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Lecture-2
Materials Characterization
Fundamentals of Optical microscopy

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Hello everyone welcome to this material characterization course in the last two class we are discussing about the diffraction phenomenon in the transmission electron microscopy and especially in the last class we have seen that the diffraction phenomenon what we have seen in an x-ray diffraction is all applicable to this TM also and the only difference we have seen in terms of the wavelength difference between the an accelerated electron beam versus an x-ray beam.

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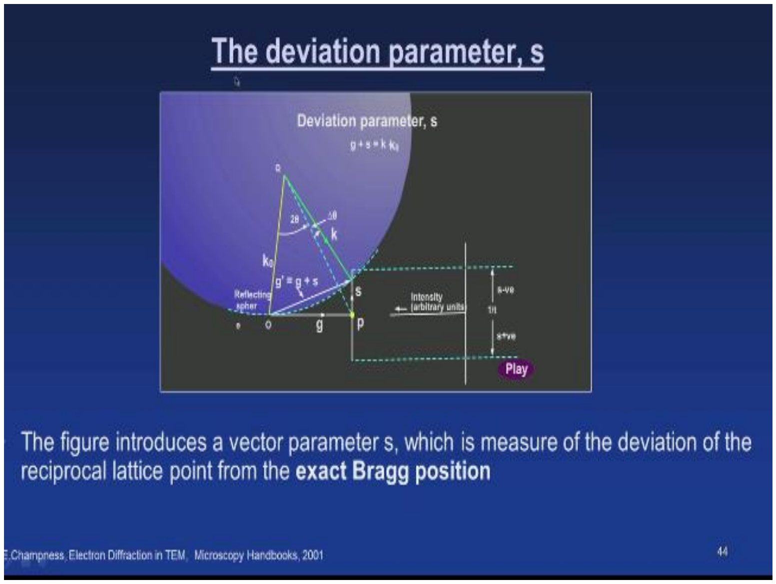

And then we also discussed all this aspects in terms of a waltz fear concept as well as reciprocal lattice concept and then today we will continue in this discussion to complete the mean all the aspects of diffraction involving the vaults fear as well as reciprocal lattice so if you look at the vaults fear and its relation to reciprocal lattice another important aspect can be visualized what I am trying to describe in this slide a parameter called a deviation parameter yes in annex rd.

Also we have discussed that there is something called you have the set of planes which diffracts with exact black condition and which is not exact back conditions so we have discussed those aspects in much more detailed manner and similarly the use of avoid sphere concept and reciprocal lattice also clearly demonstrate this aspect through this deviation parameter S so look at the schematic here is this is the deviation.

I mean this is the vaults fear and then you have the intersection points which is designated as I mean Q and s and I can play as a schematic what you are seeing here is basically the vector form of a Bragg's law that is G =K- K not that we have seen the, the deviation parameter is actually defined as to what extent.

The diffraction is occurring with from the exact Bragg angle so that is the idea so how far it is deviating from the exact Bragg angle so that is what it is depicted here so you see that you look at this Q OS this kind of a point where your diffraction happens exactly at the point S but then suppose if you are the is slightly different are deviating by the amount $\Delta \Theta$ then the point is slightly away from this avoid sphere surface so you can see that the intensity profile also is drawn accordingly so you have two aspects where you have the maximum intensity you have a negative as well.

As a positive yes over the I mean the in the thickness of the diffracting volume and accordingly you can write that know the new formula for this vector form of bragg long prime is equal to g plus so since it is a positive s now it is G+S . so the figure introduces a vector parameter s which is a measure of deviation of the reciprocal lattice point from the exact Bragg position so you have some a quantitative data hereto measure this how, how far the diffraction takes place away from the exact Bragg angle and so on so this particular concept we will be using this even we can demonstrate in terms of in a diffraction experiment.

When I discuss the kikuchi line I will be able to demonstrate particular peak positions where you can see exactly whether it is a positive S or a negative S and also where exactly you can see S=0 all these situations you can demonstrate easily with the MA kikuchi pattern. okay this is the actually the allowed condition for the I mean conditions for the allowed reflections in the most of the crystal systems and where you talk of me talk about a different.

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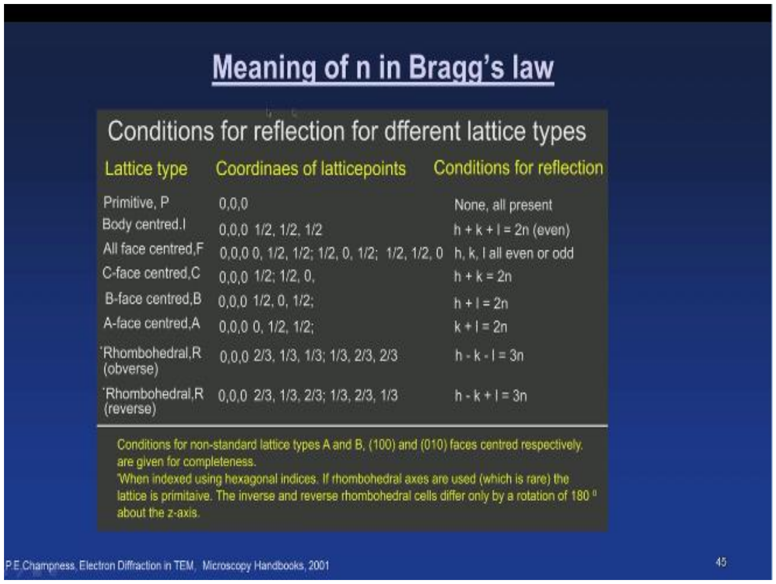

I mean unit cells where you have the selection rules which is present we will use this table in an appropriate time when we when we when we do the I mean CB ed and then other indexing procedures for time being we will skip this we will come back to this table once we go to this CB ed and so on.

