#### Indian Institute of technology Madras Presents

## NPTEL NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING

## Lecture - 34 <u>Materials Characterization</u> <u>Fundamentals of Transmission Electron</u> <u>Microscope</u>

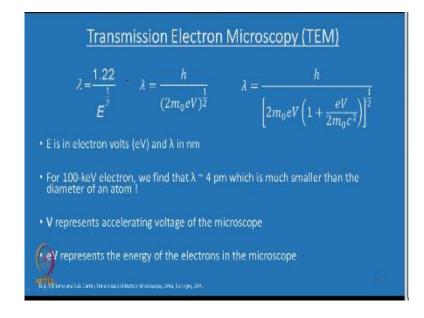
# Dr. S. Sankaran Associate Professor Department of Metallurgical and Materials Engineering IIT Madras Email: ssankaran@iitm.ac.in

Hello everyone welcome to this material characterization course, from today's class onwards we will discuss about transmission electron microscopy and we have found so far the characterization in terms of x-ray diffraction and also you use two electron microscopy that is scanning electron microscopy and so on and then we have enough background to take up this another advanced variant of microscopy in terms of electron optics and also in terms of diffraction physics.

So with all that background will be able to appreciate this microscopic technique also without any problem I believe and nevertheless I will reemphasize some of the concepts then and there whether it is a diffraction physics us or an electron optical system wherever we need to emphasize with respect to a transmission electron microscopy so I will just begin the introduction of the transmission electron microscopy course what I will do is I will first introduce a very gentle introduction I give about this technique what is that people expect or what people want to do with this microscope what are the information they get.

And then I will touch upon or I would say that we will refresh whatever the basic diffraction and then basic physics behind our optics electron optics once again we will touch upon and then I will take you to the instrumentation details how what are the various parts and the functions especially and then how they facilitate the imaging system and so on then in the later what I will do is I will take to the diffraction in tem in much more detail what are all the possible experiments one can perform in TM which exploits the diffraction phenomena and what kind of information you get and then I will focus on imaging part and imaging we will also discuss about what kind of contrast mechanisms very briefly because it is a part of a characterization course which I would like to finish in our 10 to 12 lectures the bay the fundamentals of all this transmission electron microscope.

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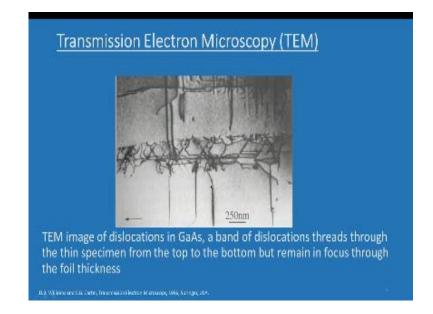


So if you look at this in the fundamental of electron optics I will I would like to look at this expression is much more carefully this kind of expression can be derived from the d Broglie hypothesis and he showed that in an energy of the electron can be related to lambda or you can say that you know if the acceleration voltage increases you can bring down the  $\lambda$  to the very small value.

Of course this where this expression is value I mean valued or you can use this if you ignore the relativistic effect and this is the basic relation but if you if you do not ignore this relativistic effect then you need to go to this kind of an expression because at the very high voltage the electron travels about 100 times the speed of light, so we cannot ignore this relativistic effect and

if you ignore this relativistic effect then you can use this relation and then you can approximately derive  $\lambda = 1.22/e^{1/2}$  where e is the electron volts, please remember electron volt is the energy of the electron in acceleration and  $\lambda$  is in nanometer for a 100kilo electron volt electron we find that  $\lambda$  is approximately equal to 4 pico meter which is much smaller than the diameter of an atom and we represent the acceleration accelerating voltage of the microscope and EV represent the energy of the electrons scope.

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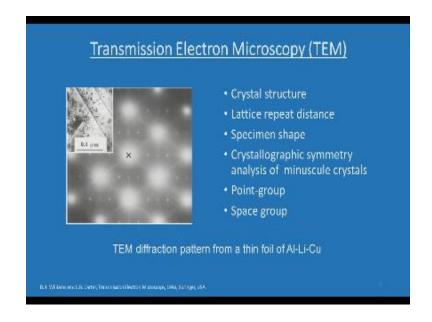


And then I would like to give you some very brief you know introduction about this images are the results which you can obtain from an electron microscope normally what we are interested is in a microstructure micro structure containing features interms of you know if it is a metallurgical sample people are interested in defects and if it is you know physics and then you are interested in defect density and atomic positions and so on.

And so but what you have to keep in mind in totality I mean the TM image gives a an average image interms of that depth it does not have the depth sensitivity so what you are seeing in the slide is the image of an edged is location in a gallium arsenide layer band of dislocation threats through the thin specimen from the top to bottom but remain in focus through the five, so what I

try to say is from an TM image you will not be able to see these features are in the top surface or the middle of the surface of the bottom of the surface so it gives what you are seeing is in a actually a projection which we will see what I mean by projection and what you are seeing is in a an average you are we are not able to distinguish whether these features are lying in the top or whether this features are lying in the middle of the foil or bottom of the file and so the TM micrograph will not have that depth sensitivity that is one information.

