Electron Diffraction and Imaging

Prof. Sundararaman M
Department of Metallurgical and Materials Engineering
Indian Institute of Technology, Madras

Lecture - 17
CBED and Precession Electron Diffraction

Welcome you all to this course on Diffraction and Imaging. In the last class we considered parallel diffraction, some different types of parallel diffraction and; what is the effect of dynamical scattering on parallel diffraction we considered. Then we started discussing about convergent beam and diffraction also.

Since, I had done some introduction to convergent beam diffraction, what is one advantage of a convergent beam diffraction is that from whatever is the region we are trying to generate the data it is from a very small region of the sample. When we converge the beam essentially we get the information from an area of cross section may be of the order of we can make it up to some 10 nanometer or 1 nanometer depending upon the type of filament which we have. If we have an FEG type of a filament we can focus it to a very sharp point and still we have very high intensity of that beam.

So that is one. So, from a very small region we get that information, because if we take a parallel beam; getting a crystal which is free of various types of defects is going to be extremely difficult. If we choose a very small region of the sample, the probability of having region which is defect free that is point defects will anyway be there, but at least free of dislocations and all other defects is going to be high. That is one advantage.

Then I mentioned that this could be used to find out lattice parameter measurement accurately.

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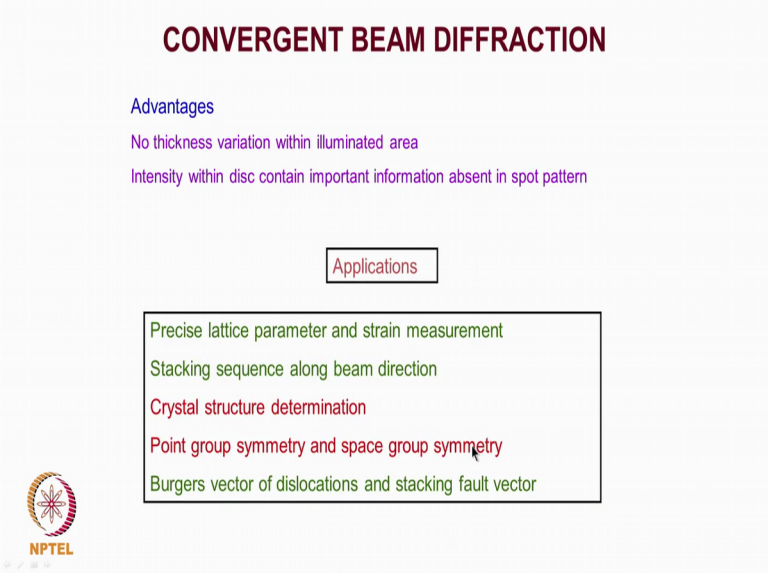

If we can measure the lattice parameter accurately, then the variation lattice parameter is what strain is all about. So, we can find out strain distribution in the sample that information we can get it. Then I mentioned that a unit cell lattice parameter we can get that information. Then we can find out the point group and space group symmetry of the crystal. Then we can find out characteristics of the various defects also could be done.

What I will do today is that try to cover briefly like lattice strain measurement, how point group and space group could be determined, and then also about some recent development in diffraction whatever has come that also I will briefly mentioned that.

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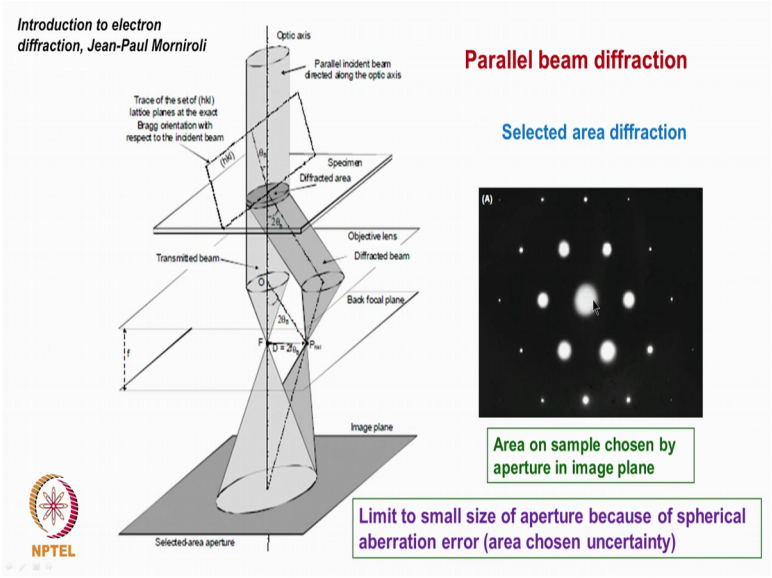

See, if you see here this pattern, how do you as per kinematical condition all the structure factors which we have derived is that kinematical diffraction is taking place correct only single scattering. If that is the case, the central spot should be very strong and all diffracted spots which are coming adjacent to it they should be really weak. But here if you look at it, almost all the spots are equally strong. This happens because of dynamical scattering or multiple scattering taking place within that sample that is the transmitted beam contributes to diffracted beam as well as the diffracted beam contributing to the transmitted beam.

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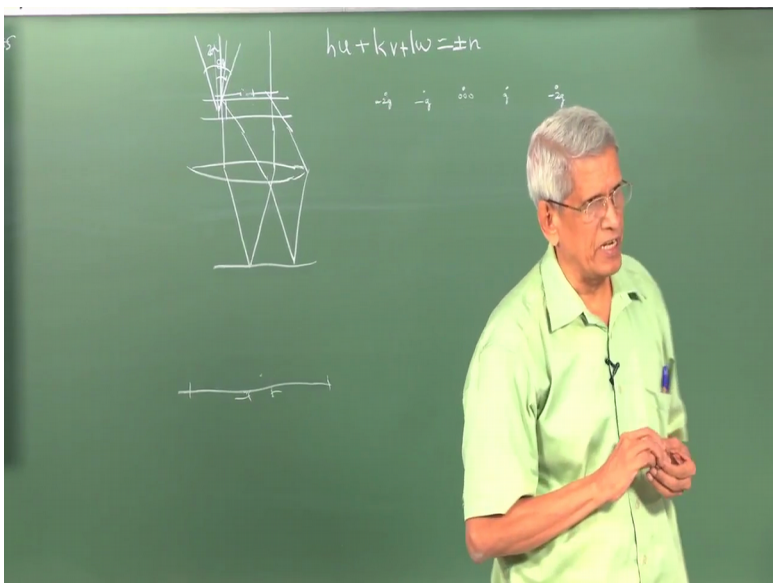

And, with the parallel beam when we take a diffraction normally we have a sample, we make a parallel beam fall onto the sample surface, when this comes out of this some diffraction is taking place then beams which are falling on the lens they are focused to a point, these beams are also focused to a point. So, at the back focal plane we have the diffraction pattern. And there is an image plane which is going to be there.

Suppose I put an aperture here. So, that only this much of the area of the sample I choose; it by putting an aperture on the sample I can or we can decide from which area we wanted diffraction to come. So, the two way in which we can do it is: either you can keep the aperture at the center of the optic axis and move the sample, that is the best option so that all the resolution the since the beam passes through the optic axis resolution will be very good.

But if we do that what is the sort of an aperture size which we can choose. If we go to really small aperture sizes then what is going to happen is that sudden diffraction will take place that is going to mask the effect of; that aperture itself will not be able to see whether it is; what is the size of that aperture correct it is going to be very difficult. How this can be avoided is in the image plane, we get an image of this region which has been where the beam has fallen, so we get some magnified image here.

In this, if I introduce an aperture here choose a small area this is equivalent to, because choosing a very small aperture its equivalent to putting a small aperture on the sample. It is a reciprocity theorem that is if I put an aperture here its magnified view will be coming here only this much region will be seeing. It if I put an aperture this is equivalent to choosing only a small region on the sample. That way the effect of this diffraction effects could be avoided.

But there is a limitation on how small that aperture which we can go. If we make that because all lenses like especially objective lenses got a spherical aberration associated with it. What is the effect of the spherical aberration? Is for a point object, it does not form a point image we get a spread. That means that closer to the edge that can be a spread. So, as the aperture we make it smaller and smaller we will not be sure from which area of the sample we are getting the diffraction; that problem will come.

When such a situation comes we cannot use a aperture at all to get a diffraction pattern. So, then what was done was that can we use a parallel beam, make the beam as small as

possible or you can use a small convergence that can be converged it to a very small beam. This sort of diffraction was the ones which was came in the late 80s, this is used to be called as nano diffraction or micro diffraction.

Especially when you have the source of electrons as field emission gun since the current is high even for a nano beam we can have sufficient current to get diffraction pattern from a such a small region. These essentially one which is taken from the literature if you see it, it is from a carbon nanotube.

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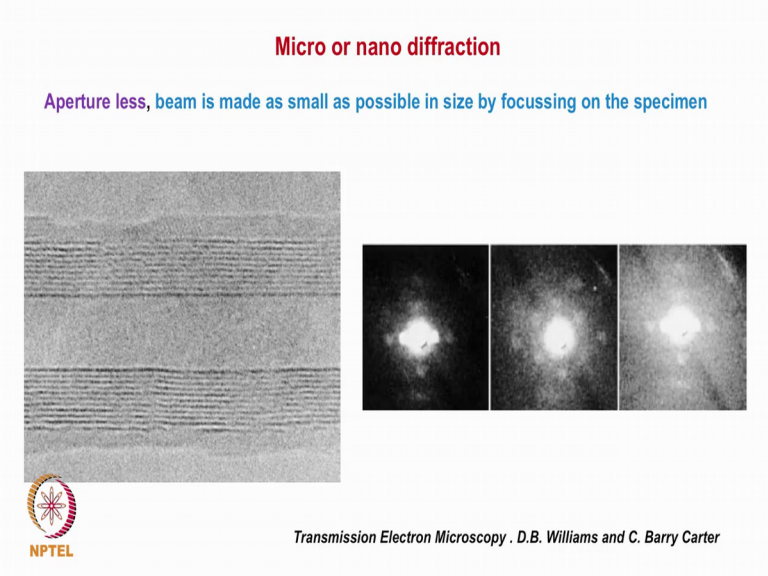

From different regions of it you can put a very small beam that is what we are not using any aperture beam itself has been made small only from that region and diffraction will come. When the beam is being made very small they falls on a small region you know that there will be a spread of spot also will take place, that is what you can see that here and this is how the sort of aperture, the sort of diffraction which we get it. This is called as a nano diffraction.

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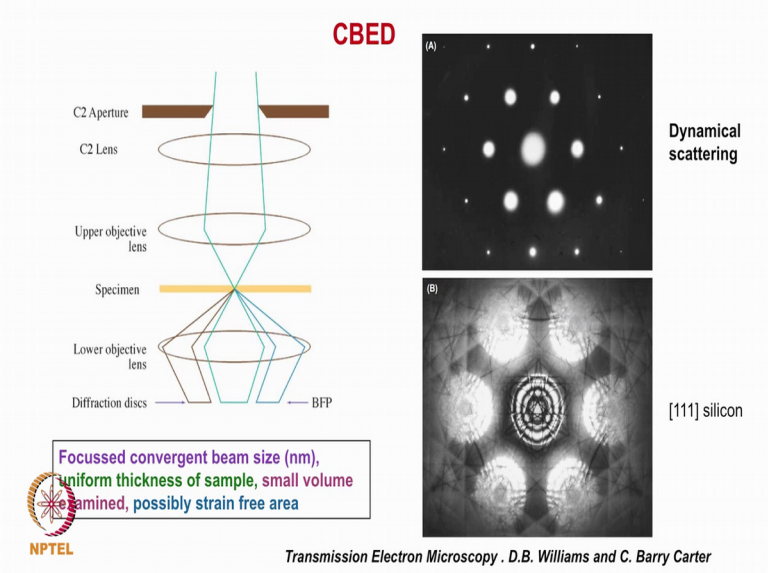

And then in CBED, when we make the beam very small like this nano diffraction or we converge a beam to a very small area only from that region we are getting the diffraction, what is the consequence of that. That means, that suppose you are made the beam into may be assume 10 nanometers; that means, that if we have a second phase particle of the size of 10 nanometer we can make the beam focused onto that region and we can get information about the point group space groups symmetry and a all we can get it from such a small precipitate.

