### Indian Institute of technology Madras Presents

## NPTEL NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING

Lecture-38 <u>Materials Characterization</u> <u>Fundamentals of Transmission Electron</u> <u>Microscope</u>

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Hello everyone welcome to this material characterization course. So far we have seen a diffraction principle in TM and in various techniques involved in the diffraction and how to extract information from the by simply looking at the diffraction pattern of transmission electron microscope. And also we have seen that how to use the line and how to use the convergent beam electron diffraction.

And various advantages of using these techniques in much more briefly. Similarly we will just go through the imaging techniques in transmission electron microscopy before you look at the laboratory demonstrations of the actual experiment in TEM. So we will not get into the detailed analysis of TEM imaging in this course, because it is only a part of a characterization course. But you will get the basic idea between each imaging techniques in the conventional transmission electron microscope. (Refer Slide Time: 01:33)

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So if you look at the major classification of imaging the first one goes like this amplitude contrast imaging that is formation of a bright field image by using only the transmitted beam. B, formation of a dark field image by using only one strong diffracted beam. So the other category is phase contrast imaging where the formation of image by more than one beam interfering each other.

The contrast s therefore a composite of interference and amplitude changes produced by defects. So we will first go through the amplitude contrast imaging or we can also say that it is a diffraction contrast. And the name itself says that once you say that it is a diffraction contrast and amplitude contrast it is directly related to the diffraction intensity. So you have some idea about how the diffraction intensity is proceed which we have seen so far.

But just for a clarification we will go through that once again and in a x-ray diffraction we have seen that, you know in a diffraction we have kinematical theory and assumptions. Similarly in TEM also we have couple of theories which arises out of these specimen. So all the kinematic relation what we have seen in x-ray diffraction course could here, and then we will see that in another theory called a dynamic theory and then we see the difference the key difference in these two theory and how they are explained in the origin of the contrast. So these things we will see.

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So if you look at the kinematical theory is applicable to only thin specimen and for conditions away from the exact Bragg condition which is S0=0 you have to remember this point very clearly here, you will be using this parameter called deviation parameter called S which is, which describes the diffraction takes place away from the Bragg position.

So as I mentioned in the previous lectures also this idea you have to keep in mind whether the diffraction takes place with the exact Bragg condition where S=0 where the diffraction density where we have looked at where S is not equal to 0 that means away from the back position. So kinematical condition to applicable to only in this situation for a thin foil.

So if you look at the diffracted intensity this is a  $\psi^2$  D stands for diffraction which is equal to  $(F/t)^2 \sin^2 \pi t s/(\pi s^2)$ . And the intensity for a transmitted beam  $\psi^2 = 1 - (F/t)^2 \sin^2 \pi t s/\pi s^2$ , so you can just simply write this  $\psi^2 + \psi^2 d = 1$  so the kinematic theory protects that the bright field and the dark field images are complimentary, because even in the x-ray diffraction we have mentioned it

assumes that the only one diffracted beam and the one transmitted that is the two beam condition.

So that is why this equation predicts that you have a bright field image as well as the dark field image they are complimentary to each other.

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So the kinematical function predicts periodic variations in intensity with the thickness for a constant S the thickness fringes or variation in intensity with S for a constant thickness which lead to fringes about the Bragg contours. What we are now trying to say is this function whatever we have just said to the function the kinematical function which predicts the periodic variation intensity with the thickness for a constant S.

Suppose if S is constant so how they periodically the intensity varies or the variation in the intensity with S for a constant thickness, so these two will describe how you see a kind of fringes. So the kinematical theory I would try to explain this the fringes which we see in a bright field image. The intensity maximum occurs in the dark field whenever S=n/t that is the constant thickness or when t=n/s for a constant S. So this we will see in a example.



And before you go to the actual imaging let us look at this slide, very interesting slide this is a most fundamental idea one should get before you get into the interpretation of image contrast. You see this is the, we assume that a schematic shows the thin specimen of thickness t and then you have this incident beam  $\psi 0$  and which enters the specimen. And then you have from the diffraction phenomenon you know that the beam enters the specimen and then try to interact and some of the, assume that some of the beams are diffracting.

So the intensity of the transmitted beam as well as the diffracted beam will oscillate like this. Why it oscillate like this, we have the reason we will see in a few minutes. But now look at this suppose we assume that the beam enters this sample surface and as it goes inside the deeper into the crystal then the transmitted intensity follow this oscillation and the diffracted intensity follows this solution.

So that point you remember and this distance that means if you look at this transmitted intensity this portion is characterized as  $\xi g$  called extinction distance we will in a minute we will see what is it. And so much of intensity is lost from the transmitted beam so this distance is a characteristic distance for a material and depends upon various parameters that also we will see.

And you see that depending upon the number of this  $\xi g$  n  $\xi g$  in the sample which is equal to thickness of the food foil, this also we will see how it is valid.

But before you for time being you ignore about this, but what I am trying to show here is you please start thinking before you interpret the image contrast in a TEM the electron beam enters this sample and you are transmitted at a diffracted beam have the intensity oscillation in this manner that is in that point you remember to start with then we will see how we can manipulate this idea to understand some of the image contrast in the TEM operation.

So when you what is written here this intensity oscillation we have perfect crystal section of the thickness n  $\xi g$  so we are talking about a perfect crystal that means I do not have any fault within the top two the bottom surface of the crystal it is the perfect crystal, I do not have any, you know boundaries, I do not have any vacancies, I do not have any dislocations, I do not have any interface and so on.