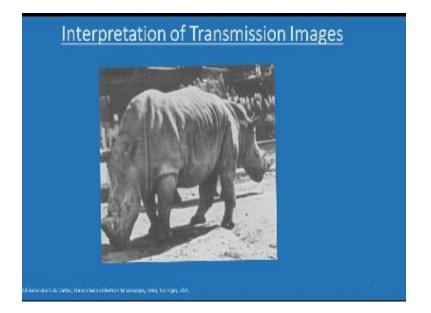
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And what are the information you will get from this TM results in terms of crystallography you get the basic idea of the crystal structure lattice repeat distance specimen shape crystallographic symmetry analysis of miniscule crystals you can derive fine group and space group and so on, so this is the typical electron diffraction pattern one you can one can get from a tem and this is a TM diffraction pattern from a thin foil of aluminum lithium copper.

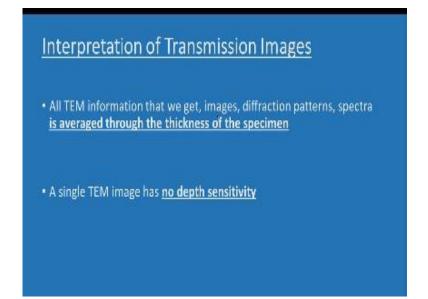
And these are the wide range of information one can derive it from the transmission electron diffraction pattern.

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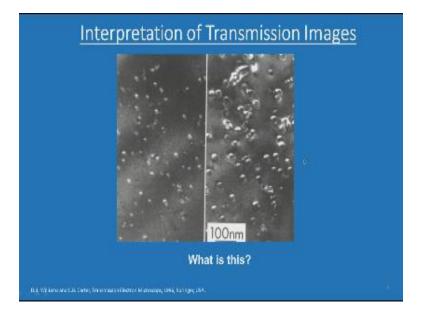
So another very important information from this you should as I mentioned that what you are seeing that there is an photograph where you see that two animals are standing behind one another but it is appearing as if the head of the animal is appearing in the same both sides but which is not the true so the depth sensitivity is not available in the TM micrograph this is a point I would like to emphasis here you have to be very you have to keep that in my information in mind.

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All TM information that we get images diffraction patterns spectra is averaged through the thickness of the specimen the most important information on how to keep in mind whether it is an image whether it is a diffraction pattern or a spectra is averaged through the thickness of the specimen what is the thickness the thickness of the specimen how it varies we will see when we prepare the sample preparation and atypical thickness we will arrive at and how that is affecting the imaging condition that we will see it in the in the coming classes.

A single TM image has no depth sensitivity so this aspect has been illustrated in the previous couple of slides.

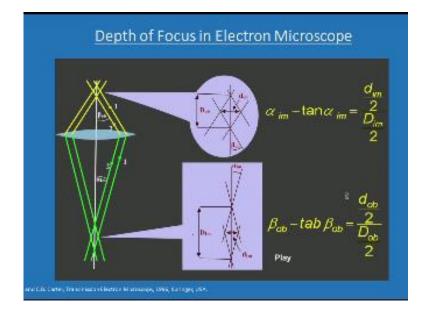


And another important thing is you may get a very different kind of features like this you may consider that may think that this is kind of a second phase particle and this is another cautioning effect of the what precipitated something like that but you have to be very careful before we use this TM results this could be simply not at all related to the material at all it may be due to the some of the you know irradiation damaged by the electromagnetic radiation itself.

So unless you have a combination of all the you know the typical requirement to interpret DTM results I will just mention it when we come to the interpretation in an actual TM results you have to be very careful you cannot just unless you are well experienced in this field it is very difficult to interpret these results and then looking at this kind of image you can be very easily misled because you do not have enough supporting evidence to prove that these are all second phase particle or something like that.

So what has to be very careful about presenting just a one bright field image or a not field image something like that and then talking about a second phase particle and so on which will be highly you know misleading or may be completely wrong also so you need to have appropriate results in combinations of crystallography data through diffraction and we have to prove the second phase particle through the simple adduct field imaging and then you have to correlate with that a bright-field amazing and so on so the what I am talking about all this are different techniques which I will be explaining it in due course of time so what I the information I want to derive from this slide is you have to be very careful about talking about the features in the in a tem micrograph just putting at the one slide you have to have additional information to talk about the features of what you are seeing in the micrograph.

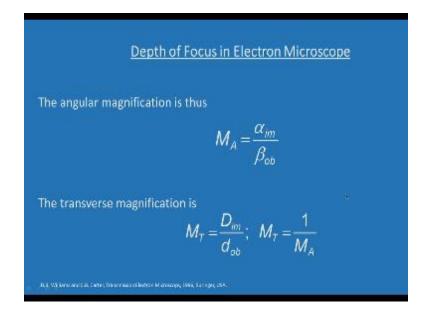
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Now the depth of focus in electron microscope is very high which we have already seen it I will play a small animation what you have to look at it is this is the simple lens and then you see that in  $\alpha$  and  $\beta$  are the angle correspond to the object side and an image side and this crossover is projected here and you can simply simple geometry you can derive that  $\alpha$  image is equal to tan  $\alpha$ image if you incorporate I mean if you consider this triangle and this distance is d objective and this distance is at a small d objective.