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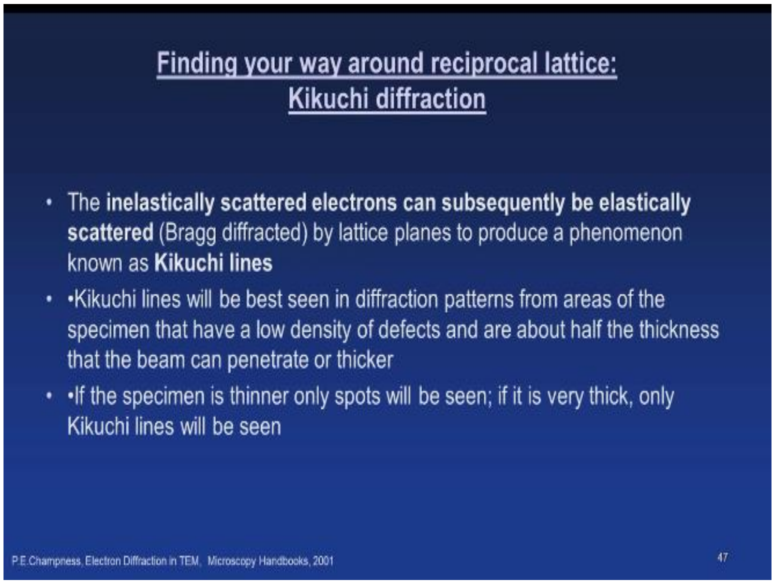

So now I would like to discuss something a little more detail on a kikuchi pattern in a transmission electron microscopy so we have some background about this kikuchi pattern in even in scanning electron microscopy a lecture and one important point. We have to understand is the diffraction whatever we have discussed so far dealt with elastically scattered electrons now we are going to talk about the inelastic scattered electron.

So that is the primary difference then we will like to look at the initial remarks the inelasticity in a sari in elastically scattered electrons can subsequently be elastically scattered that is bragged affected by a lattice planes to produce a phenomenon known as kikuchi lines please remember this is in elastically scattered electrons. That means the electrons has much I mean lost their energy and you they will form in a form of a diffuse spot in an electron diffraction when such an electron further subjected to a elastically scattered that means without losing further energy if it undergoes a Bragg diffraction it produces a kikuchi lines.

So that that point you have to remember kikuchi lines will be best seen in a diffraction patterns from areas of specimen that have low density of defects and are out about half the thickness that the beam can penetrate or thicker so what it means is you should in order to obtain are in order to visualize a kikuchi pattern you need to have a sample where the defect density is minimal as well as it should be sufficiently thick your sample should be sufficiently thick enough to visualize this effect otherwise you will normally see a a single crystal diffraction pattern rather than a kikuchi pattern.

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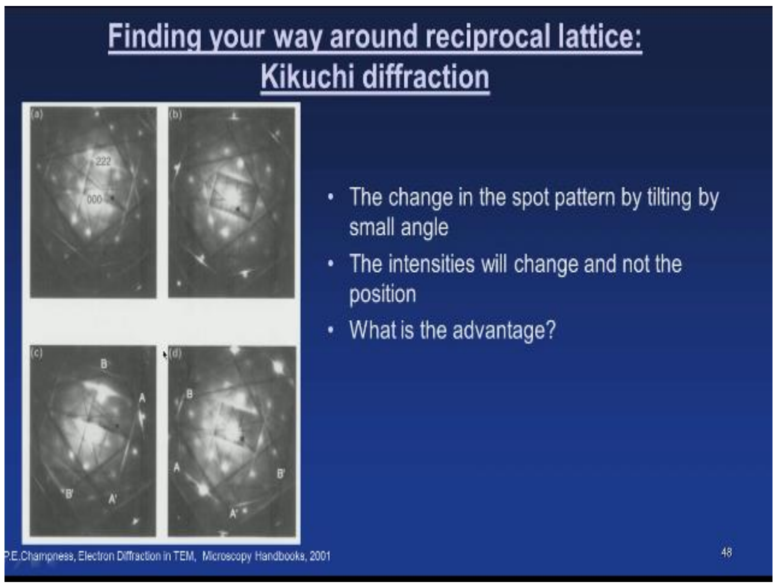

So that is the another reference one can have if the specimen is thinner only spots will be seen if it is very thick only kikuchi line will be seen so okay so look at this electro I mean electron diffraction pattern where we are going to discuss about the, the exact Bragg condition as well as the deviation from the exact black condition and so on so what we are now going to discuss is finding your way around the reciprocal lattice so we are looking around the reciprocal lattice.

And we will see what is the effect of this thickness specimen thickness and then tilting also we can be discussed so the change in the spot pattern by tilting by small-angle the intensities will change and not the position what is the advantage so you can just do this kind of a simple tilting experiments to in order to obtain to beam conditions or if you are interested in particular exciting a particular diffraction positions and so on so look at this for diffraction pattern carefully you have both a spot pattern as well as a kikuchi line.

What you have to remember in a in a kikuchi pattern as we discussed in the previous diffraction lectures you it is it is the 2D projection of a parabola so you have one pair of a bright line and a dark line so you can see that this is a bright line in a dark line we have it has been marked here a dash BB dash so this is the pair like that you can always call a pair kikuchi pair off lights are you can say kikuchi band and so on.

You can induce this can be described in many ways and what you are seeing here is you see that the 2 2 to set up plane I mean the diffraction spot designated as to 2 2 has been excited more and then you have after a small tilt this band has moved into the center that means you have the, the two to two and this the corresponding the negative indices to bar to bar to bar indices have the equal intensity here and this is this condition belong to your deviation parameter S=0 and you have you see that the, the two two, two spot is now excess in intensity in this tilting exercise.

So that means it has got or we can say that the bright line has further away from this 22 to spot and here you see that the the excess intense line it is further the other side just opposite right side 22 22 spot so these two conditions belong to a positive is as well as n- s so you have positive deviation from the black condition this is a negative deviation from the black condition.

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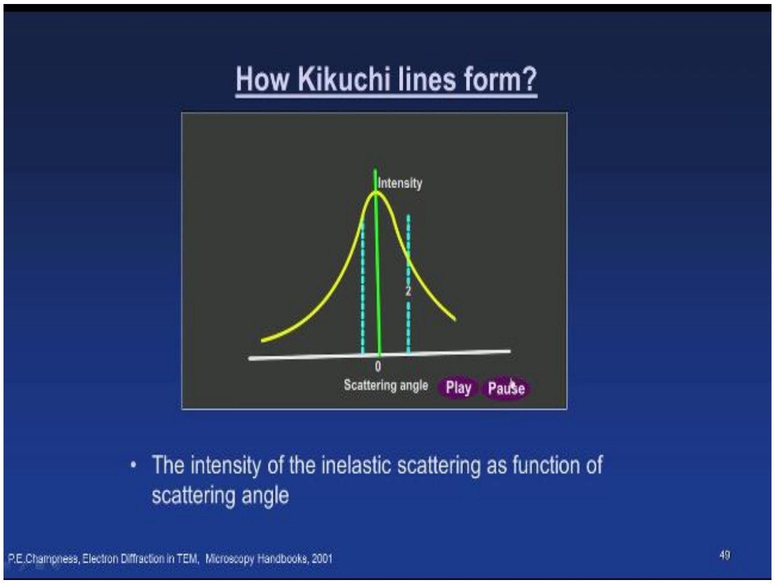

So this is what I just said a kikuchi line diffraction can clearly demonstrate the, the deviation parameter and so on and we can just see what is the origin of this kikuchi line from how the KPG line form so look at this schematic value suppose if you are looking at one in elastically scattered beam and then we are now talking about a to raise way one and then the rate to please remember the Ray one intersects at this point which is close to the imax and the ray to intersects the, the profile at this point which is slightly away from this way .