If we use x ray diffraction we require at least about millimeter size samples which will be required. If we have to go for a neutron diffraction to get single crystal we will be requiring a single crystal of the order of maybe about a few millimeter to a centimeter size sample samples will require. Whereas, with electron diffraction we can use samples of the order of 10 nanometers or even 1 nanometer, because with the field emission gun we can make the gun into 1 nanometer size also.

So, from precipitate as small as 1 nanometer or 5 nanometer; that means, that when you are working on a nano particle a particle sizes are very small. For those nanoparticles on each nanoparticle if it is a single crystal you can find out point group space group symmetry of the crystals using convergent beam diffraction. Is it clear?

And then another factor also which is important is that whenever most of the TEM samples are wedge shaped. So, if the thickness varies continuously the intensity of the

diffraction spot also vary from region to region. So, what we get it as the intensity of each of the spots, like the spot here is going to be an average over the thickness. If we choose a very small area the probability of thickness variation in that area is small so that we can assumed to be of a constant thickness.

And I have told yesterday, not as in; the last class that how to find out the thickness of the sample using convergent beam diffraction. I will anyway just repeat it again.

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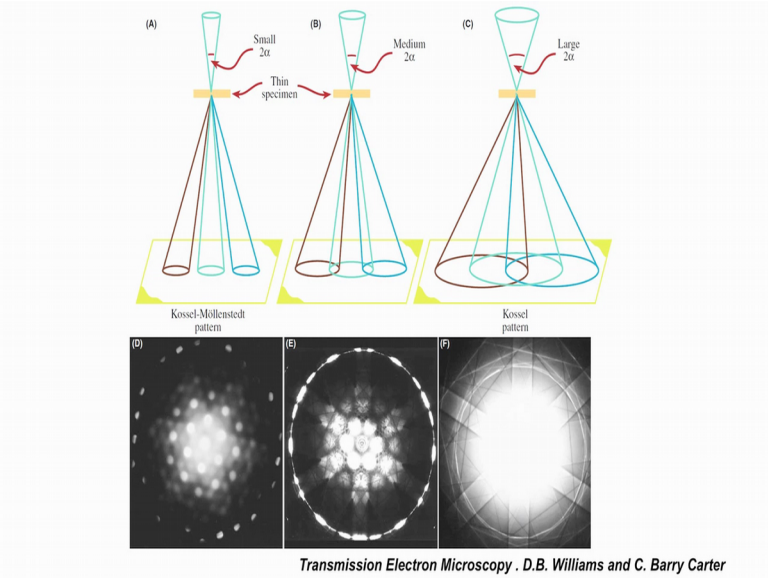

Transmission Electron Microscopy . D.B. Williams and C. Barry Carter

This I mentioned that if the convergence angle is very small and the spots do not overlap, then the type of spot pattern which we get it. That is the type of a pattern which we get it here. In these sorts of patterns what is the convergence angle, the convergence angle is smaller than the Bragg angle.

This also has a consequence. What is the consequence of it; if we consider like a parallel beam like the case which we consider. If the plane is diffracting is also parallel to the beam exactly parallel and the beam is also parallel will we get a diffraction; we will not, but why do we get diffraction when the beams are parallel. That is because though we consider the beam externally to be parallel still many electrons have got a trajectory which has slightly deviated from it which is responsible for a diffraction to take place; that is one. And what is the consequence of it? Is that, when we satisfy a Bragg condition we will be getting a intense one and when we move away from it the intensity drops off drastically.

Another consequence of either that means that for this convergence angle exact Bragg angle diffraction cannot take place within this beam size. If I make this beam size larger so that the angle is larger than the Bragg condition then always a Kikuchi diffraction can also take place. That is even though the beam is convergent most of them though satisfies the Bragg kind angle that is like here. In these one it is a parallel one like here.

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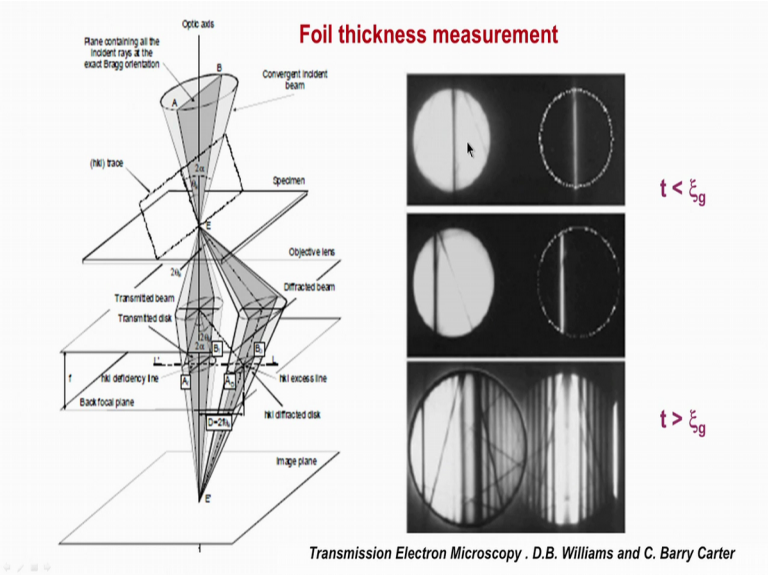

If you consider here the angle two alpha convergence beam is larger than theta b; theta b is the Bragg angle. So, if it is larger than Bragg angle even when the beam is falling onto it for that particular plane most of the beam do not satisfy the Bragg condition they are inclined, but there is one particular direction that is from here to here the beam which is falling in these direction oriented like this. They satisfy exact Bragg condition.

So, when they satisfy exact Bragg condition this will give raise to excess and deficient lines can come: deficient line in central spot and excess line in diffracted spot. That is what we have seen in the last class, correct. That is here there is a deficient line, there is an exact line. And you do not see any other contrast here because of the simple reason; that the thickness of the foil here is less than psi g- psi g is the extension distance which I had introduced in the last class what the psi g means. If the extinction (Refer Time: 15:34) if the thickness of the foil is larger than psi g then some fluctuations in amplitude can happen that will give raise to a fringe contrast.

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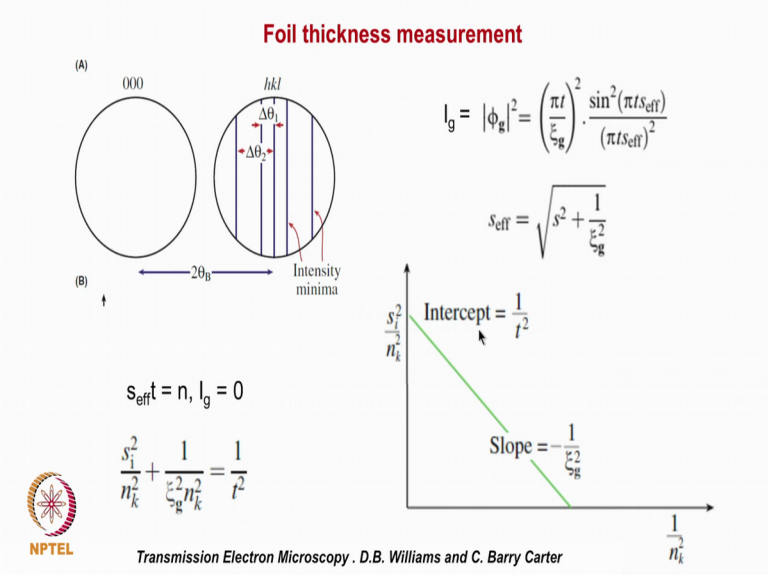

Using this fringe contrast measuring the separation between the fringes and using this condition for the intensity equals sin square pi t S by this factor. So, the t into S effective if it becomes n the intensity will start fluctuating right, if it becomes pi by 2 this factor then it will become intense (Refer Time: 16:16).

So, there will be an alternate bright and dark fringes will come. So, that is what essentially is S effective is nothing but root of S squared plus 1 by psi g; this we will come later in the next week will talk about it. So, then we will be able to write an expression like this. With these sort of an expression we can draw a straight line connecting these two terms and the intercept is going to be there 1 by t square that slop from which we can find out from the region that beam has fallen the thickness of the foil. And this is occurring because of we have considered here only a two beam condition; that is means that only one plane is satisfying the Bragg condition. If only one plane is satisfying the Bragg condition what will be the effect under diffraction spot, how will the diffractions pattern look like if only one plane is responsible for it- only one or more.

Student: No, there will be more, but it will be (Refer Time: 17:27).

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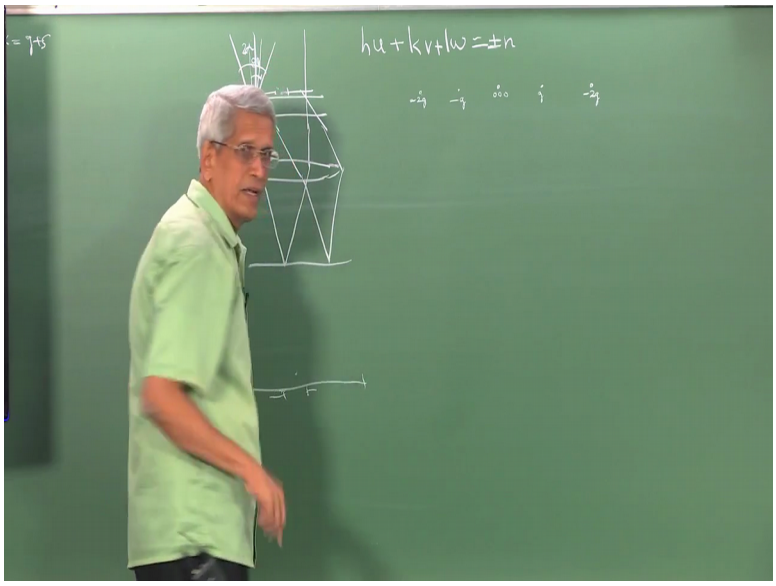

Will be all on that is this; this is the central spot this will be g minus g that is along a row reflections can come, because if g can come first order, second order, third order reflections also can come from the same plane, correct. So, only one row of spots will come. So, in that case we will be getting only fringes like this.

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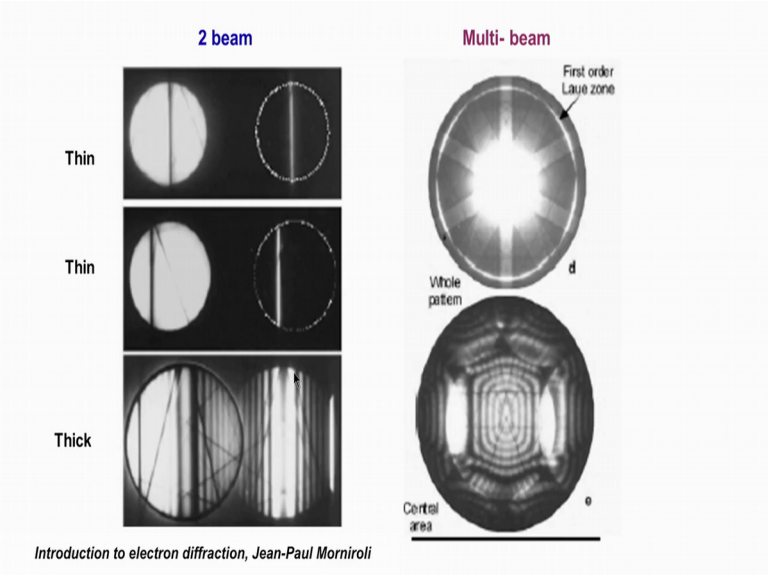

Suppose the orientation of the beam is such that it is along us own axis then many more spots are simultaneously getting excited. That means, that many more planes are satisfying Bragg condition. So, we can assume to be each one of them is giving raise to a

fringe pattern like this. What will be the net effect of it? It will be getting a variation in contrast within the central spot in this.

Student: When do we get a single line?

Which one?

Student: When do we get the single line?