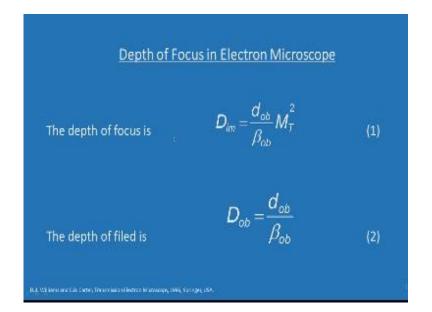
And if you see this is in a  $\beta$  objective and for example then if you consider this geometry triangle then you can derive an expression like this for  $\alpha$  image similarly you can do the  $\beta$  this is distance B D image and this is an  $\alpha$  image angle and this is the small D image then you can write considering this triangle  $\beta$  objective which is approximately equal to tan  $\beta$  objective is equal to D objective by 2 that is this distance half distance divided by D objective by 2 this is this distance this half distance you can write  $\alpha$  image by  $\alpha$  and also  $\alpha$  sorry  $\alpha$  image as well as a  $\beta$  objective angles.

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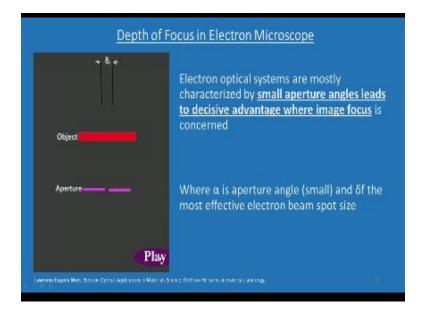


So what we do with this we can use this relation to calculate the angular magnification in the microscope that is  $M_A = \alpha_m / \beta_m$  objective this is angular magnification and then we can also calculate the transverse magnification which is t is equal to D image by small D objective and which is nothing but  $MT = 1 / M_A$  that is inverse of angular magnification is your transverse magnetization this is not small relationship so you should appreciate this you have enough background to appreciate this now.

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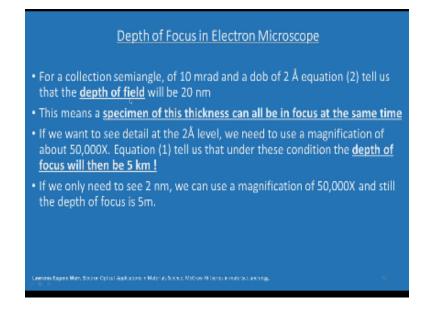


And we also know that the depth of focus d means is equal to D objective by  $\beta$  objective times MT Square and the depth of field is d objective is small d objective /  $\beta$  objective so this is see the relation between the depth of field and depth of focus with respect to that ray diagram what we have seen so you have this background to appreciate this we have already discussed enough what is depth of focus on depth of field it is just to give you a recap.



And now I will just show you a schematic where you see that in the electron optical systems they are mostly characterized by small aperture angles leads to a decisive advantage where the image focus is concerned so you can see that the rays which are converging here and then converging there that is above and below the objective you have a finite distance where the image can be sharply focused that is in general this schematics in general displays the very small aperture angle effect and then the concerned depth of focus. Where  $\alpha$  is the aperture angle and DF that is this DF the most effective electron beams spot size.

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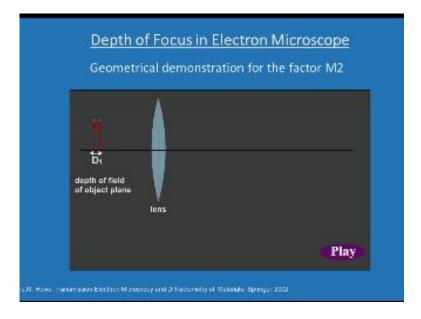
For a collection semi angle of 10 milli radians and dob of 2 angstrom equation 2 tells us the depth of field will be 20 nanometers so the D objective small D objective of 2 ironstone if you substitute that into the equation to what we have seen you will get about 20nanometers this means a specimen of this thickness can all be in focus at the same time if you want to see a detail at the 2 angstrom level we need to use a magnification of about 50,000 X.

Equation well one tells us that under these conditions the depth of focus will be about five kilometers if we only need to see two nanometers we can use a magnification of 50,000 X and still the depth of focus is 5 meters so all this ray diagram and the small mathematical expressions illustrates a point that the depth of focus in an electron microscope is very high and then you will see that the this aspect has been exploited in the at in a transmission electron microscope hardware itself.

Where you though you will see that image formation is occurring in the fluorescent screen and the on the table but your recording system will be much below where you may have a plate camera or a film camera or it is a CCD which is much below but still whatever you are focusing that image on a fluorescent screen will be nicely recorded in a CCT camera of the same focus so that Is one evidence that the electron microscopes have a significant step. So few more remarks on the depth of focus the depth of focus is related to the depth of field through the magnification M where capital  $D = dM^2 / \alpha$  compared to the object plane the extra factor of M square for the depth of focus arises because the image is larger by a factor M so the ray intersections define in the image plane move M times more rapidly than those on the object plane.

Ray's of different angles that converge at the same point on the image have mutual angles M times smaller than what they left the object plane.