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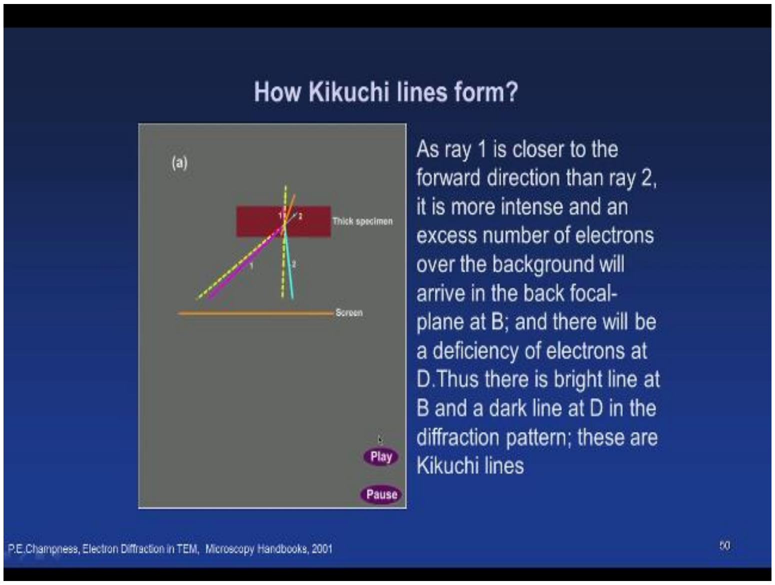

Where the way one intersects here so it is far further from the imax this is closer to the imax the intersection point so keep that point in mind then we will continue the discussion so to explain that kikuchi line this is a schematic which is shown assume that this is a specimen tick specimen and this is an incident beam and you have the ,the diffracted beam which represented as G and D and you can see that this is the way one and then this ray tube with reference to the, the previous schematic on the, the intensity profile of in elastically scattered beam.

You have ray one and ray to suppose if you assume this then we have the remarks which is pertaining to these two rays are as follows as ray one is closer to the forward direction than the ray to it is more intense and an excess number of electrons over the background will arrive in the back focal plane at B and there will be a deficiency of electrons rd thus there is a bright line at b and a dark line at din the diffraction pattern and these are all kikuchi lights so there is a very simple not complicated explanation.

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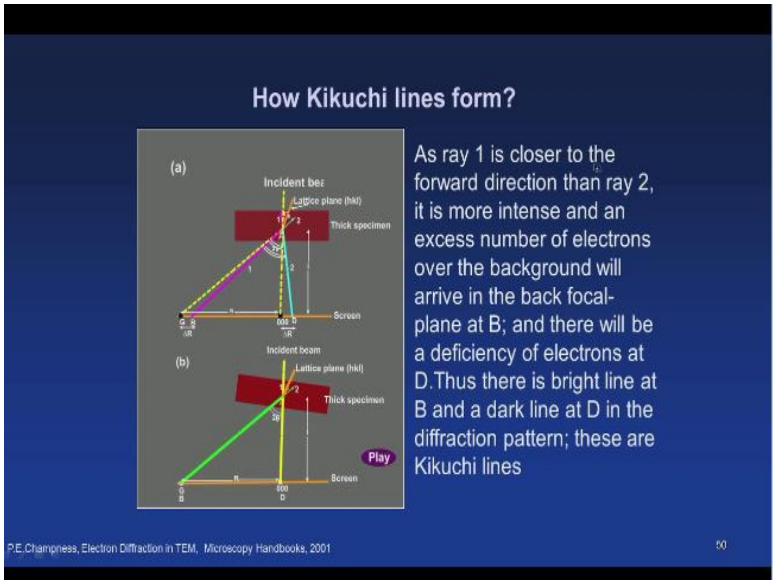

Just with respect to this unless an elastically scattered intensity profile namely ray1 Andrade to how it reaches the, the screen and then the, the brightline and dark line is simply X I mean explained on the basis of the intensity of each of this slide that is the excess of electron and then less I mean I would say that excess line are a dark line.

We can say that the bright line or a dark line we can say that so if you till this specimen in a small angle you can bring back this G and D coincide with your transmitted beam and the, the diffracted beam originally so this is a one simple explanation how the kikuchi line will form the diffracted race actually form cones of semi and 90 minus θ called coastal codes what we see in the diffraction pattern is pair of parabola swear the cones intersect the awards fear the parabola appear as straight lines in the diffraction pattern.

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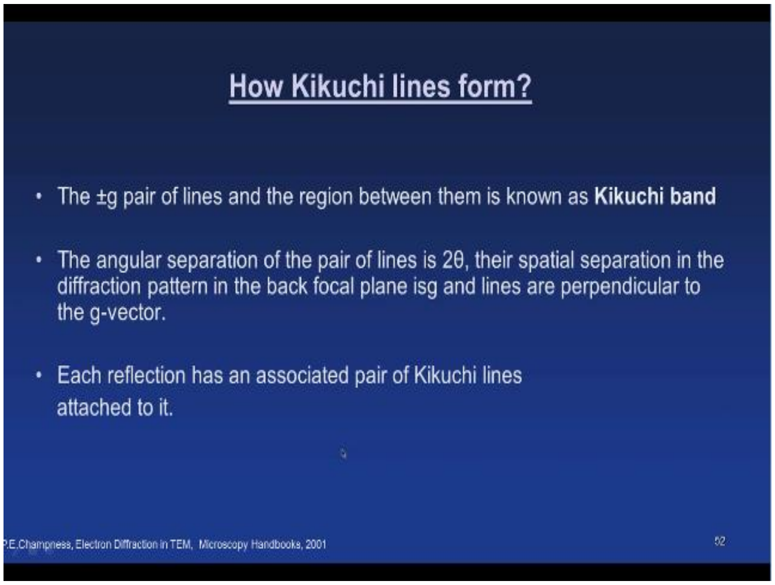

Because the angle involved or very small the plus or minus g pair of lines and there on between them is known as kikuchi band the angular separation of the pair of line is to θ their spatial separation in the diffraction pattern in the back focal plane is G and the lines are perpendicular to the G vector each reflection has associated with associated pair of kikuchi lines attached to it and this is demonstrated in this schematic diagram.

