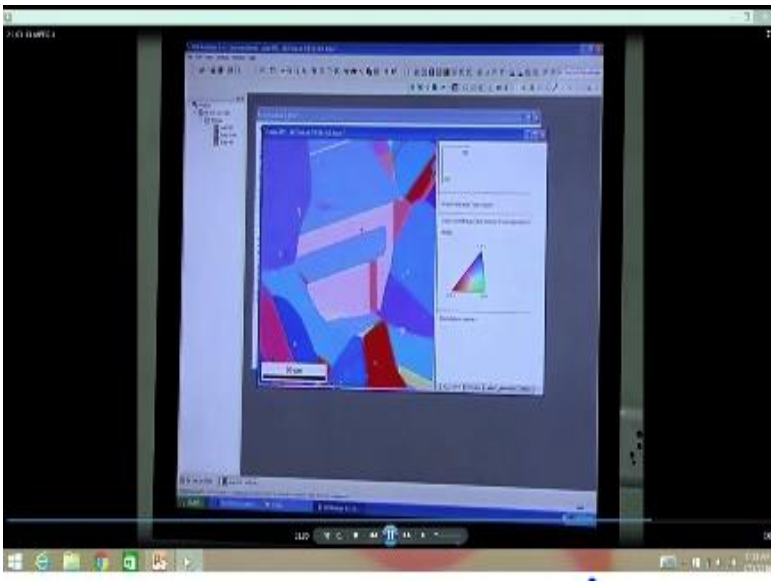

And finally it will get recorded so what, what, what I will do now is since it is going to take long time I will go to the final result for example typically you get the this is a inverse poll figure map. So you see a very nice color colorful picture like this so you have to be very careful in understanding this each color indicates it is a orientation mapping. So be very careful about it this is not a micro structure 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, 0 orientation this green color belong to 1, 0, 1 orientation. And red color belong 2001 orientation so the each color indicates the whole grain orientation belong to this particular number that is what it means. And another

important thing we can do is see what this color is trying to do is to look at the miss orientation between these two grains.

See he has what he has done is, is just taken the cursor and then drawn this line here between deuced two lines these two boundaries you can see that the miss orientation angle between these two is about 60. So that he confirmed this as a twin so you can really readily understand the miss orientation between the two boundaries. So these boundaries are characterized as twin boundaries on the other hand if you do a scan here and then this is only about 30°. So definitely it is not a twin boundary so these are the very, very powerful tool to determine the, the grain orientation instantaneously. And you can do a lot more calculations like you have the orientation spread. And then you have the miss orientation distribution.

You can be Show your distribution also we can see from this sample. So like that you have all this very useful quantitative information can be obtained from this technique and another very important aspect is like you can also look at the surface texture information yeah.

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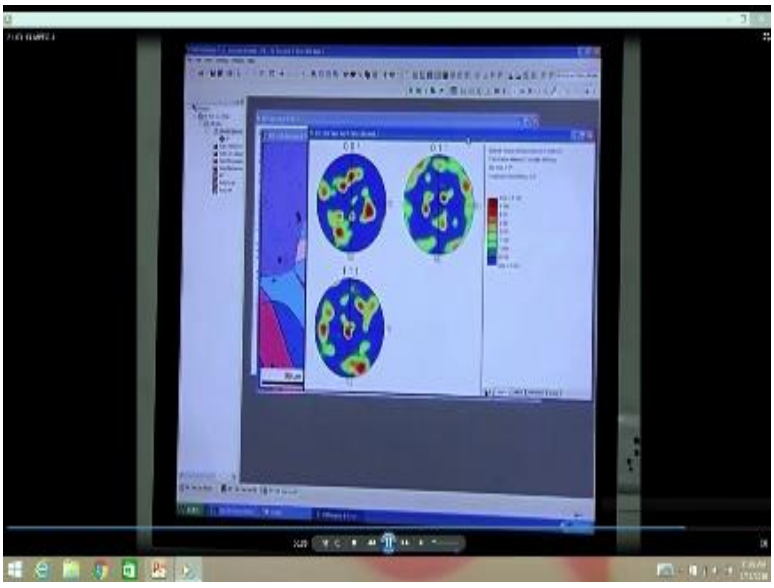

So this is a poll figure which also shows the texture within this top and 20 nanometer layer of the sample. And it shows kind of a random texture here it is not showing any particular texture and we will show you some of the sample where it exclusively shows a very nice texture. And you can also look at the quality maps like this and some of this IQ maps that is quality maps also widely used in some of the re-crystallized grains and deform grains and so on. I am just giving you every glimpses of it I am NOT getting into the details so just basically I am just highlighting the, 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.

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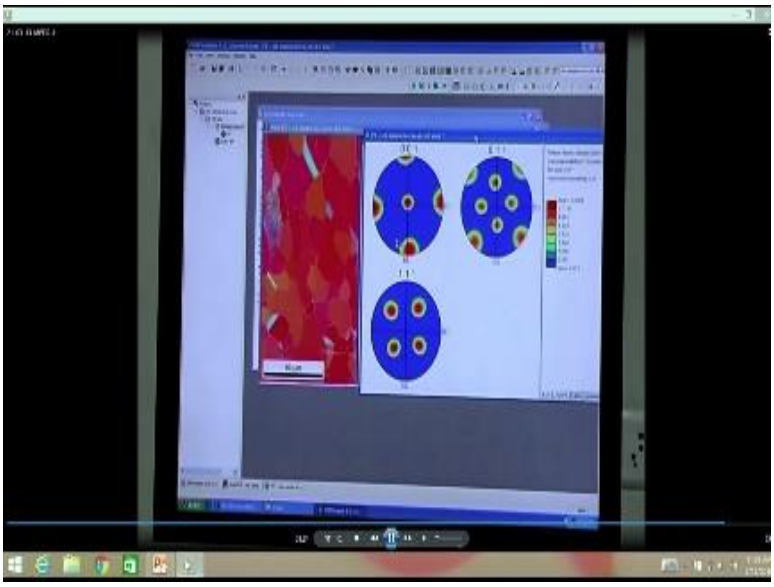

Which I want to give you an example so let me go back and take one more shot okay. So look at this map where it shows mostly 001 orientation that means most of the grains are oriented towards 001 orientation. So if you take a poll figure then it will clearly show you the cube texture 001 texture this is one classical example you can see. How the a cube texture is shown yes so this is very nice a poll figure shows a cube texture 001 any material which exhibits cube texture will show the poll figure of this kind of pre different orientation here. So in this case this is a nickel sample wherever a student processed it to obtain this cube texture.

And again I am telling you this is this information are 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 an x-ray texture EBST is not a characterizing the bulk behavior of the sample. So that point you have to be very careful other than that it is very useful to characterize this again this you can see that miss orientation angle you can see that a low angle and high angle boundary distribution which is readily available used using this 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 may ACM apart from the conventional imaging technique like scanning I mean secondary electron imaging or back scattered electron imaging.

And we have also very briefly introduced the special contrast mechanisms and, and this particular technique just we have just shown I have not gone into the details for the lack of time constraint but then the EBSD itself a separate course one can go through to get into all the details but as a part of this ACM course I think. Whatever I have just shown is I hopefully it 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.

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