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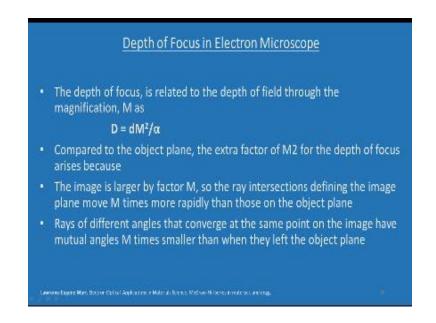


So now we will try to demonstrate whatever we have just read through it through a schematic you just observe that this is a glass lens and then this is an object for example a solid line D 1 and then see that there is a small correction it has to be I know that these two lines supposed to intersect and then diverge and it has drawn as a parallel line it is not true it has to be an intersection but I will assume that it is intersecting and then diverging like this and then diverging in this direction like this.

So you see that suppose if you assume that the solid red arrow is an original object and it is being imaged and what you are seeing here is suppose if you see that you know the distance d2 is the it is a limit of the blurring image because the you know which when you use this when you move this d1 slightly to the distance d1 with the dotted line then these two rays will trace like this you can follow this suppose if you assume that this is my solid line original object if I move that into slightly a position d 1 then the divergence happens.

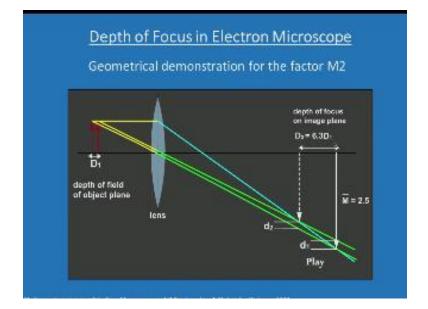
And then the green ray will trace like this because of that the distance D2 the blurring will occur and your d1 is because of the mis-positioning of the yours the image plane where you have one here and one here intersection so it is a mis- position of the image plane so that causes a d2 so what we have just seen as a if you go back.

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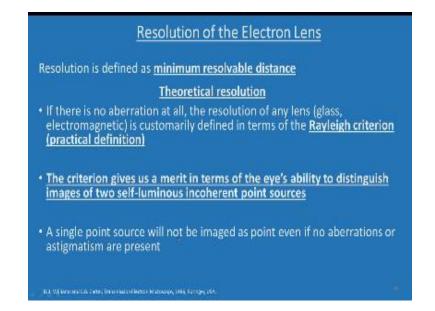
You can you can just verify this that is the depth of focus is related to the depth of field it is a M square times the I mean depth of field that is that we can prove here.

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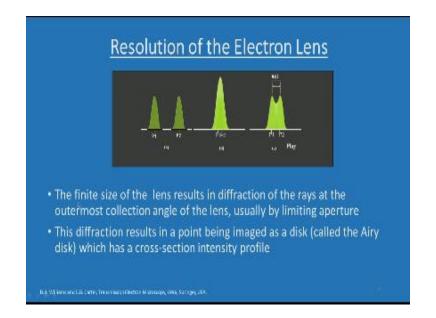
Suppose if you have this distance is approximately you know I mean the magnified the object here is magnified here approximately 2.5 times this is to the scale and you can see that if you square of this that is you can see that 6.3 d 1 times the d 2 that means the depth of focus is equal to six point three times the distance of depth of field depth of focus is M square times the d1 so that you can dramatically prove this illustration clearly shows that the geometrical demonstration for the facto M square it is not m2 it is M square.

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So that clearly shows about a depth of focus now we will quickly review the resolution of the electron lens we have already seen this in a fundamental of electron optical system and just to recap you would like to go through this the resolution is defined as minimum solvable distance and then if you consider the theoretical resolution if there is no abrasion at all the resolution of any that is a glass or electromagnetic is customarily defined in terms of Rayleigh criterion which is also a practical definition.

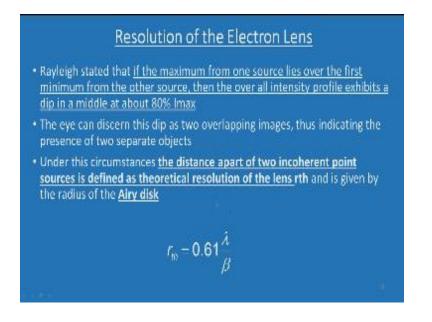
The criterion gives us a merit in terms of the ice ability to distinguish images of two selfluminous incoherent point sources a single point source will not be imaged as a point even if no abrasions or astigmatism are present.



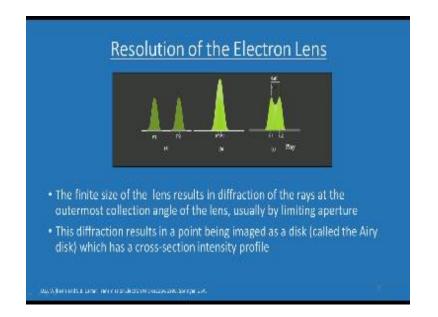
I will play this animation for this to self-luminous point sources which are trying to converge and then these two point sources will be recognized as a independent source only with the distance of 0.61 times the lambda that we have already seen it so you just recall that the earlier discussion the finite size of the lens results in diffraction of the race at the outermost collection angle of the lens usually by limiting aperture.

This diffraction results in your point being imaged as a disc called the Airy which we have already seen it so I will not discuss that further which has a cross section intensity profile.