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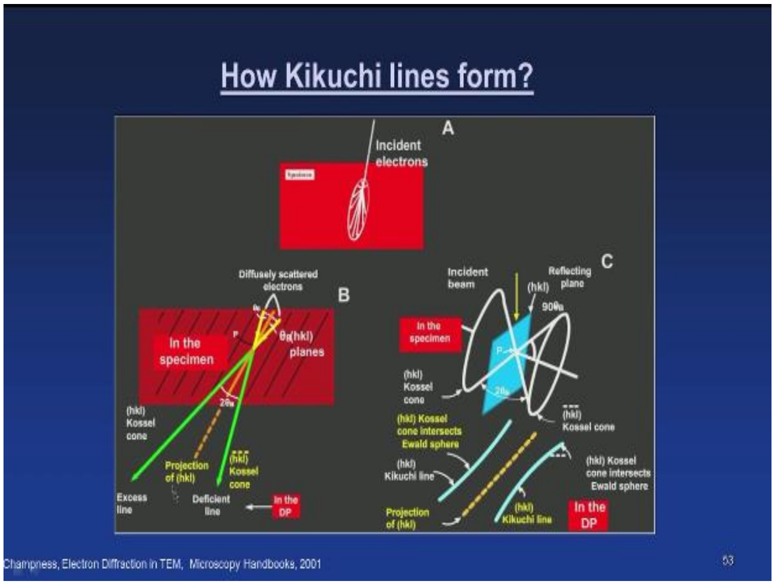

This schematic we have already seen in the x-ray diffraction as well so just for a recap so how this supposed if you have the HKL plane and this is the order diffracted beam from the both the sides since we are talking about everything sample I mean you have both sides the, the pattern comes and then when this parabola intersects a 2d plane here you see and these two param I mean cones are called a coastal cone and which intersects the vaults fear appearing and I get equity line here so you have a positive I mean positive hkl and as well as a negative H scale we can say that and similarly you can you can you can visualize these line formation through this simple ray diagram.

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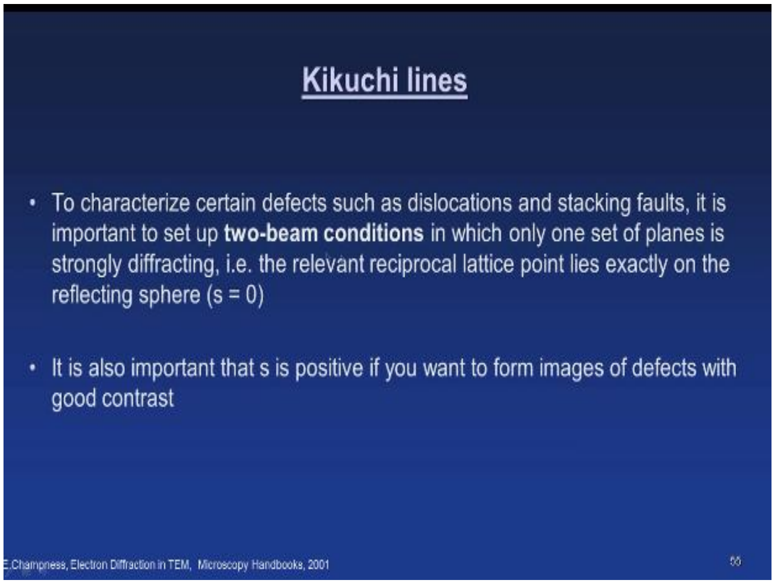

In the specimen where the electron diffracts in this form of excess line as well as deficient line what we have shown in the diffraction pattern if the specimen is tilted by a small angle α the lines will move a distance R across the pattern on the screen and from the simple geometry we can say r is equal to Lα where L is the camera length to characterize certain defects such as dislocations and stacking faults it is important to set up to beam conditions in which only one set of lanes is strongly diffracting that is the relevant reciprocal lattice point lies exactly on the reflecting sphere that is equal to zero it is also important that s is positive if you want to form images of defects with good contrast.

So now you appreciate from these two points why we talk about kikuchi lines I mean why it is important how it can be used and also the importance of the deviation parameter s so I have just shown how the deviation parameter can be shown whether it is a positive yes or a negative s with respect to the intense line whether it exactly intersects the, the for example the 2 2 to set up I mean plain diffraction spot.

Where you have the excess line going further or the closer to the transmitted beam the, the condition of SS decided R it can be visualized practically and this condition is important to obtain the images of some of the import defects with a good contrast so that is how these parameters are used and at least these are all the preliminary are very basic use of usage of this a techniques.

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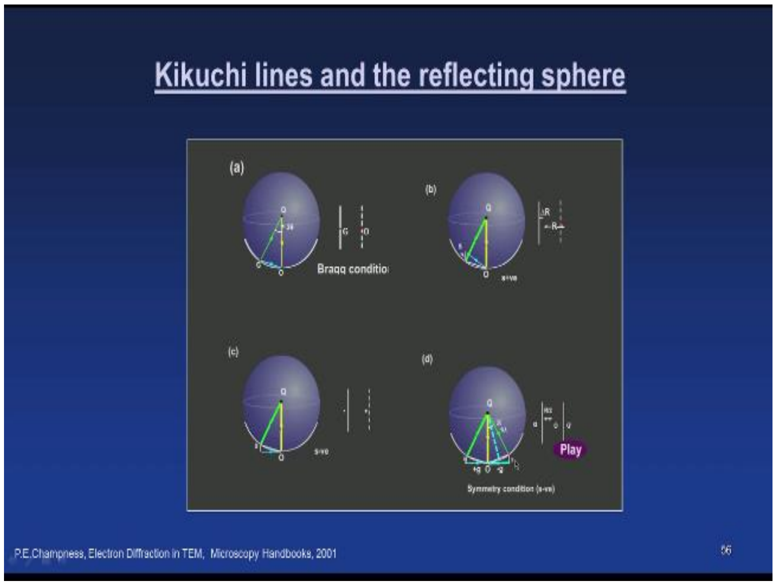

So you may wonder why we do all this so we are not discussing the, the complete details of all this techniques and the phenomenon just for a basic idea you should have how this kikuchi line forms and what is the use of it too to begin with so we can now look at the sum of the schematic where it clearly demonstrates the deviation parameters with respect to reciprocal lattice point and then exact, exact black conditions so what you are now seeing is this is a brag condition and you have a positive yes that means you are actual reciprocal lattice point is completely inside the sphere.