The single line you get it when it is only a two beam condition; that means, only one plane is responsible for Bragg diffraction. When you have the beam direction is such that like for example in a simple cubic structure if the beam is along 0 0 1 you can have 1 0 0 plane, 0 1 0 plane, 1 1 0 plane, 1 1 bar 0 plane all of them can give raise to diffraction, correct. So, in such a case you will be getting a sort of this, sort of a fringe which will be like this, which will be essentially displaying that symmetry of that zone axis. Is it clear?

Student: Sir, but the frame, what was the relation of the thickness?

The relation with the thickness: he if it exactly satisfies the Bragg condition then there should be a dark line here at the center of this disc and a bright line should come here, correct. That is why the central spot is. Then S effective into t should be there as the beam is a conical beam other beams it is away from the Bragg condition. So, since it is away there is a variation in s c's continuously varying. The S effective into t for whatever be the value of becomes integers there will be a dark fringes will come in between white fringe will come. So, that depending upon what the separation is going to be there. And on the lattice parameter of the material all this you will be getting a set of fringes which will be coming. So, that is what is responsible for it.

See the whole contrast in microscopy when we talk about it is all interference contrast or you can see is the phase contrast; phase contrast is nothing but an interference effect. Then I mentioned that if we take a zone axis pattern; if a beam is parallel beam and it is falling on the sample in one particular direction like in this case.

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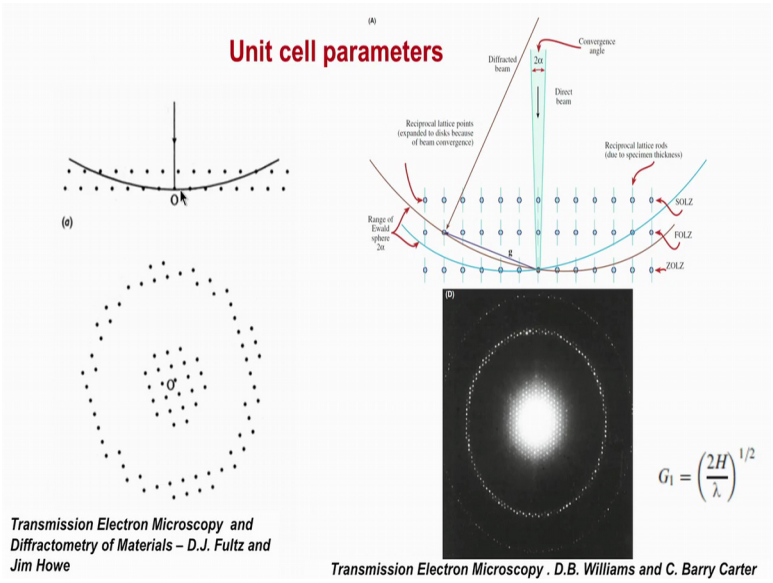

And if we make the camera constant very small then in the size of the screen which we have chosen we can see not only the Zeroth Order Laue Zone, the Higher Order Laue Zone some spots will also be seen. What will be the distance from here to here? This we can measure it on the screen film. And if we measure this and the radius of the Ewald's sphere is 1 by k, and this distance is h. And so using this relationship we can find out what is going to be the value of h.

Student: Sir, but the circle will not cut like the point exactly, right.

Which one?

Student: The exact we will not know the exact.

Essentially when you reduce it and many orders are coming; essentially like here if you see it- that which is the one which you take it to be the central that is the one which exactly satisfies all the points satisfying the Bragg condition exact Bragg condition.

Student: (Refer Time: 22:24) which one will exactly some of them will be the half curves.

When the some of them are half only for one particular one it is going to satisfy with respect to a center on exact circle. There will be some error will be there associated with

it, but the distance which is being measured is large. So, essentially what you can do it is with this we will be able to find out what is going to be the h.

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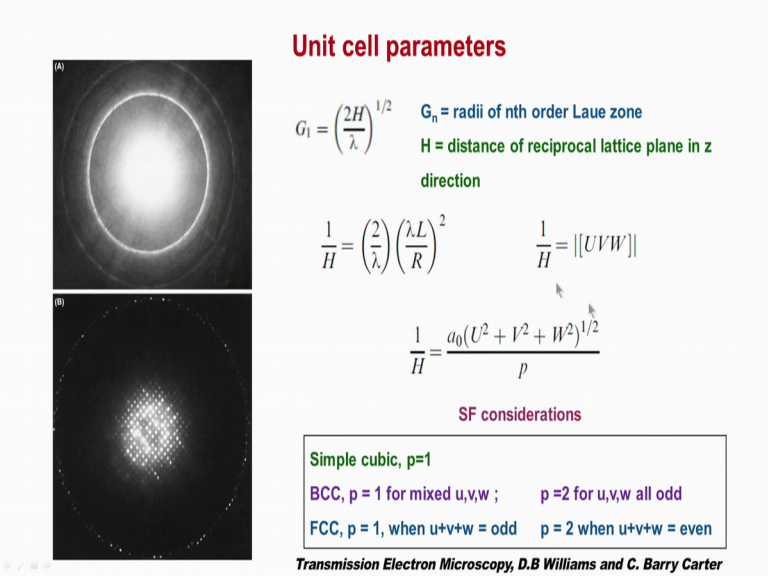

This h how we can relate it, depending upon the type of crystal which we have whether it is a primitive or body centered or a face centered this 1 by H could be returned as UVW. This is in terms of direction also we can write it with respect to a crystal structure, the magnitude of the vector. If you do that then this will turn out to be a factor P which we can write it, this factor will be depending upon simple cubic this p will be 1. BCC this p will be 1 for UVW, this you can work it out because you know the structure factor consideration and the same basis it comes for the vectors, then 2 for UVW all odd. Similarly for FCC also these factors will be there.

This way we can one can find out what.

Student: This UVW will be zone axis.

This UVW is the vector in the real lattice UVW; that is a zone axis direction.

Student: Zone axis.

Correct.

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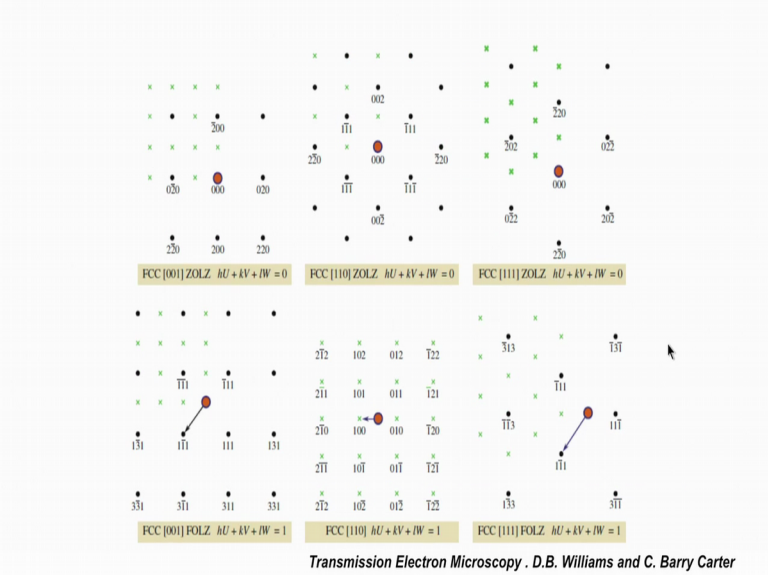

Then I showed also that when you have the reciprocal lattice; say when you have constructed it for difference zone axis condition like hu plus kv plus lw equals plus minus n; n can be 0 then it is the Zeroth Order Laue Zone, n can be equal to 1, 2 like that then we can generate different reciprocal lattice section. This is what it is being shown for FCC for a 0 0 1, 0 1 1, and I think 1 1 1 type of zones for the zeroth order zone as well as for the first order zone.

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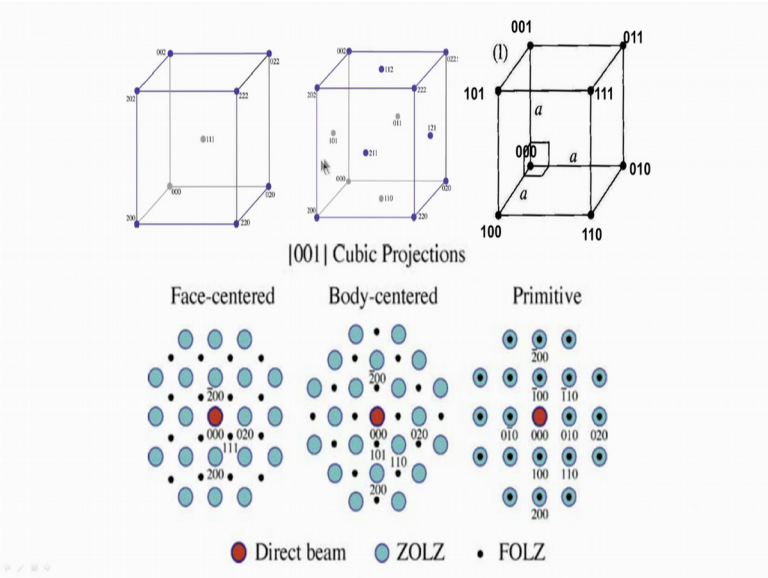

This same thing which I had shown it here I had just shown the reciprocal lattice section. So, if you consider with respect to a when the Ewald's sphere goes, Ewald's sphere will cut here and Ewald's sphere if it cuts 1 1 1 from that you can make out that which is going to be the one which satisfies this condition. Then one can generate patterns like this. And for a face centered this is how the pattern should look like.

But this central spots like 1 1 1 type of a spot which satisfies this condition hu plus kv plus lw equals 1 will not be visible on the central spot. That is actually coming as the First Order Laue Zone a few spots which you get it. But if we try to extrapolate it and complete that full reciprocal lattice plane then we will be finding that this is where they will become a sitting on top of the reciprocal lattice point; that is what it has been drawn it is a simulated pattern. Is this clear?

Whereas, for a primitive lattice all the spots will be for this particular zone axis direction it should be one will be coming on top of the other.

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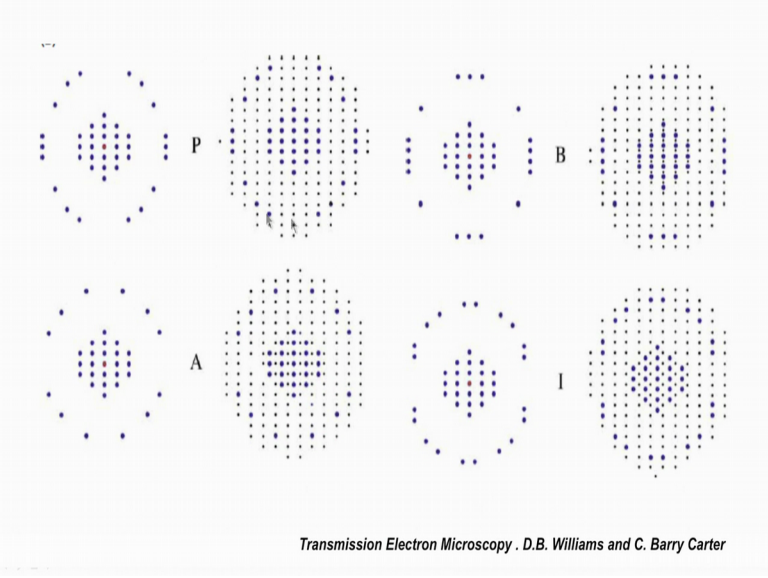

That is exactly what is being shown here- is if it is a primitive lattice this is how the Zeroth Order Laue Zone and the First Order Laue Zone looks like. And this if we try to we know this distance then we can generate this lattice and when it merges this is how it will look like overall when we simulate it. If it is for a A centered, this if we try to extend it and superimpose it this is how it will look like. For a B centered this is how in these direction it will be summed. If is body centered this is how it will look like.

This sort of patterns we have to generate it. That is all this things since you know all the structure factor rules and reciprocal lattice can be constructed, we can generate this sort of patterns so that we are familiar with it and when you do a microscopy, you immediately know that the if it appears like this you know this is the crystal structure with you are dealing then immediately you can make out to which zone it corresponds to.