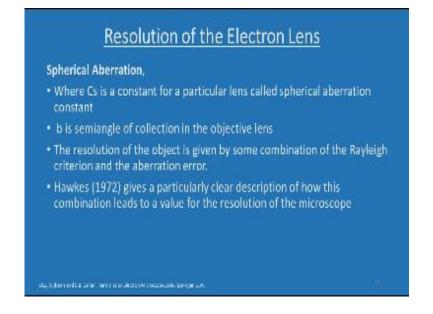
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And Rayleigh stated that if the maximum from one source lies over the first minimum from the other source then the overall intensity profile exhibits a dip in a middle at about 80% of imax so this also we have seen.

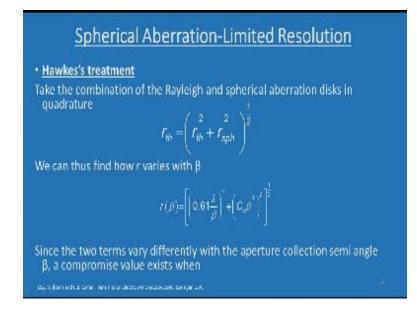


So the minimum of the next source will be matching with the maximum of the first source so and this dip will occur at about 80% of the imax so this also we have seen previously they I can discern this dip as two overlapping images thus indicating the presence of two separable sorry, two separate objects. Under this circumstances the distance apart of two incoherent point sources is defined as theoretical resolution of the lengths and rth and it is given by the radius of the Airy disk or that is theoretical resolution is equal to 0.61times  $\lambda/\beta$ .



And we also seem about the spherical aberration where Cs is the constant for a particular lens called spherical aberration constant and B is a semi angle of collection in the objective lens the resolution of the object is given by some combination of the Rayleigh criterion and the aberration error. So we will now look at the some treatment by Hawkes is gives the particularly a clear description of how this combination leads to a value for a resolution in a microscope. So suppose if you include the spherical aberration coefficient how it is going to be.

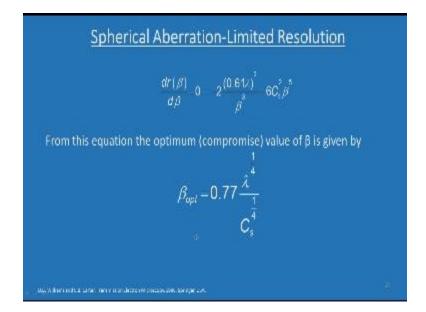
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So this is a Hawkes's treatment suppose if you assume that we are taking a spherical aberration into Rayleigh criterion and take the combination of Rayleigh and spherical aberration disks in the quadrature rth=rth2+r spiracle aberration because of where cabaret are due to spherical aberration is called  $(rxph^2)^{1/2}$  we can now thus find how r varies with  $\beta$ using this relation or as a function of  $\beta$  is equal to 0.61 times  $(\lambda/\beta)^2+Cs\beta q^2$  I mean to the power 2 whole to the power 1 by half but being the square root of all this expression.

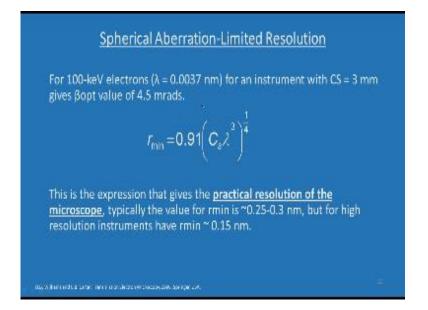
Since the two terms very differently with the aperture collection semi angle  $\beta$  a compromise value exists when.

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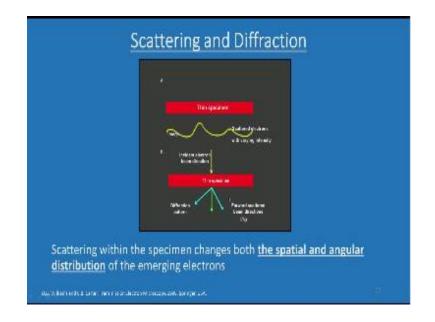
 $Dr/d\beta=0$  is if you can differentiate that expression you will get this kind of value from this equation the optimum value of  $\beta$  can be obtained like this be opt is equal to 0.77 times  $\lambda^{1/2}/Cs^{1/2}$  so this is a called a spherical aberration limited resolution.

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And for 100 keV electrons a  $\lambda$  is 0.00 37 nanometers for an instrument with a Cs=3 mm gives a  $\beta$  opt value of 4.5 millions, so you have our minimum is equal to 0.91 times (Cs  $\lambda^3$ )<sup>1/4</sup> this expression that gives the practical resolution of the microscope typically the value for the r minimum is 0.25 to 0.3 nanometers but for high-resolution instruments have they are minimum which is approximately equal to 0.15 nanometers.

So what we have now trying to say is we have already stated that spherical aberration is very important operation which is very difficult to eliminate from the lens so if you keep that spherical aberration into a system and then how the resolution is getting modified that is the bottom line and these are all these small I mean steps are derivation which demonstrates to you what is the how the resolution expression get modified as well as the how the  $\beta$ angle getting optimized so it is that is a basic information nothing to get confused here.