I mean and then you see that the, the corresponding effect on the hit which align on the diffraction pattern and this is a negative s then this part will come out of the vault sphere and then this is the point where it is in the equiv-distance I have shown this kind of situation in an actual diffraction pattern in the pattern B where I have shown that it is equal distance from the both positive as well as negative.

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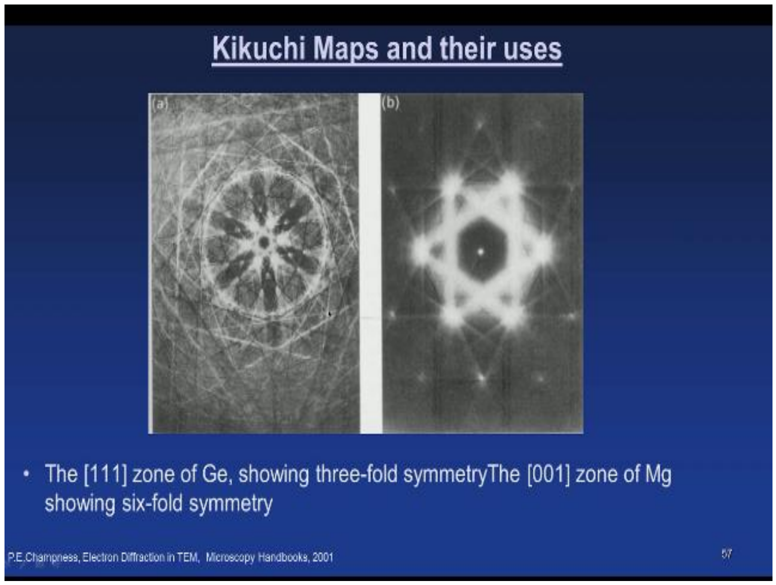

So this is different from the exact black edition do not confuse these two so this is you have a positive and negative effect s parameters are all equal magnitude at this orientation here it is a completely oriented towards exact drag condition and so on so other some of the uses of kikuchi maps is to just to find out the symmetry you can just by looking at the kikuchi maps you will be able to tell what kind of symmetry the your crystal has for example if you have the 111 zone of germanium showing a threefold symmetry.

This is threefold symmetry what you are seeing here is this is threefold symmetry and the 001 zone of magnesium showing a six-fold symmetry so these are all some of the immediate application just by looking at a diffraction pattern you, you are able to obtain a basic information about the system crystal system and so on they are very powerful in that manner so you can also look at the zone axis when the electron beam is close to the beam axis the position of the zone axis relative to the undefeated beam can readily be identified.

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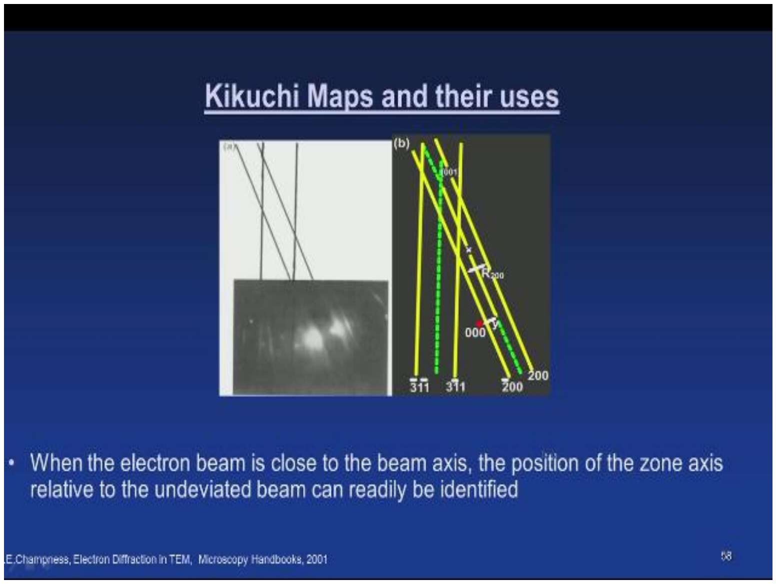

So look at the zone axis identification of zone axis also will be easier once you have some details about this kikuchi maps and this is a schematic of a kikuchi map of for a diamond cubic crystals which I have already shown this for the completing this section I am just giving this example again so the kikuchi lines and the kikuchi maps are one of the most important aids we have when orienting are determining the orientation of the crystalline materials identification of orientation of the specimen is essential for any form of quantitative microscopy.

So the bottom line is if you are interested in carrying out quantitative microscopy in electronic transmission electron microscopy the knowledge of kikuchi lines and there you know identification are operating this for a exact condition to generate this is very essential whether you are analyzing the dislocation budget vector by diffraction contrast imaging grain boundaries with lattice resolution are measuring chemistry variation by electron energy loss spectroscopy our x-ray energy dispersive spectroscopy they are especially useful.

When combined with the map of zones and poles that is direction and plane normals on the stereo graphic projections so using the stereographic projections as I told you yesterday you will be able to identify the plane normals which are very parallel to the zone axis if you recall in this today's lecture where I showed how the, the planes which are connected to the I mean if the plane which are parallel to the zone how the diffraction pattern exactly appear the diffraction pattern appear at the 90^{O} to the, the plane orientation so that concept is exploited simply using a stereo graphic projection if you have a zero, zero one zone then the, the plane normal which all intersects the, the periphery of the stereographic projection will be normal to the zone axis.