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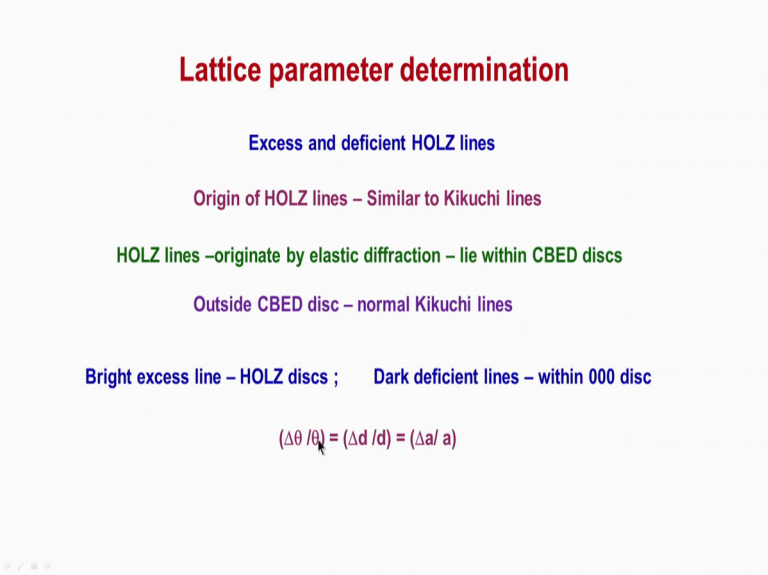

Then another is for the lattice parameter determination if you are looking for or strain measurement both I mentioned that there will be excess and deficient lines will be there, correct. The convergence angle is very small. Due to elastic scattering of the convergent beam we need not get a deficient line at the center, because that beam is not in a condition to satisfy the Bragg condition; because the convergence angle is not. Whereas, in a conventional sample even when we have parallel beam we do get a Kikuchi diffraction pattern, correct.

What is the reason for the Kikuchi pattern to take place? Even though the beam is parallel, for some electrons as it entering into it, some point in the sample incoherence scattering is taking place- incoherent and inelastic and because of that that beam becomes a divergent beam. That is what is responsible for the Kikuchi line.

So, if you consider an elastic scattering within the converged beam unless the angle of convergence of the sample is made very large. That is suppose the planes are like this and if this is what the angle theta b has to be. If I make the convergent beam of the angle

to alpha larger than this definitely for this orientation of elastic diffraction is going to take place in one region which satisfies exact Bragg condition the other regions there will be a uniform intensity, because from this region the beam has been that is from this plane the beam has been diffracted so intensity of the transmitted beam has come down that will give raise to a deficient line.

Though this is similar to; and this line will be seen only within this spot because center this one if you look at it this is like a cone in which it is happening this is the beam.

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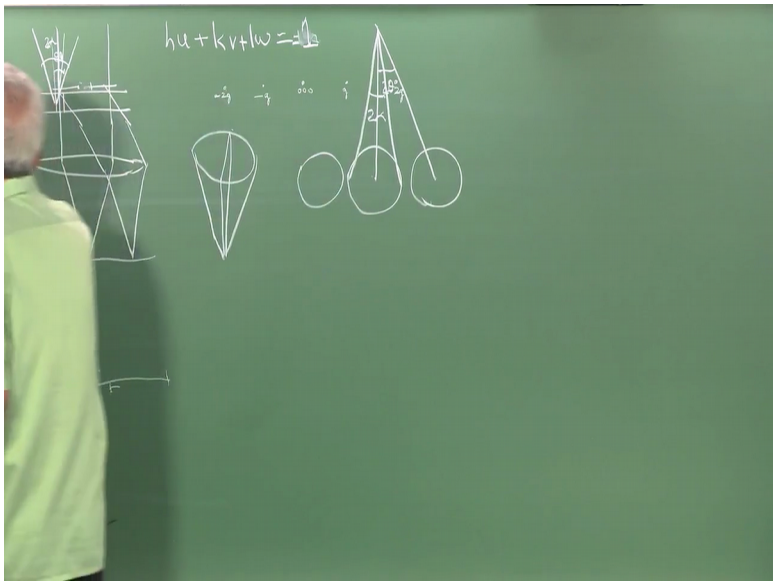

So, in this particular beam if you look at it, the Bragg condition is being satisfied only for this particular area of it, correct. So, only within this the diffraction spot which we get it within the undiffracted spot or the central beam only we will be getting this deficient line. And corresponding to that an excess line should come in the diffracted spot or the diffracted beam.

Suppose, the convergent angle is smaller than that of the theta beam then what can happen. That is what the case in when the diffraction spots are not merging. Each diffracted that is convergent beam the patterns are like this, if we get spots like this. When the spots do not merge, because from here to here with respect to a sample if I see take it this is two theta correct. So, what is the convergence angle of the beam is going to be only this much to alpha. So, this is always smaller than the Bragg angle.

If that is the case for the Zeroth Order Laue Zone mostly we will not get deficient line within the direct beam. But what does this h u plus k v plus equals 1 means? For this case the planes are in the sample; the planes are very much inclined with respect to the beam direction.

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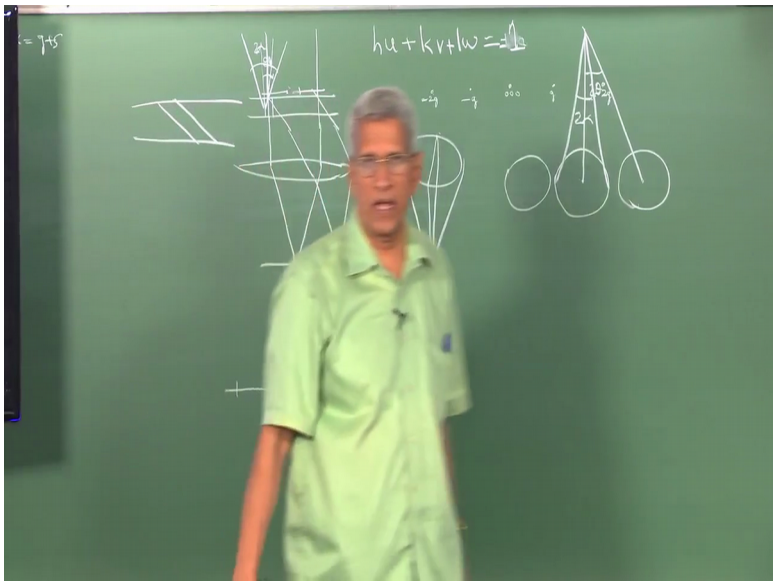

For those planes this convergent beam could satisfy, it can still be falling within the Bragg condition. Because of that only for higher order reflections you will be getting Kikuchi type of a pattern or called as deficient Higher Order Laue Zone spots in the central spot, and the excess lines will come in First Order Laue Zones only- first order or second order those Higher Order Laue Zones. Is this clear? Outside of this we will come to I will just show you that. And these bright excess lines and they are called as the HOLZ disc and dark deficient lines are the ones which we see within this disc.

Then another thing also which you know that delta theta by theta is going to be equal to delta a by a; the larger the Bragg angle that suppose a sample is there some strain is there the lattice parameter and delta a b is the strain. For the same strain larger the theta value which we choose delta theta is going to be large; that means that the separation between the lines which we were going to see is going to increase.

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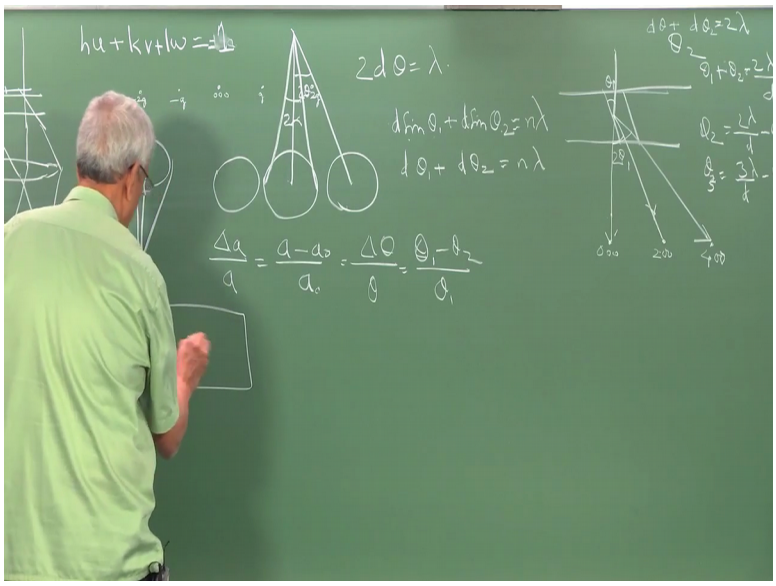

What does it mean? Delta a by a is equal to a minus a 0 by a 0 you write this will be like delta theta by this is a strain. So, corresponding to a as well as a 0 you should be getting two lines. That means, that delta theta by theta will also be equal to theta 1 minus theta 2 by you write theta 1. The larger the theta value this delta theta 1 minus theta 2 is going to be large, because what is the formula you write it? 2 d sin theta when theta is small 2 d theta equals lambda, you differentiate it you will be getting this expression.

So, for larger Bragg angle or Higher Order Laue Zones if you use it they correspond to from larger theta Bragg theta values so then we are going to get this expression.

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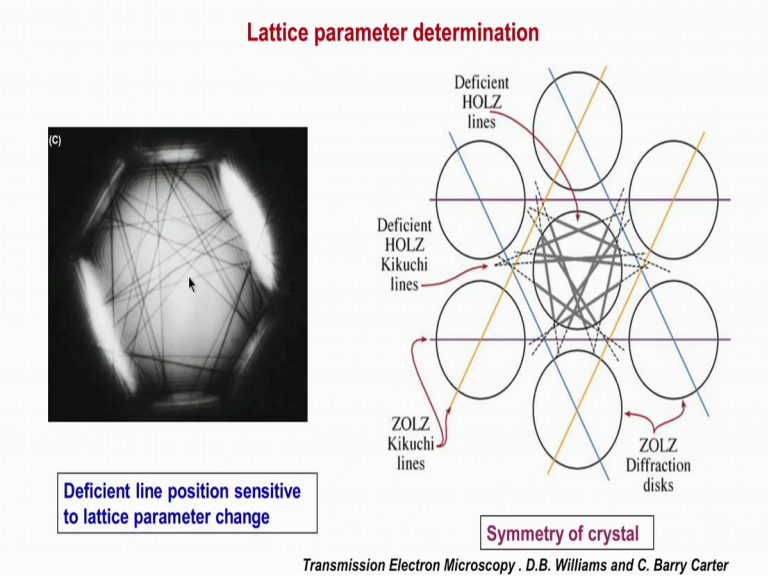

So, you look at the central spot there are lots of lines are there. These deficient lines are corresponding to some elastic diffraction is taking place so this is like a Kikuchi type of a diffraction which is occurring because the beam is a convergent beam for some particular direction that satisfies the exact Bragg condition otherwise do not and to find out the lattice parameter from this.

Since, we know the value of the camera constant if we know the lattice parameter of the sample a camera constant and the distances can be measured. So, we can simulate the pattern and try to match it. Some computer simulation will be required to exactly match this different. That is essentially what is being shown in the next slide.

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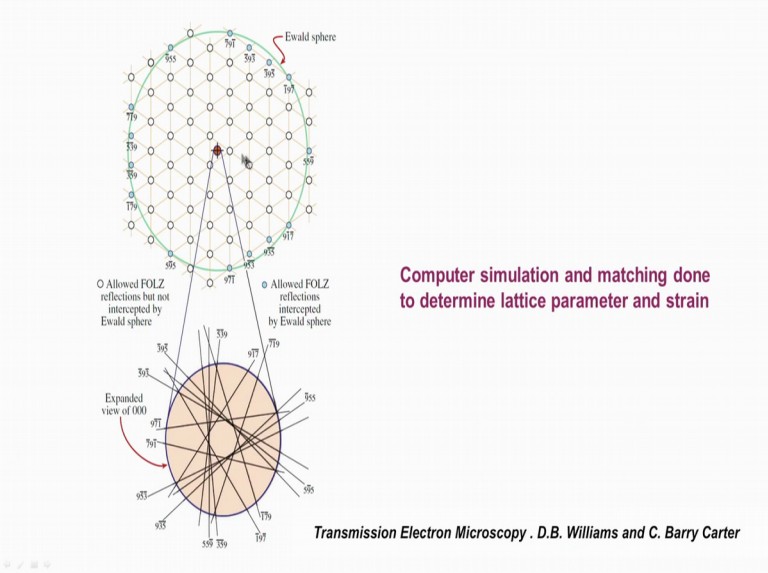

You see this, this is a computer simulated diffraction pattern where within this circle is what you see all this lines and these are all the ones where the excess Kikuchi lines will be

Student: The bright line.