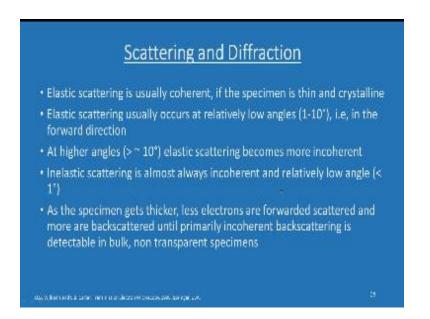


So now we will again go back to some of the basics look at this animation what you are seeing is suppose if you assume that this is a thin specimen which being related by the electron beam and then either transmitted beam will have or if the image of the specimen will have an oscillation in the intensity that is scattered electrons with varying intensity you will see and also you have the incident beam you have a diffraction pattern as well as the forward scattered beam of electrons so these two you are going to get in the transmission electron microscopy where you have a thin specimen is placed.

So what you are seeing here is scattering within the specimen changes both the spatial and angular distribution of emerging electrons so that is the idea you have to appreciate this the scattering within this thin specimen changes both spatial angular distribution of emerging electrons so that is that and you can see other schematic. So the schematic is self-explanatory so you have a background to understand this so a coherent incident beam is falling on the thin specimen then you have backscattered electron secondary electron and coherent elastic scattering scattered electrons and then you have direct beam incoherent in a in elastic scattered electrons and so on.

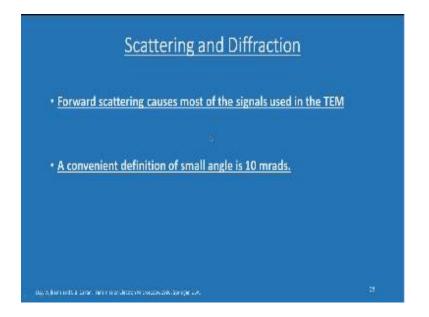
So if it is a bulk specimen there is nothing like you see the forward scattered I mean signals forward scattered signals only you get the backward scattered signals only a thin specimens permits electrons to be scattered in both the forward and backward directions while the bulk specimen only back scatters the incident beam electrons so very fundamental idea you know it but you have to why we are saying this because in transmission electron microscopy we use only the forward scattered electrons we do not look at the backward scattered electrons.

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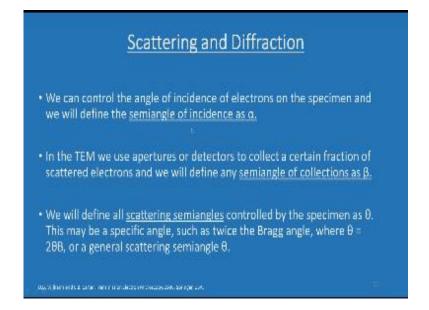
And we quickly rush through this basic idea again once again elastic scattering is usually coherent if the specimen is thin and crystalline elastic scattering usually occurs at relatively low angles one to ten degrees that is in the forward direction at higher angles for example greater than 10 degrees elastic scattering becomes more incoherent inelastic scattering is almost always incoherent and relatively low angle that is less than one degree as the specimen gets thicker less electrons are forwarded forward scattered and the more or backward scattered until the primary incoherent back scattering is detectable in bulk non-trans participants.

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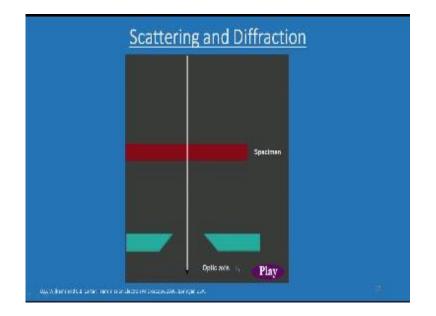


So this point you have to remember forward scattering causes most of the signals used in the TEM so the convenient definition of small angle is about 10 milli radians in TEM.

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We can control the angle of incidence of electrons on the specimen and we will define the semi angle of incidence as  $\alpha$  in the TEM we use apertures and detectors to collect the collect a certain fraction of scattered electrons and we will define any semi angle of collections as  $\beta$  we will define all this scattering semi angles controlled by the specimen as  $\theta$  and this may be a specific angle such as twice the Bragg's angle where  $\theta$  is equal to  $\theta$  B or a general scattering semi angle  $\theta$ .

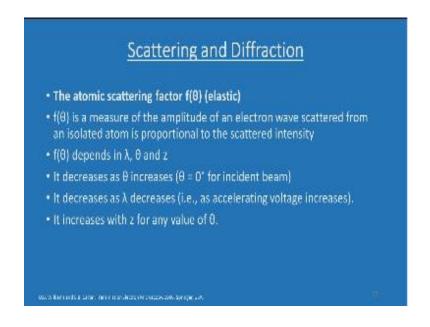


So again you can look at the schematic how the electron beam comes and this is an  $\alpha$  whatever we have just stated in the previous slide you can just look at them as a schematic this is an  $\alpha$ beam converging semi angle and this is a specimen and then you have general scattering angle  $\theta$ and the collection semi angle is  $\beta$  and this is your aperture and this is an optic axis so I can play it again, so that takes care of all the definitions in an TEM and these are all the typical diffraction pattern one get in a TEM and you should know as a beginner what is the difference between all four of them.