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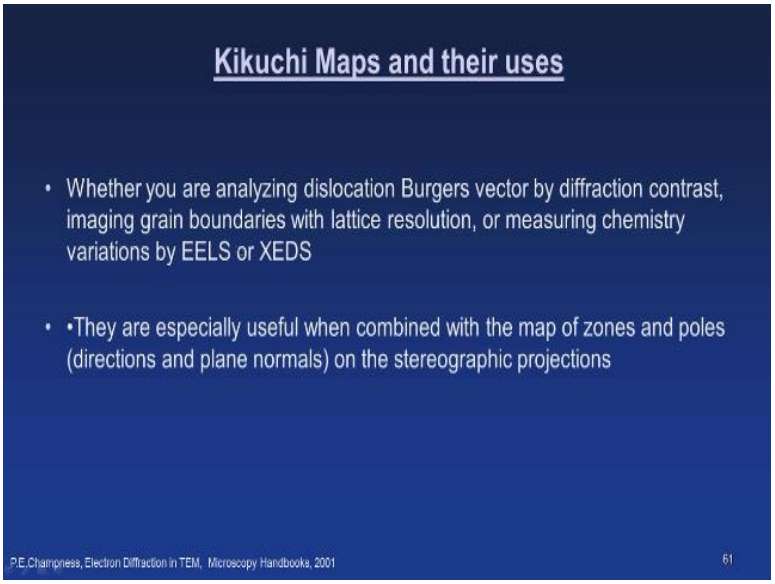

That means all the planes will be parallel to the zone axis so that also can be exploited in combination with kikuchi maps to do quantitative analysis so that is the information you should have at this point of time so it is a summary the kikuchi lines consist of an excess line and efficient line in the diffraction pattern the excess line is further from the direct beam than the deficient line the kikuchi lines are fixed to the crystal.

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So we you can use them to determine the orientation activity the trace of the defecting planes is midway between the XS and the deficient lines so these are all some of the key points one can remember now we will go on to the another important diffraction phenomenon called converging beam electron diffraction in T so we have some background for this topic when I introduce the transmission electron microscopy we talked about some of the instrumentation operations like parallel game as well as converging beam and then the name itself clearly says that it will give you very a small probe.

Whether it is a microprobe or nano probe and then lot of you have the teaching possibility in a TM then you have the advantage of looking around a large volume of reciprocal space please watch my words a large volume of a reciprocal space not the real space so you will get lot more information using tilting experiments and converging beam electron diffraction we will see what it is need good understanding of crystallography and space groups to follow this technique.

So you have to have some basic knowledge on crystallography and space group point groups to exploit this technique that is the prerequisite otherwise this is very difficult so in principle it is a straightforward matter to decide whether or not the crystal is cubic hexagonal or has a lower symmetry from the observation of the geometries so that is so powerful by looking at the pattern you will be able to decide all these things.

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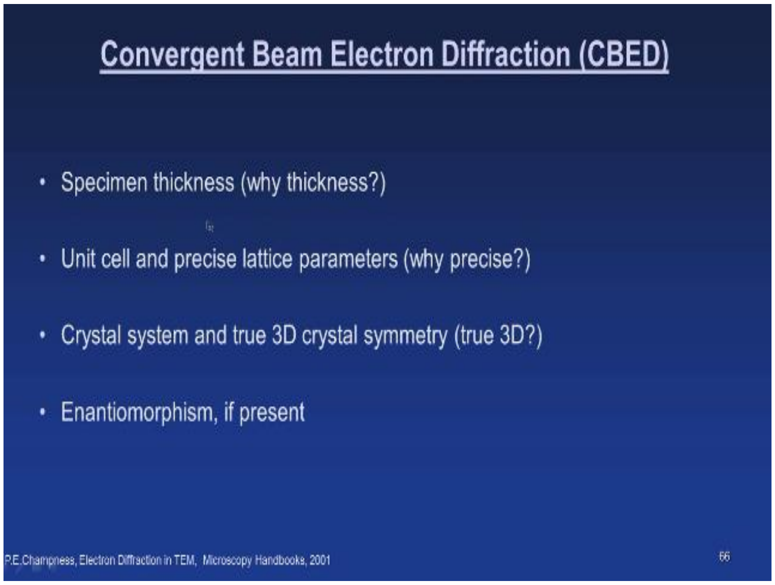

If for example load diffraction patterns with the four or six fold symmetry are found the crystal cannot be a cubic and hexagonal with the large tilt angles that are available in modern TMS it is possible to examine large volumes of reciprocal space so you will be able to extract lot of information from the very small volume that is a geometrical work information or crystallography information.

From the very small volume that is what it means so what are the typical things one can get from this technique specimen thickness unit cell and precise lattice parameter and crystal system and true 3dcrystal symmetry and initio morphism if present so all these things can be analyzed using this technique we are not going to demonstrate all these things because of the that is not the scope of this course but you should know what is this technique and what is the meaning of it what it does and what is the use at least to that extent you will be able to have some idea.

So when a convergent electron beam is used to form a diffraction pattern a range of incident angle leads to a significant excitation of the reflections from the higher-order lava zones or holes if the electron beam is exactly along the zone axis of a crystal it is clear that the spacing H star why H star we are now talking about a reciprocal space of the reciprocal lattice layers perpendicular to the electron beam the zone axis repeat in the crystal can be derived from the radius G of the higher order la-based zones.

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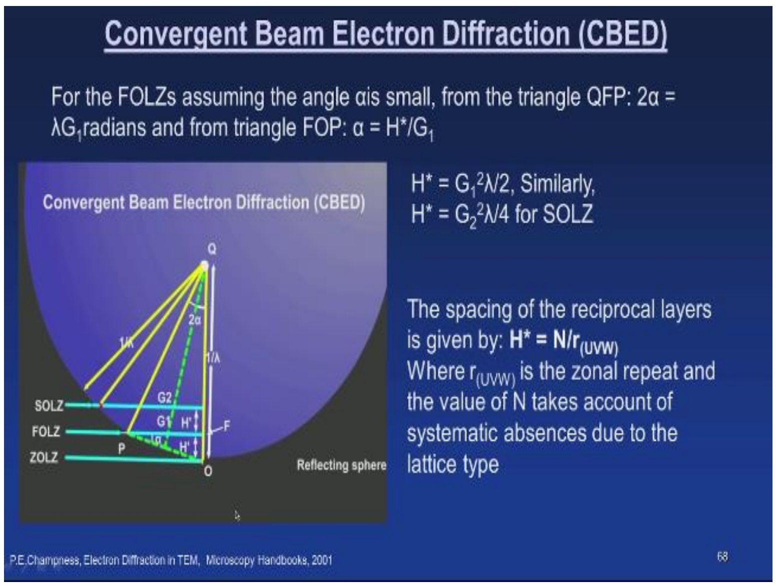

So look at this schematic again we are talking we are exploiting the evolved fear concept as well as the reciprocal lattice here just what we have just seen in the kikuchi lines also for the first-order lava zones assuming the angle α is small if you if you assume this alpha is small from this geometry you look at this, this is the you know this is an evolved fear then whatever the, the point which are intersecting will form a diffraction pattern and you just look at the q FP qf p this triangle and if you assume this α is too small then we can show that to α is equal to λ g 1.