Bright lines will be appearing. With these one can try to find out the lattice parameter very accurately.

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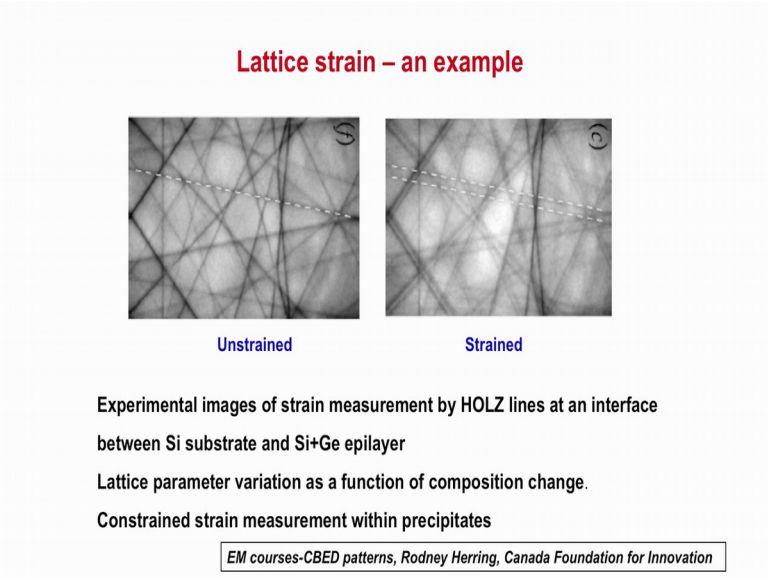

And another also as the Bragg angles becomes large the way the intensity falls of sharply on either side from the Bragg condition is (Refer Time: 36:54) because of that these lines also become very sharp. So, the sharper the deficient lines you can measure their separation measure their distances very accurately.

Here, I just showing an example where in the central spot you have deficient lines are there. Is in the sample in the unstrain condition? The sample is being strained. Now you have this lines of all are split, the separation between this lines if you can measure and one you know the lattice parameter is has to be all done using a simulation then you can find out the strains very accurately.

This can so happen that you have a sample in some region a composition is changing. If the composition changes from region to region the lattice parameter also will change. That means that from one region to another region that is a strain which is introduced in the sample, correct. Then suppose it is a precipitate which is there like gamma plane precipitates which forms in nickel base super alloys they have very large size or the order of micron size.

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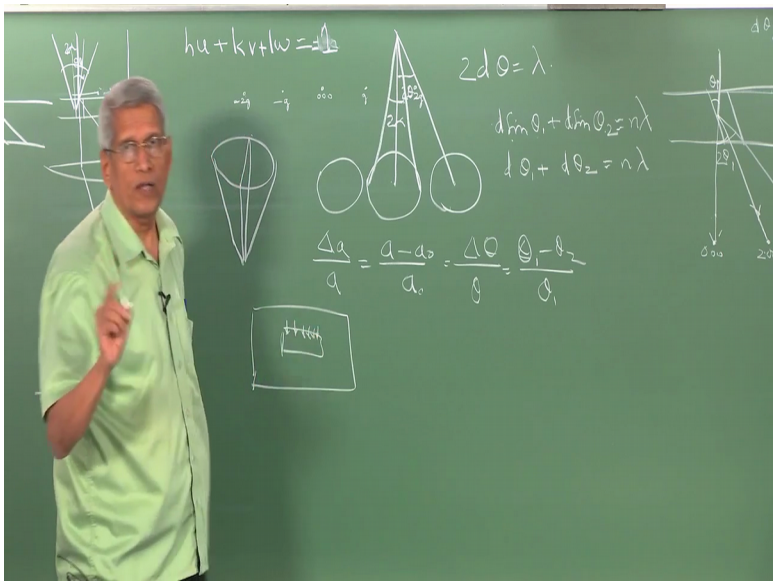

So, those precipitates are essentially in these region then I can put a beam on different parts of the, because since I can make the beam into few nanometers I can make the beam fall everywhere and find out what is going to be the strain which can be measured.

So, the strain mapping could be done at different regions of the precipitants. This has been done routinely by researchers.

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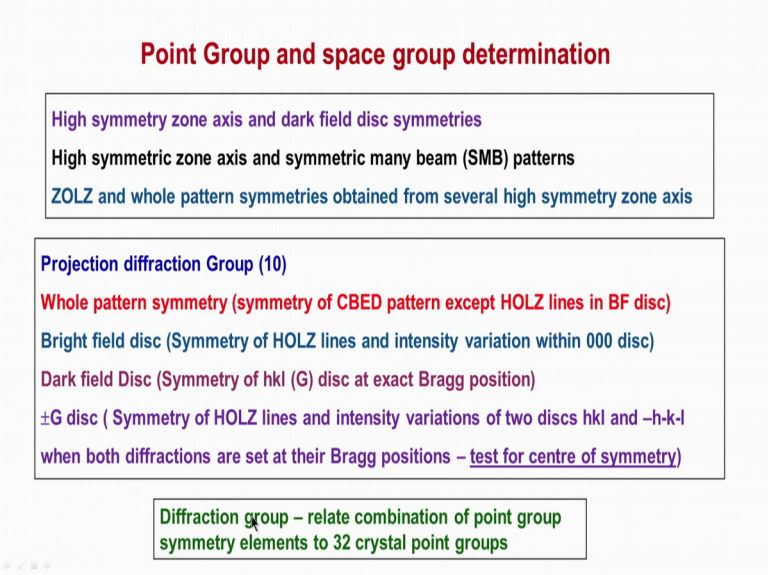

Point group and space group symmetry. This is clear? This way we can find out by matching this and finding out the separation between this Kikuchi lines with respect to the spot. We can measure the lattice parameter as well as the strain. Point group and space group determination; I will not go into the detail, because it is a tough thing one has to understand quiet a bit. But I wanted to tell is that this requires lot of patterns which one has to take.

The patterns essentially which are required are a high symmetry patterns and then plus g minus g condition also we have to take some patterns. And these are called some different types of projection. That is there are some terminologies which are being used in this analysis. One is called as the projection diffraction group projection diffraction group means that if we take a two dimensional diffraction group how many two dimension diffraction group which we have- that is five Bravais lattices, correct. And 10 point groups are there, plane groups are there. That is all which we can get it here.

So, in the diffraction pattern also we will get it that will exhibit that. Then another is that each of the disc can contain some intensity variations which will exhibit particular type of symmetries associated with it. This is like if you remember; if you look at the

stereographic projection of the different point groups each is typical of the point group associated with it right, like if a mirror you know that how a mirror should appear.

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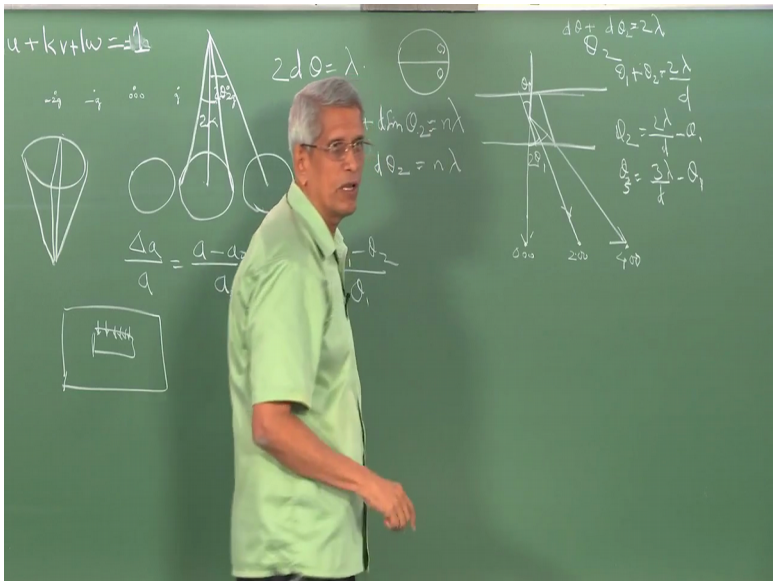

That is if I take a; suppose this is the mirror plane any point which is there a correspondingly there will be another set this is how it will appear. So, in the disc also each of this disc which we are seeing it the diffraction disc also exhibit this sort of symmetries. That the only thing which I can tell you. But what is the origin of it how it comes? That is beyond the scope.

So, then we look at the bright field disc; that is bright field disc means that the undiffracted one. What sort of symmetries which it exhibit? Then plus minus g you tilt it and look at the symmetry or with the convergent beam only. If we do that then we will be able to find out various point group symmetries. That requires a lot of things which I am. But what I will just show you is that from this you generate what is called as the diffraction group all these things are beyond this class.

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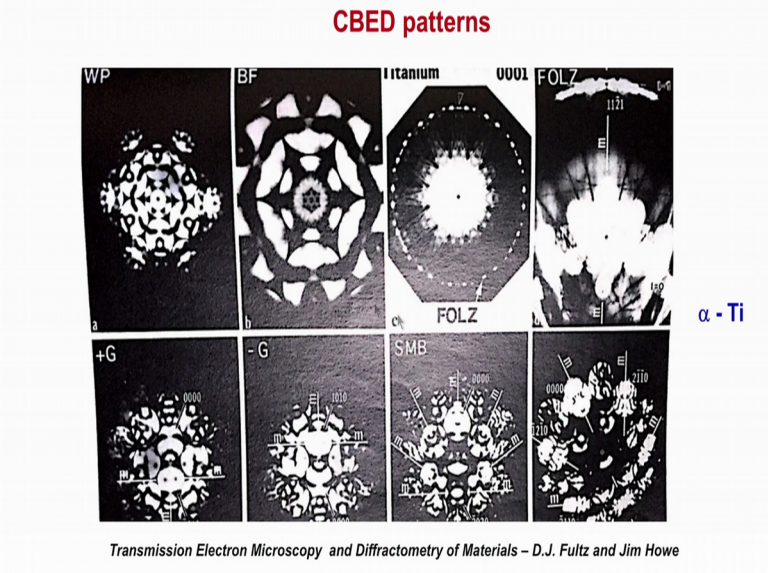

Example you see that this is the whole pattern which is called as, that is central spot and nearest neighbor spots are being shown, it exhibits some symmetry. The bright field pattern means that only the central spot. What is sort of symmetry which it exhibits? This is what is called as the bright field spot. You look here, this is plus g the same thing is tilted so that this part becomes instead of the central spot one of the diffraction spotter becomes stronger. If you look here you see two sort of dots come in the picture and with respect to this if you try to see it exhibits symmetry. With respect to this if you try to see it there is mirror symmetry is there.

Similarly, here if you see it like this various symmetries can be seen. This sort of patterns have to be taken on various zone axis and you look at the type of symmetries which they exhibit. This is used to find out the point group symmetries very correctly.

Student: These patterns come out of?

These patterns are all coming out of convergent beam diffraction only. What you have shown is that this is the symmetric pattern, this is from a alpha titanium, but if I tilt the beam in such a way that the central spots moves- this part becomes very strong the additional spots shifted then this is the sort of a pattern. Now you look at within each of the spots symmetry has changed. You understand that some variation in contrast which comes that exhibits point group symmetry. These are used to find out the overall point group symmetry that is all which you have to remember.