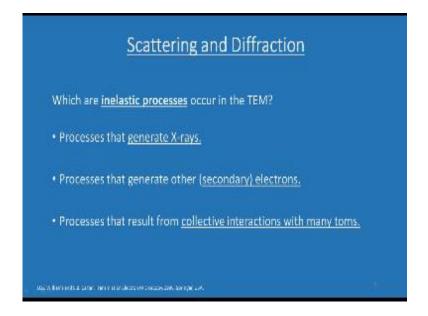
What you are seeing is a diffused ring which typically comes from an amorphous material and this is a single crystal electron diffraction pattern and this is a poly crystalline single I mean some poly crystalline electron diffraction pattern as a ring sharp rings and this is converging beam electron diffraction. So we will explain I will explain all these things when we discuss the diffraction in TEM and what is the reason you see this kind of a pattern that also will be discussed in detail and I am just trying to give you an introduction introductory feel that what kind of diffraction you will be able to get from these transmission electron microscopy so you have these four typical types of electron diffraction is possible and then they are very powerful I

mean information it gives you can derive little more significant micro structural aspects from this diffraction pattern so we will go through them when we come to that section.

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And little more fundamentals again let us again a recap the atomic scattering factor F $\theta$  which is elastic F $\theta$  is a measure of amplitude of an electron wave scattered from an isolated atom is proportional to the scattered intensity F $\theta$  depend sin  $\lambda\theta$ +Z it decreases as  $\theta$  increases and it decreases as  $\lambda$  decreases and it increases with Z for any value of  $\theta$ . So we have discussed this aspects will be discussing the x-ray diffraction so you have enough background for this. (Refer Slide Time: 36:40)



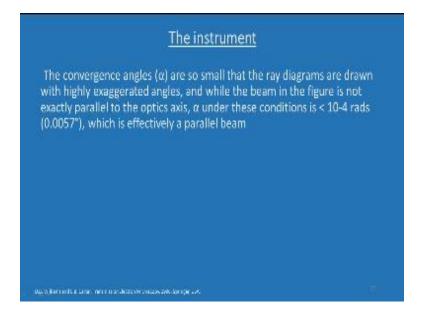
So I will skip this which are inelastic process are occur in the TEM process that generate x-rays process that generate other electrons something like secondary electrons processes that result from collective interaction with many atoms there is a type of here atoms. So these are all the general inelastic process in a TEM.



And now you recall this animation I will introduce an instrument through this animation what you are seeing is an electron source first where you have the high voltage applied and then you have an anode there is an aperture is a condenser lens and then you have a specimen you have an objective lens and follows by an aperture, intermediate lenses, projector lenses and then final screen and what you have seen is how the electron beam comes through various apertures and lenses and falls on the specimen and it produces some signals second signals.

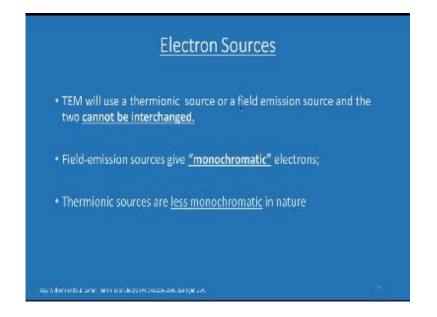
And then it further transmits through some of the electromagnetic lenses and apertures and it falls on the so you have the you are now familiar with this kind of an electromagnetic lines we have already seen the functions of this and how they are exploited here and also we have seen that you can look at the corresponding light optical system where you have the condenser of condenser lens and you have the specimen you have objective lens and you have a projector lens and then screen so you have one is to one comparison with the light optical system so both have almost similar I would say the ray diagram except that they are all electromagnetic lenses here it is.

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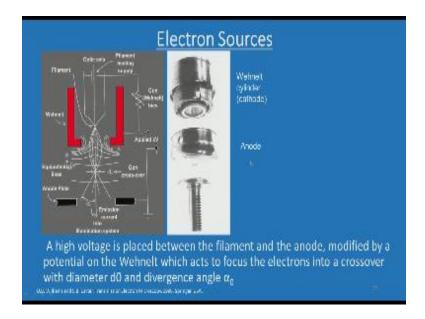


So the convergence angles  $\alpha$  are so small that the ray diagrams are drawn with highly exaggerated angles and while the beam in the figure is not exactly the parallel to the optic axis  $\alpha$  under this condition is less than 1 I mean less than 10 to 4 radians that is 0.005 7 degree which is effectively a parallel beam.

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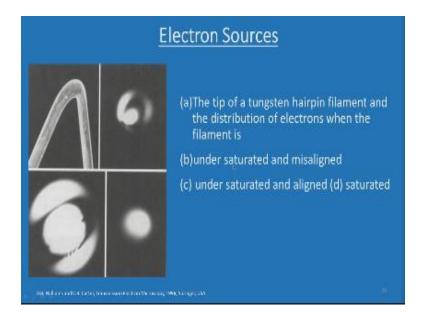


And then we will look at the electron sources we will start with the electron sources this also we have seen it in the introduction just for the set of completion I will just go through this TEM will use a thermionic source or a field emission source and the two cannot be interchanged field emission source gives monochromatic electrons the thermionic source are less monochromatic in nature.