So what is g one g one is this, this distance and λ is 1 by λ here so it is to α is equal to λ g 1 radians and from the triangle f 0 p you can say that f 0 p this, this small triangle you can write α is equal to H star by g one so from these two relations you can write H star is equal to G 1 square

λ x 2 for the folds line that is first order lava zones you can write like this and similarly H star is equal to G 2 ^{2} λ x 44 souls that is second order lava zones so you have you can do this for a second order lava stones which interacts from this.

This point so please understand what we see normally the diffraction pattern is zero order law is owned and then you have first order lab in zone second order law is and so on as you claim this you know the later like this wherever the you have the intersection point here you will see the higher order lavas own pattern so we will see some practical example so the spacing of the reciprocal layers is given by H star is equal to n divided by r u v w where are u v w is the zonal repeat and the value of n takes account of the systematic absence due to the lattice type.

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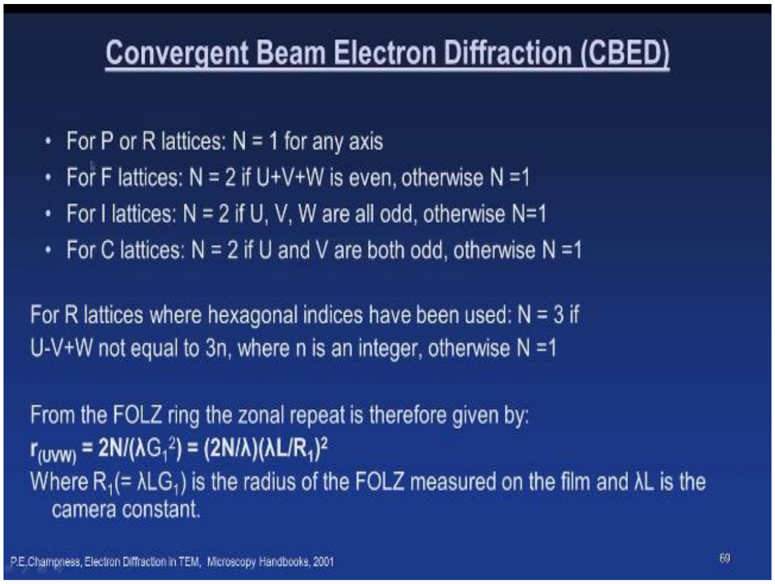

This is say again a selection route if you remember we have just shown some table where all the crystal systems where do you know the systematic absence of reflections will be tabulated so that table can be referred to I mean to arrive at this conclusions what is that absence systematic absence due to lattice type whether it is a cubic or above bcc or FCC and so on so for a primitive lattice n is equal to 1 for any axis for in a face centered lattice n is equal to 2 if u plus V plus W I seven otherwise n is equal to 1 for a body centered lattice n is equal to 2.

If you v w r all odd otherwise n is equal to one for a see lattice n is equal to 2 if you and B are both or are otherwise n is equal to 1 the same information we just table at ed in the previous slide I have brought it back for the reference for or lattice where the hexagonal indices have been used n is equal to 3 if u minus V plus W naught equal to 3 and this is a selection rule where n is an integer otherwise n is equal to one from the first order law is old ring the zonal repeat is therefore given by our UV W is equal to 2can divided by λ G 1 square which is equal to 2 n divided by λ into λ L which is a camera constant divided by $r1^2$ where r 1 is the radius of the foals measured on the λ is the camera constant so you have the general relation to obtain a different lava zones basically.

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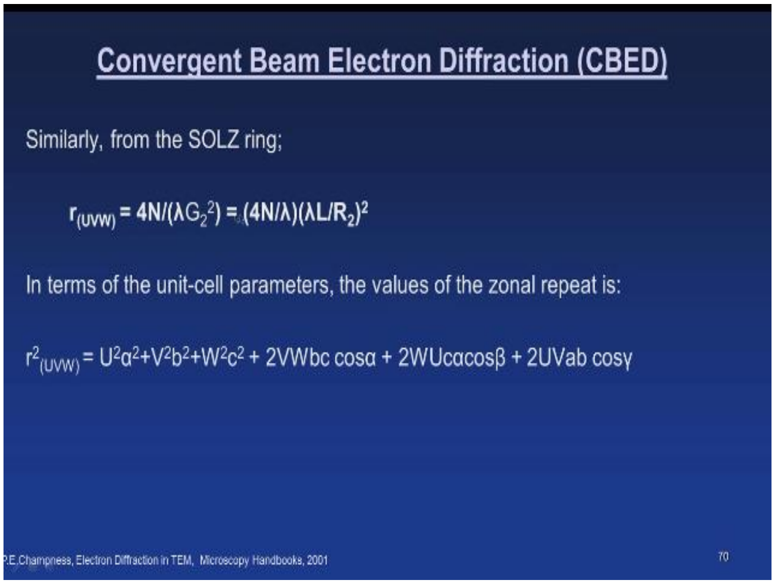

Similarly you can write for the souls ring a for nr is equal to 4 n divided by λ g 2square is equal to 4 n by λ into λ l by r 2 whole square in terms of unit cell parameters the values of the zonal repeat you can also represent the, the zonal repeat in terms of unit cell parameters then you can put it in this formation r square is equal to u square α square plus z square b square plus w square c square plus 2b WBC cos α plus 2w you see α cos beta plus 2 UV a b cos gamma so this is a general information about the zonal repeat please remember this is with respect to the vault sphere intersecting the reciprocal point so and it is done with the converging beam electron diffraction so now we will see some typical example again the before.