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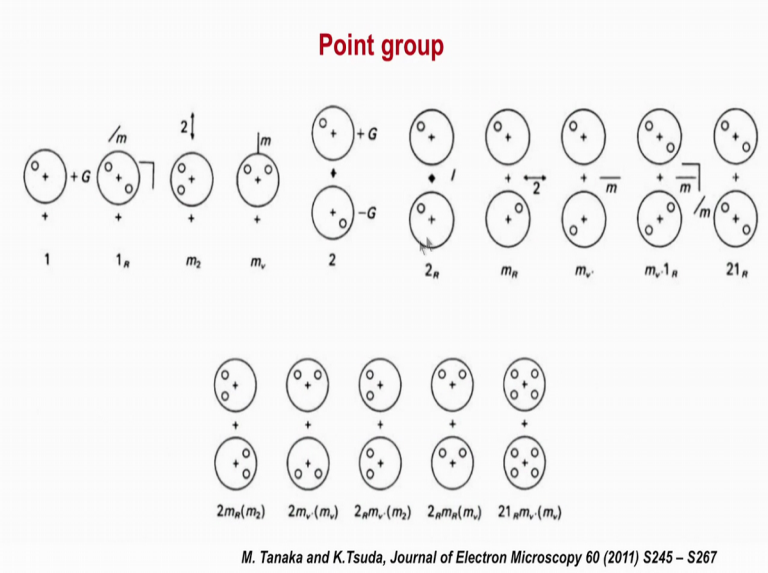

This is essentially some of the things which has been theoretically done; how it will look like, which I do not want go into the details of it. But these are used as the comparison to compare with the other one to find out that is why is that is a very involved work.

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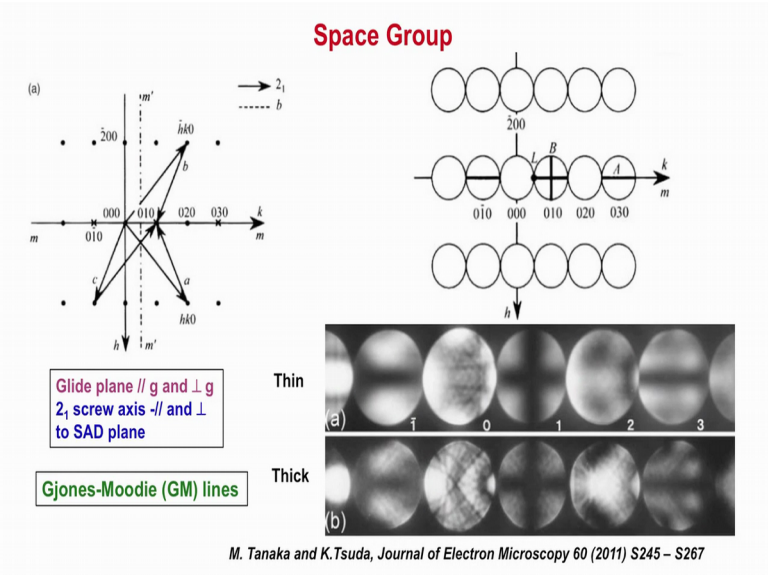

But this is very interesting and exciting one. And this is how do we find out a space group. Space group can be determined very easily.

Using a kinematical condition suppose you assume that somehow multiple diffraction does not occur: the foil is extremely thin then the central beam will go only single

scattering is taking place. We will be getting some diffraction pattern. What will be the intensity of that each of the diffraction spots?

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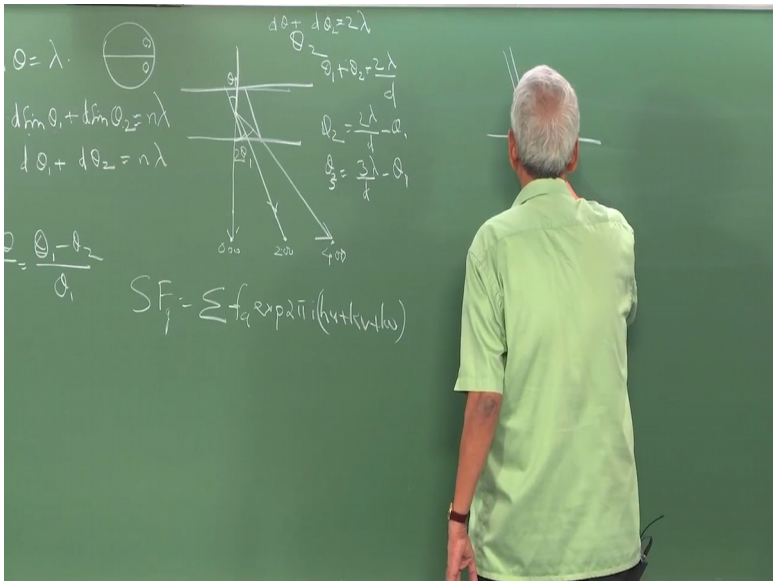

It is decided by h u plus k v plus l w, correct. This is done over all the h u v and w for a particular k. This structure factor is essentially for f of g I should put it right, for particular g value. Like that you can calculate it for different g values one can calculate the structure factor, square of structure factor gives intensity assuming thickness is small otherwise you multiply it, you take the shape factor also you will be able to find out intensity accurately. And since we are taking the position of each of the atom in the lattice, not the lattice in the unit cell the intensity now corresponds to what positions the atom is having it, right.

If you are able to match these intensities we can go back and tell that these are all the positions atom should have occupy. And in the earlier class I had told that the structure factor conditions are not only there for the Bravais lattice for glide as well as the screw axis you have some structure factor considerations are there, correct.

If you take all these things into consideration this is a conventional normal diffraction pattern. And even under convergent beam conditions some kinematical condition is being satisfied where, some you can say the line of no contrast comes in these ones. These lines of no contrast is called as the GM lines the origin of it; you do not have to bother about it, but like here if you see this is some analysis which has been done for a pattern it is an

orthorhombic structure. If you look at it there is a one mirror which is going to a one glide here and there is a glide which is perpendicular to it these are all the two glides which are being shown. Then one should be getting a class within this particular one.

This is what the theory says, and this is the simulated pattern you look at the experimental pattern this is what you get it. Then it alternates for others. That is why is this is an one this sort of looking at the type of extinction lines which appear in these spots and the symmetry associated with it we can find out point group and space group symmetry.

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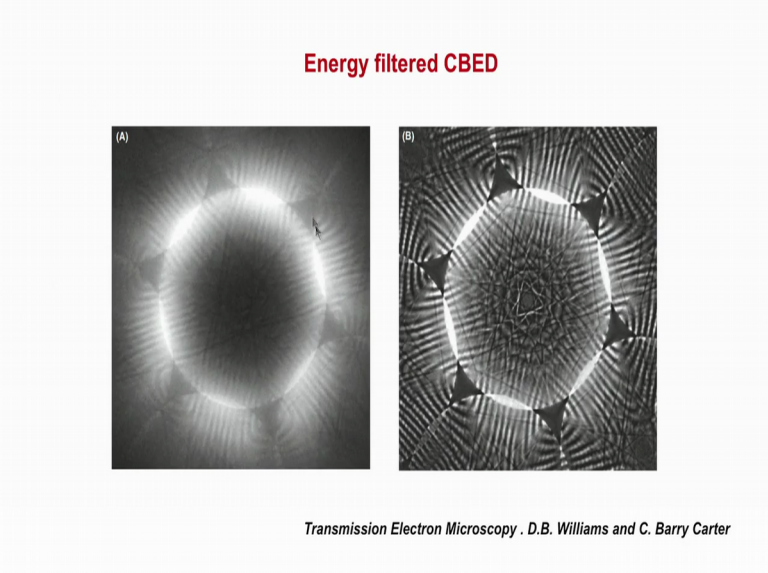

Before that I will tell you that some of this path if the sample is really thick then what happens is that lot of inelastic scattering will be taking place incoherent elastic scattering, that will give rise to a uniform background and the convergent beam diffraction pattern. If we use an energy filtered EFTM- Energy Filtered Transmission Electron Microscope then we can filter all the inelastically scattered electrons out of the way and make the diffraction patterns similarly image also with only the elastically scattered electrons. Then we can get much better contrast.

This is the same one where in elastic scattering has been removed.

Student: You will not the dark fringes go away, sir?

We should know nothing will be (Refer Time: 48:06) because the inelastically scattered electrons are coming from as the sample beam passes through the sample electrons are scattered for various reasons other than diffraction. So, there is an intensity reduction. They give rise to a uniform background, that those electrons are being removed. And now we form an image or a diffraction pattern only with elastically scattered electrons. Then the image will be very sharp that is precisely you can see these two difference, difference between these two patterns it is the same pattern without energy filtering this is with energy filtering.

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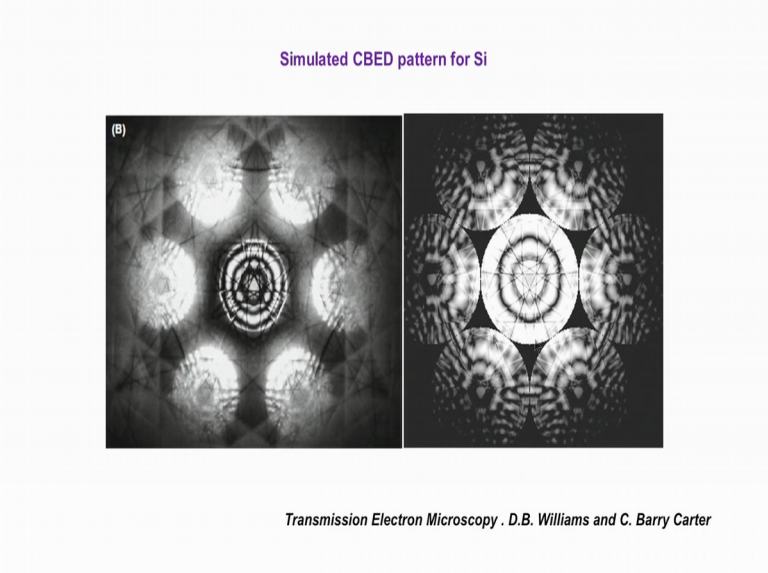

And many patterns if we have to analyze it completely we have to do a compute the simulation also. This simulation has been done I think taking some 30 directions for the beam for the convergence angle. Then you can see that there is a good one-to-one matching between the experimentally observed and the theoretically determined convergent beam pattern.

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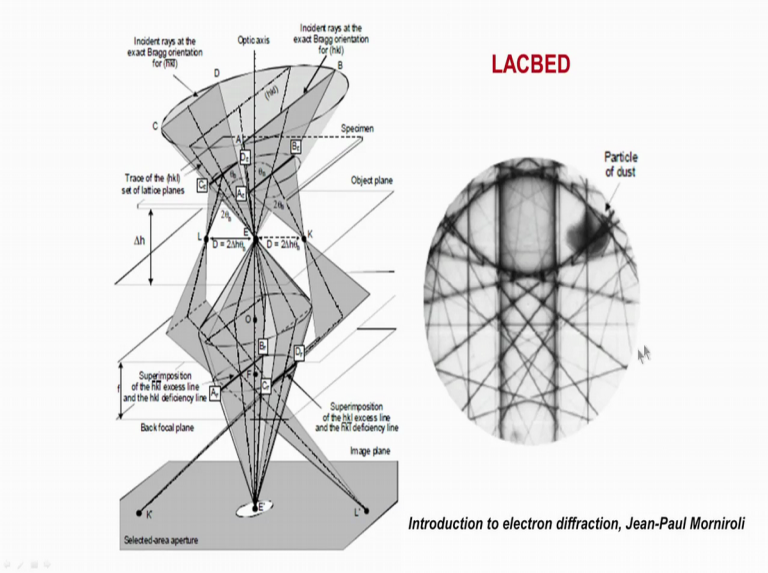

Then another one is called a large angle, this I will just keep it.