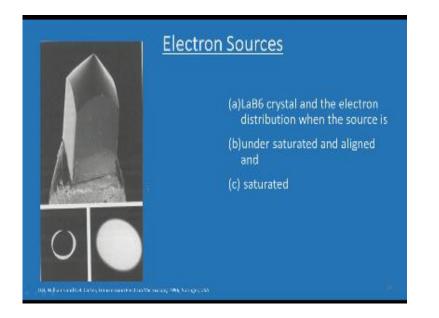
And this is the typical many electron source are then design you have this electron gun this is a Wehnelt cylinder this is an actual photograph and this is an optic axis and you have the filament here and then you have the anode so corresponding anode is shown here and you have the gun crossover and applied voltages there and we have looked at the function of this lens I mean the electron source and it is designed earlier also a high voltage is placed between the filament and the anode modified by the potential on the one at cylinder which acts to focus the electrons into a crossover with a diameter D0 and the divergent divergence angle  $\alpha 0$ .

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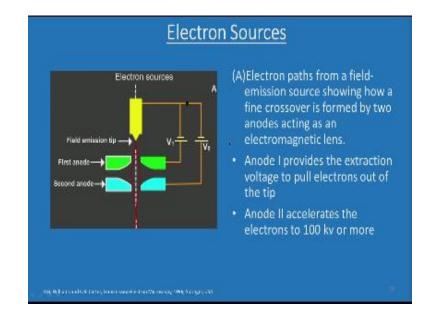
And then if you look at the electron sources for example it is at unction helping the tip of attention hairpin filament and the distribution of electrons when the filament is under saturated and misaligned and then saturated aligned so you have the this is an under saturated and misaligned beam will look like on the screen and you have the under saturated aligned beam will look like this and this is the saturated p so we will look at this when we you will evidence this action why we operate the microscope.

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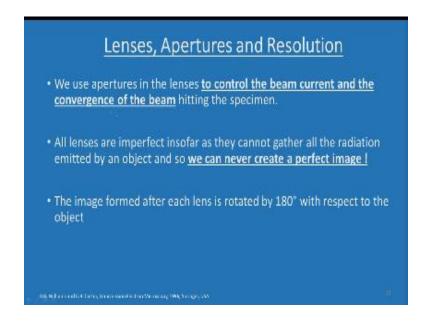
And this is another Terminix source LaB6 crystal and the electron distribution when the source is under saturated and aligned see is a saturated beam which will appear like this.

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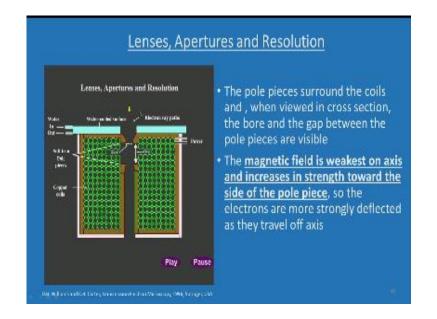
This is a feed emission than a tip electron pulse from the field emission source showing a how a fine crossover is formed by two anodes acting as an electromagnetic lines and at one provides their extraction voltage to pull the electrons out of the tip and our two accelerates the electrons to 100kv arm or whichever is designed. So again we are looking at a second time we have discussed this.

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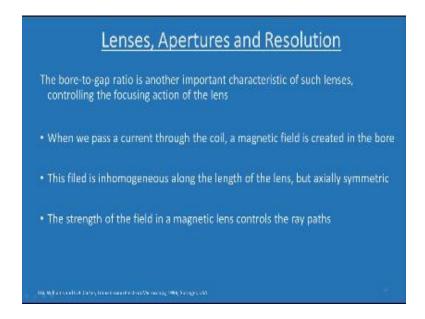
So we use apertures in the lens lenses to control the beam current and the convergence of the beam hitting the specimen all lenses are imperfect insofar as they cannot get all the radiation emitted by an object and so we can never create a perfect image the image formed after each lens is rotated by 180° with respect to the object we will see how this aspect is taken care in the modern microscope when we discuss the image and so on image formation in the TEM.

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And a typical electromagnetic lines is shown in the schematic you can see that these are all the copper coils which is this is across-section of a electromagnetic lines which I have shown in the fundamentals of electron optical system as well so you have the soft iron pole pieces and this is the this is a boar and this is the gap and then you have the water inlet and outlet for the cooling and this is an optic axis electron optic system the pole pieces surround the coils and when viewed in a cross-section the bore and the gap between the whole pieces are visible.

The magnetic field is weakest on the axis and increases in strength towards the side of the pole piece so the electrons are more strongly deflected as they travel off axis so you can see that while the schematic is shown in this manner is because of this effect. (Refer Slide Time: 45:05)



The bow to gap ratio is another important characteristic of such lenses controlling the focusing action of the lens when we pass a current through the coil a magnetic field is created in the bore this field is inhomogeneous along the length of the line lengths but axially symmetric the strength of the field in a magnetic lens control the ray path, so though we have we are going through this we have already seen the basic function of an electromagnetic lines just for the sake of completion and the recollection I am doing this so we will continue to look at the some of the instrumental details and then we will go to the diffraction in TEM in much more detailed manner, so we will continue our lecture in the next class, thank you.

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