We go to the example this is the schematic how whatever we have just discussed this is an another form of putting it so this is a reciprocal lattice rods and then you have the souls and then folds and soul sand so on you have the all this you know sphere which with the different, different tilting conditions you will be able to see the different, different lava zones and this is the typical CB ed pattern you see the zero-order lobbies on the first order lavas own you can see the second order love it zone very faintly which is coming here so look at this slide you have 0 0 1 CB ed pattern from the Espinel and what you are seeing here is it is a pattern obtained with the microprobe with a 30 micro meter conventional aperture be is taken with the coastal conditions with the nano probe size of 100 micron condenser aperture.

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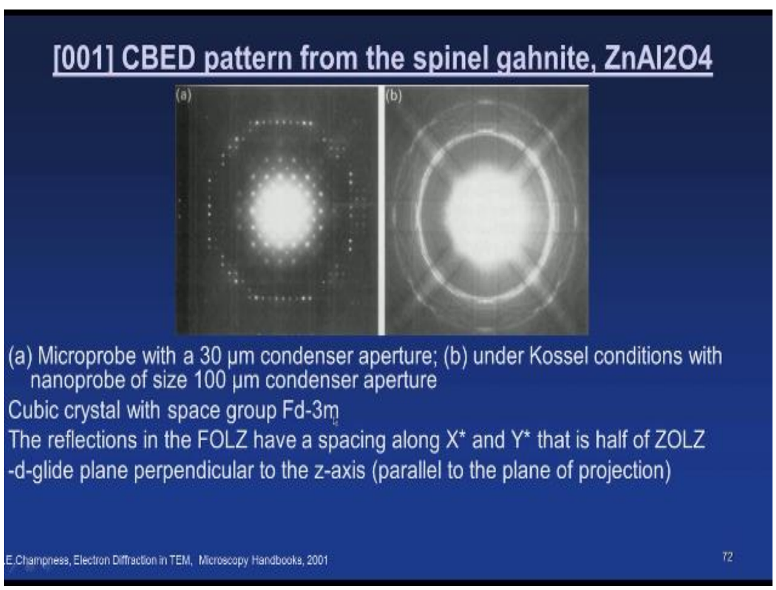

So the pattern is for this panel which is having a cubic crystal with the space group f d 3 bar m so the reflections in the folds have the spacing along x star and y star that is half of souls so what is that stated here so this is the, the first order own pattern here what is given the, the orientation suppose you consider this an X direction and this is a no I direction the spacing along X star and waste our that is half of souls that means the distance here what you see here it is only half in the first order lava stones in both x axis as well as the y axis so that indicates a presence of a glide plane perpendicular to the z axis that is parallel to the plane of projection.

So you have the glide plane in parallel to the plane of projection that is this projection so you have the you can identify a glide plane with this kind of information so this is one typical example we can we can just I just want to give you by looking at a simple a CBE d pattern you are able to get a very important information about the space group and, and phase group information from the of the crystal so that is the powerfulness of the CB ed and we can see one more example a 010 CBD pattern from the a synthetic magnesium germanium oxide the reciprocal lattices monoclinic in shape the foals is displaced by half a repeat in the X direction so the schematic of this is here because the pattern is so V here so you see that even the zero-order labra zone is is in slightly Knoblauch nature.

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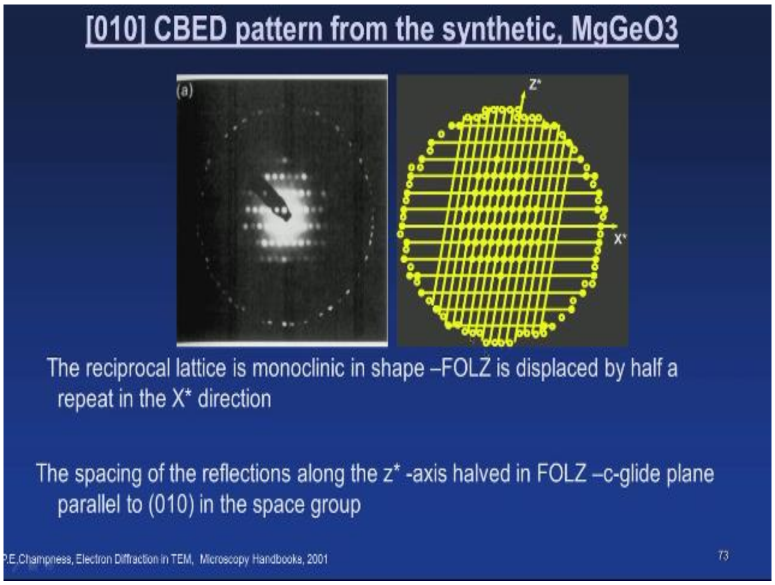

And then the repeat distance in the X direction as well as the other direction he is half of the zero-order reflection so that again gives the information of C glide plane parallel 2010 in this space group see you see this is the usefulness of a CB ed just by looking at a pattern and then you have you will be able to some of the space group information of the particular given crystal so we will not get into the details of this technique I just want to tell you what is CB ed how it is formed and what is the usefulness of this and in an advanced characterization techniques as I said when you do a quantitative microscopy here again you can you can use this C ed to after the precise thickness of the sample.

And precise lattice parameter and orientation and then in addition to this space group informations so these are all the typical applications of CB ed and again the table I have just brought back for the reference so this is the, the diffraction group symmetry seen in CB ed pattern from the different zone axis you will see that this is a point group and this is the different, different u v w directions where you will see all these informations this is being tabled here you can find this also in most of the transmission electron microscopy at standard textbooks and similar table regarding the relationship between I mean the diffraction groups and the full symmetries of CBD patterns are also tabled in most of the textbooks.

Just for any reference I have given so with that I want to finish this diffraction discussion in TM so what to summarize a diffraction in tem what you have to appreciate this diffraction is very powerful tool in transmission electron microscopy so it is I would say it has been completely exploited in this technique that is why at TM itself it become most powerful technique because of this aspect of the microscopy so if you have a thorough knowledge on a crystallography and symmetry that is a group theory.

You will be able to exploit this technique otherwise it has not been used to the fullest extent possible so now we will move on to the other topic in the TM that is an email we will just look at what are the kinds of our types of imaging possible we just mentioned very briefly what does mean by a bright field and a dark field in during the instrumentation details of the microscope and we will also look at the types of techniques which gives a different images and what are the other details we will see it in the next class thank you.

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