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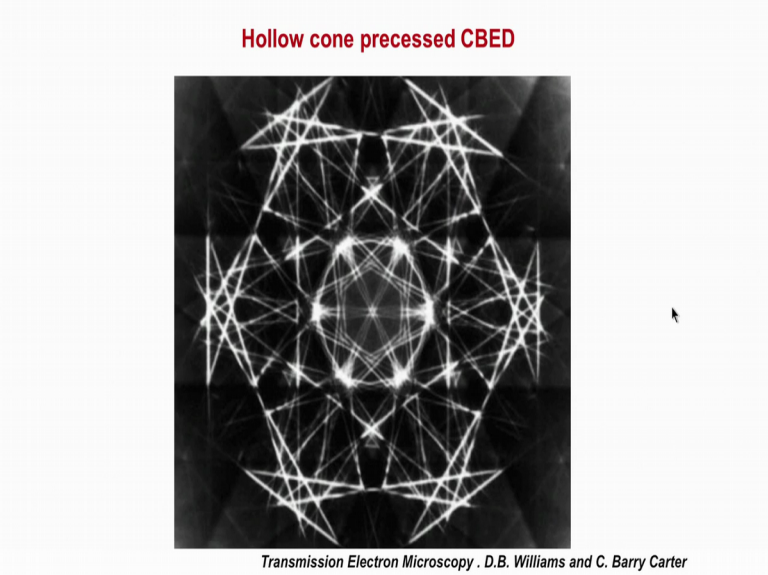

This is something which is very interesting. What is being done is that- normally there is a convergent beam is there here getting a diffraction pattern, correct. Suppose I keep the beam in this direction slightly tilt the sample, if I rotate the sample, presses it around the particular angle just rotate it then what will happen?

Student: The refraction pattern will.

Will rotate, and then many effects of diffraction gets averaged docked that is what you have to remember either. Then all the disc now have vanished, they have all average doubt. And intensity of some reflections go up, high. And now you see that only the excess lines are seen nothing else. I think this is the first time this precession was done. And this is called as the hollow cone. This same thing can be done like keeping the sample and I have a cone of a beam the cone can be also rotated. See this is exactly similar to the spinning top; when you put it what it happens it rotates around it, it spins around it, but at the same time it goes around an axis around it makes a constant angle and rotates.

Student: (Refer Time: 50:29).

Here scope that exactly what is being done in precession.

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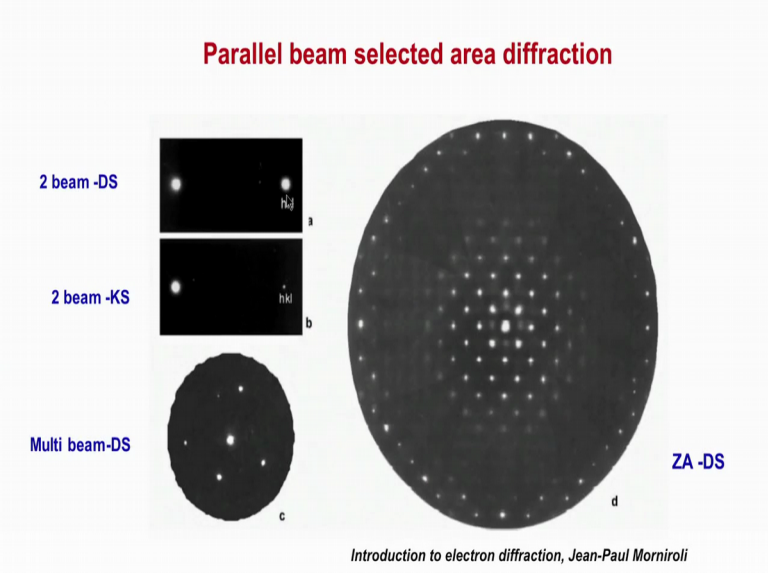

This is normally a parallel beam selected area diffraction if I take it. If it is a two beam condition I have one central spot and a bright field reflection is seen here correct. Central spot and the diffraction spot is there. These two beam dynamical scattering that is why both the spots are very strong. If it is a kinematical condition this spot is strong, this spot becomes weak. How we can do it is- if I tilt the sample a little bit go away from it still there may be some scattering which will take place, but dynamical scattering effect will get reduced.

So, you can see that identical, but the intensity has become very weak. This condition you call it as a kinematical.

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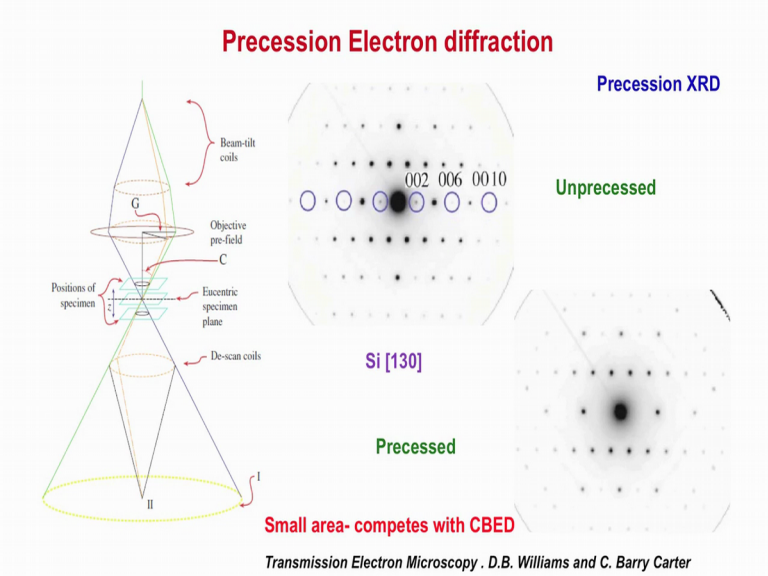

If it is a multi beam you get spots like this, right. And if it is a zone axis this is how the spots you get it, but you see all the spots are equally strong. This is a dynamical scattering.

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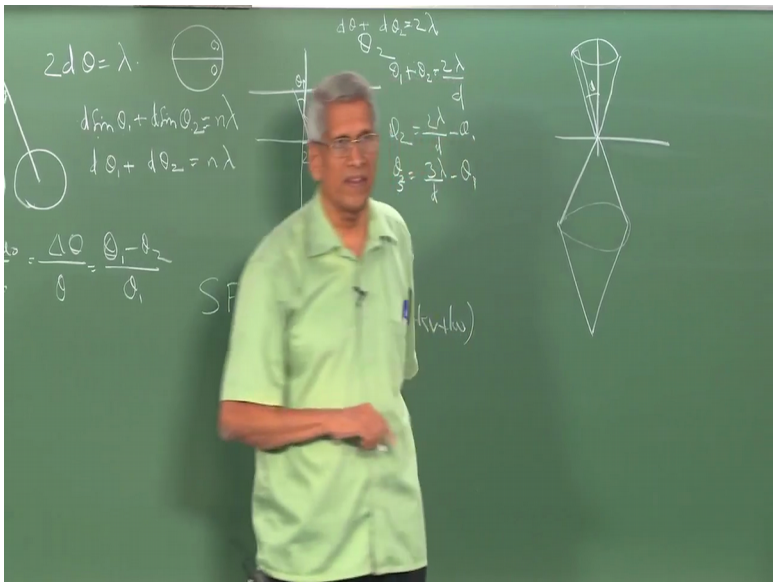

In a precession electron diffraction what is being done is that- I make a small focused beam and the beam I make it fall on the; that is the focused beam is made to fall on this,

this is the optic axis and this beam I rotate it around this over an angle theta. If I rotate it what it will happen? This same beam should rotate in the diffraction plane in the back focal plane also it should rotate like this direct beam. That is what it normally happen.

If I give another one to make it un-process again then what will happen is that all these things will be again brought back to one point again. The precipitate can be brought back. That is exactly what is being done in precession electron diffraction.

Student: How do you bring back?

Which one?

Student: Again to a point.

This is again that beam has to be tilted by applying some voltages to coils you do it in the microscope. These all by applying some voltages you can tilt the beam because electron beam is there either by applying a voltage or by applying a magnetic field you can tilt it; that is essentially what is being done.

Nowadays this attachment is available as a separate attachment which one can retrofit onto the microscope system. What is important is that? Without precession when a diffraction of a silicon is taken you see this, this is how the pattern appears. And these reflections are 0 0 2 should not appear by structure factor consideration it should be absent for silicon, but it is present in these diffraction pattern.

When a precession diffraction has been done then you see this, this reflections have vanished what essentially is being done is that over a region we are trying to collect over the all the various angles we are collecting it when precise it and all of them together. It is essentially from each orientation whatever is the beam which is coming the sum total is what we see it in the each of that spot. Now you see that this spots have completely gone. Now this pattern looks like what we expect for silicon satisfying kinematical condition, you understand that. So, from this we can tell this is what essentially.

Student: Sir, why does spots have a (Refer Time: 54:29) please explain that angle.

Which one?

Student: The like, those reflections which are should not be there over there initially

Yeah.

Student: But it is as good as the.

What is essentially happening is that when we do a precession some of the reflections over all the various angles not only from Bragg and away from it all of them are getting added together in some point, some of the reflections when they add together average over many point weak reflections they become so weak compared to this one that they are just not seen- intensities change. Now, if we look at the intensity between these two it appears to be this looks like what we expect for a kinematical condition.

Student: So, the dynamic scattering point only comes in certain angles.

Dynamic scattering point comes at certain angles, but between the different reflections if you look at the intensity ratios there are some calculations all has been done (Refer Time: 55:27) and that shows that the ratio remains the same as what you expect in kinematical condition. That is a whole beauty of it precession.

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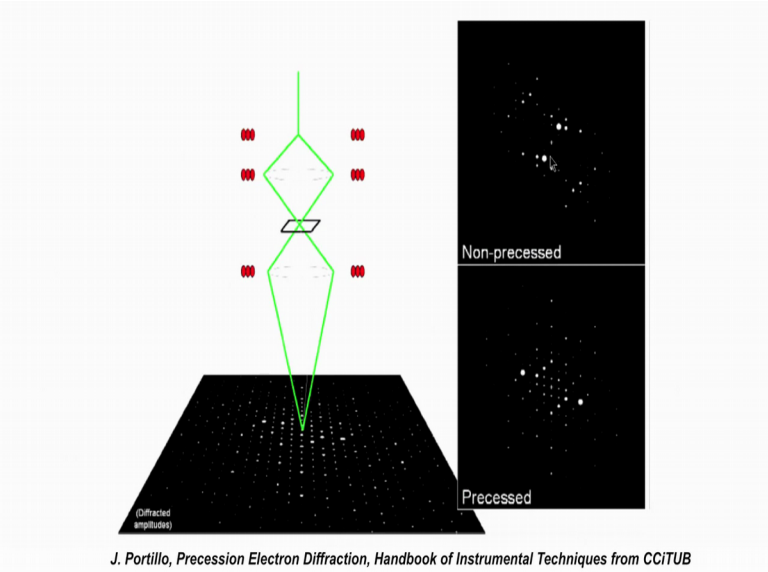

And not only that this is unprocessed one; when you process it since you are rotating many more reflections come into the diffraction spot also. Then, what you expect?

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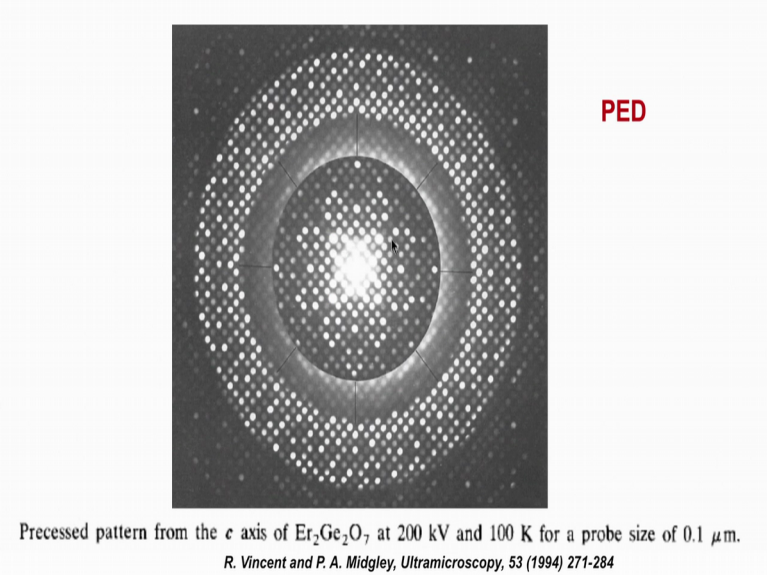

Precessed pattern from the c axis of $Er_2Ge_2O_7$ at 200 kV and 100 K for a probe size of 0.1 μm.

R. Vincent and P. A. Midgley, Ultramicroscopy, 53 (1994) 271-284

And what will be the consequence of it? The precession is that in convergent beam diffraction you got only the first order ring where one or two spots were there. Now you see that the ring has spread out, lots of spots could be seen in the diffractions pattern.

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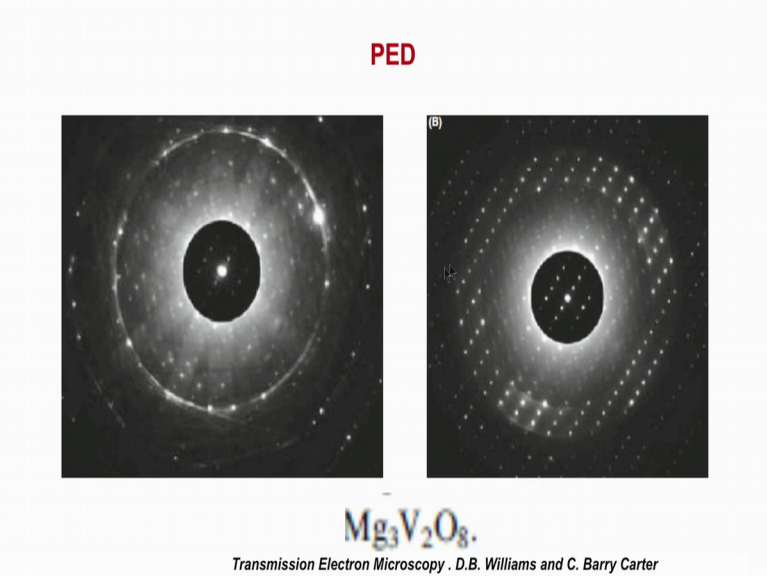

$Mg_3V_2O_8$.

Transmission Electron Microscopy . D.B. Williams and C. Barry Carter

And that is very clear here. You see this, this is the ring when a precession has been done you see that instead of this ring there is a it has become like a bang, correct And now more reflection could be seen compared to this one.

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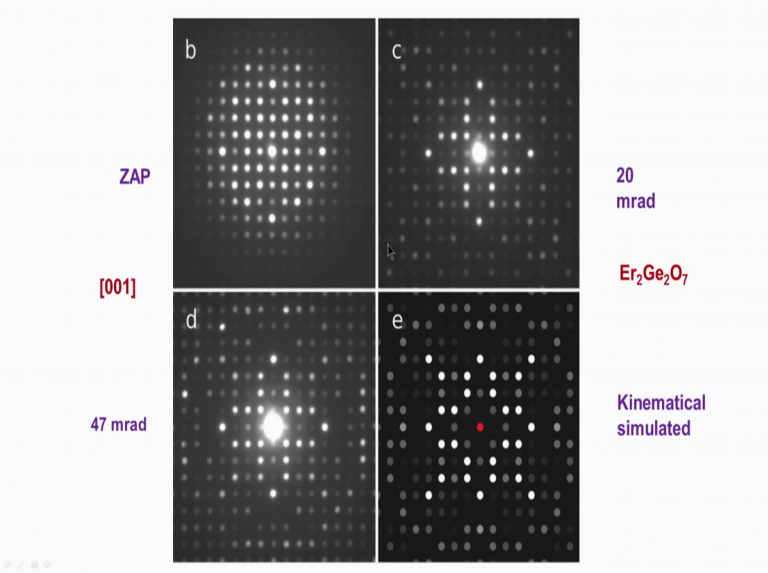

So, analysis becomes much better with this sort of a pattern compared to doing it with. And this is one classic example, where you can see that this is for this sample, this is being done by the people who had invented this technique. This technique was invented in late 90s. They did it for convergent beam, now it is being used for all parallel as well convergent. This is the zone axis pattern for this crystal: 0 0 1 zone axis.

When a precession of 20 millirad is being used the beam is being processed- 20 millirad this is how the pattern looks like. When it is 47 millirad this is how it looks like. And this is the one which is calculated for kinematical condition, without any precession kinematical theory, you apply you know the thickness of the foil, how the spot reflection should appear.

Now you can see that there is a good match between these two, correct. So, the precession diffraction is able to give a lot of information about the symmetry of the crystals. In fact, nowadays it is being routinely done using this to find out point group and space group symmetry of crystal structures. They are all software's, which are available which people are using it.

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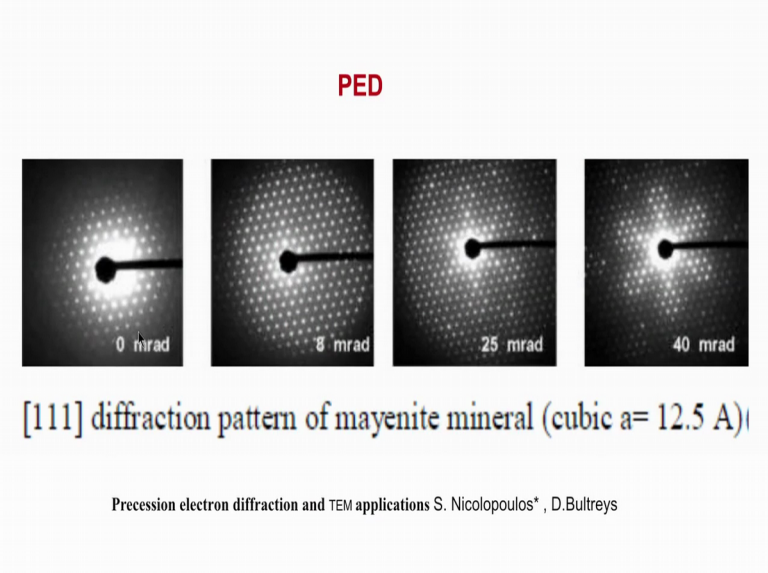

Here like this, for a as we vary the angle of the tilt that is the precession angle if we vary you can see that how gradually the six fold symmetry comes, and if you can do the calculations to find out how the intensity of the reflections will be. So, one can do a matching between a kinematically calculated intensity of diffraction pattern in the precession electron diffraction the intensity of the spots matches with that.

Student: Sir, (Refer Time: 58:25) will have a average of the values of the spots of (Refer Time: 58:29).

No, these are all separate. The precession is like a hollow cone. If I take a beam I do it like this and take a diffraction pattern with respect to an optic axis. 20 millirad means that I have done it very small angle. That is like this.

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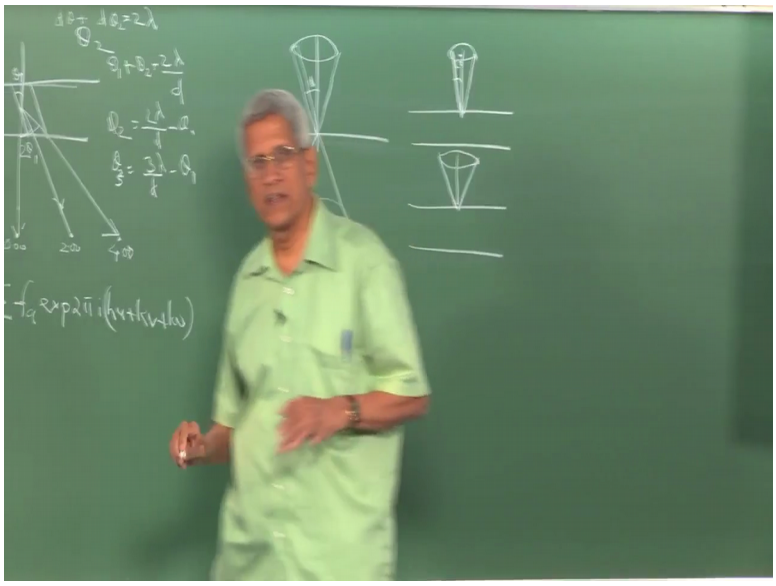

If I have a sample like this I can take one like this, this is some angle 20 millirad you take it; in another this is the one over which it is being rotated. These are two different patterns taken with two different precession. As you increase the precession angle you find that the kinematical effect becomes much stronger.

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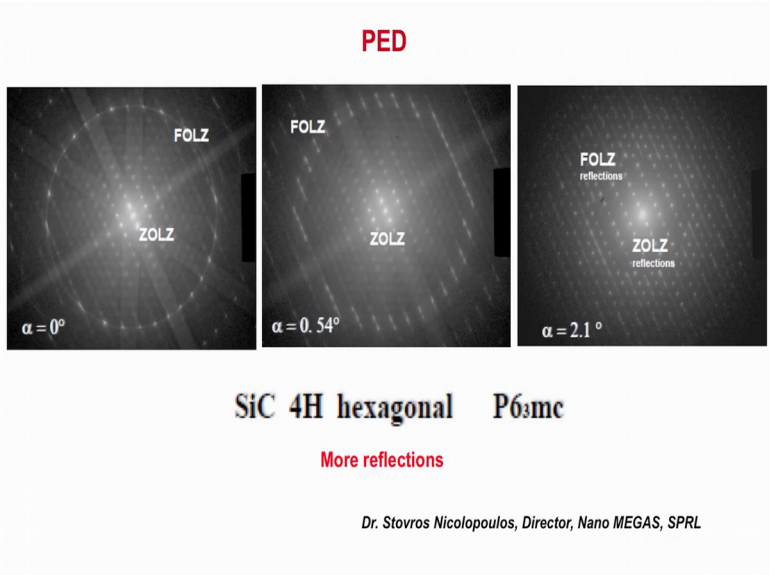

And this is from a silicon carbide hexagonal 1.

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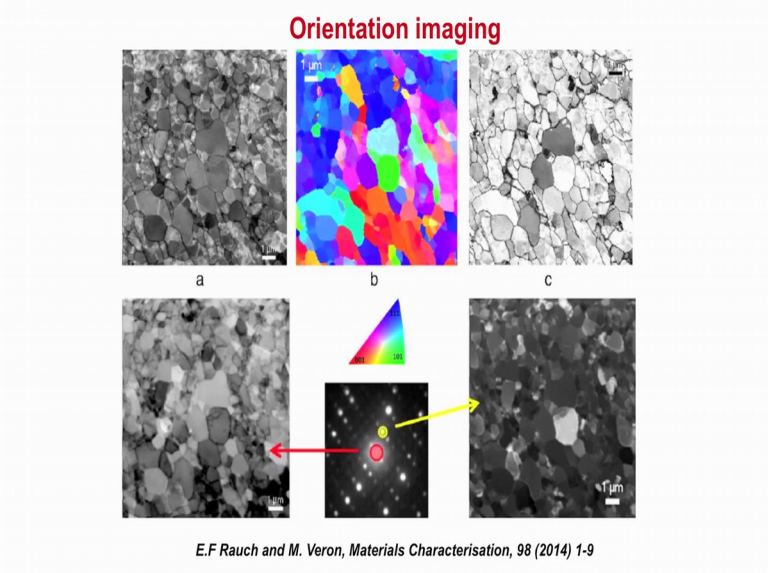

And what is the consequence of this happened is that- with the precession like conventional diffraction pattern when we take it in a stationery beam if it is half zone axis you get very few spots. Even of half zone axis if I do a little bit precession as you had seen more spots could be done. If more spots you get it on a diffraction pattern your analysis and the confidence limit of analyzing the diffraction pattern is better.

That is what is being employed in what is called as an orientation imaging. This is essentially like an SEM the way we do it. At different points on a grain you just take orientation map diffraction take precession diffraction. Then, if you know the crystal structure you can have a library of different orientation, how the diffraction spot should appear on the screen and you do a matching this is all some software driven. And then on that basis you can tell that how different regions what is the misorientation.

Then you can plot misorientation map and generate like what you get it to the DBST exactly the same thing can be done. That means, that for nano size particles are nano grain particle deformed grains you can find out micro texture and all which was not possible in TEM now it is possible.

I will stop here.