So you assume that it is an ideal crystal and then to the electron beam transmit through the sample and then that time the intensity oscillation is of this kind. We will just work on this idea as we move on -

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The periodic variation of  $\psi^2$  that is intensity with the t leads to a primary extension t0 – 1/ s forget about this t0 is equal to 1/s which is a relation which how the extension distance as got we will talk about in a while but the periodicity of the potential energy that originates with the periodicity of atom arrangements so now we will try to answer the question why the periodicity why the intensity oscillates like that.

So we will try to do have some kind a qualitative understanding the periodic potential causes the amplitude of the high energy electron to be transferred back and forth dynamically between forward scattered and diffracted wave functions at precise Laue condition for a strong diffraction the physical distance over which the wave amplitude is transferred back and forth once is called the extension distance.

So now you see that you have the accountability for why we see that intensity oscillation in the perfect crystal so it is because how the periodic potential that causes the amplitude of these high energy electron to be transferred back and forth so we have the lactic have the atoms and then the lactic as a got a periodicity under the periodicity of the potential energy of the lattice which

affects the amplitude of high energy electron so that is the basic idea so the interaction of these two will cause the beam to oscillate back and forth.

And a precise Laue condition for a strong diffraction the physical distance over which the wave amplitude is transferred back and forth once is called extension disks now you go back and see this.

Qualitative (kinematical) interpretation of crystalline image contrast

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It will make a sense so now you see that it goes forward and backward once and this distance is called a extension distance very important in understanding the diffraction contrast or anyh contrast in the bright field image.

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So this is explained again by a dynamical theory the usage of extension distance and so on in dynamical theory that means dynamic diffraction conditions the extension distance IG is defined as  $\pi v/\lambda$  Fg where v is the value of the units cell and f the structure factor for the particular reflection, so we have the idea about structure factor how it originates what it contains so you know at least you can now connect what why  $\psi$  g is characteristic of a material.

At every integral number of extension distances all the electrons end up in the forward direction that is transmitted direction under every odd of multiple of extension distances all the electrons end up in the diffractive directions, so it clearly describes what we have seen in the previous slide how the oscillation takes place and so on. (Refer Slide Time: 15:20)



And now having said that this is the kinematical approximation and how the dynamic theory explain this extension distance in kinematic theory we say that only 1 diffracted beam we are looking at 1 transmitted beam we are looking and there is no interaction between these two that is assumption of the kinematic theory even dynamic theory we say that these two beam try to interfere and then produce some affect.

So the equation which have seen in the slide describes this phenomena interaction of the transmitted beam under diffracted beam and then what it converts let us see  $dØ_0$  by  $d\xi = I \pi / \xi_0 Ø_0 + i Ø / \psi g$  into Ø g into exp ( $2\pi$  isz) this is for the transmitted beam and this a diffracted d  $Ø g / dz = i \pi / \xi g$  times  $Ø_0$  into exp ( $-2\pi$ isz) +  $i\pi \xi_0 Ø g$ .

These two equations are popularly known as Howie Whelan equations describe the variations of the amplitudes  $\emptyset$  of the undeleted and diffracted waves as function of z the distance through a perfect crystal the first term in each equation arise from the scattering from the undeflected beam and the second team arises from the scattering from the diffracted beam they show that the amplitude of each wave changes as the waves progress through the crystal due to a contribution

from each other so these two equations explains the interaction of transmitted beam as well as the diffracted beam. So that is the physical meaning of these two equations.

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Now we will just come back to the other features of this dynamical theory the exchange of electron density between the transmitted and diffracted beams is exactly analogous to the motion of two coupled harmonic oscillators which periodically exchange all the vibration energy of the system the subsidiary maxima. Occurs when  $s^2 + (\xi g^{-2}) t^2 =$  integral so this is one you can see the subsidiary maxima in the image contrast for thin crystals the value of t that is thickness can be determined from the measurement of s at subsidiary fingers either in the image or in the diffraction patterns, provide that the values of  $\xi$  are known.

You will be able to find out the thickness of the foiled if you know the  $\xi g$  of an given material so we will see that also.

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So the contrast effects in a perfect crystal are expected to be due to changes in thickness that is wedge fringes, at inclined defects. Changes in s will result in a Bragg contours, changes in orientation changes in s as well as g. so we will we will now demonstrate all this predictions by the both the theories with a simple examples, how that contrast various are what kind of costars variation will be seen by change of thickness or changes of s or change in orientation of the foil. (Refer Slide Time: 19:45)



So the important microstructure features include the changes in orientation with or without change in the structures or composition such as grain twins precipitates structure of boundaries lattices defects point defects line defects planer defects, volume defects multiphase system a changes in the composition.

But in the structure example spinodal changes in the compositions and structure the general precipitation changes in the structure but not composition from example marten site inter phase interfaces coherent partially coherent incoherent so all this features can be identified with the contrast mechanism predicted by the both the theories like kinematic as well as dynamic theories. So we will see one by one.



So this symmetric shows a general a very general diagram this is a I just brought it juts for the completion you know the basic idea behind it this is I the object and the you have the incident being coming through this object and you have the lens and this is the back focal pain where the diffraction pattern is recorded and you have the image plane so this schematic is less static condition for imaging periodic structure if D is large enough and alpha small enough.

The diffracted beam A or A dash recombines in the direct the B to give a magnified image of the planes D the diffraction pattern is formed in the back hope plane A A', n represent the diffraction pattern and Q magnifies the image of this pattern for the beam A and B, so a simple ray diagram you all familiar with all the components here.



And now we will now go to specific operation in a TM the schematic what you are seeing is, the contrast in the by field image arises through the local variation of the intensities of the diffracted beams and the diffracted beams is stop by objective aperture, so what you are seeing here is this is the specimen you have the electron beam coming through this and then you have a direct beam and the diffracted beam and this is an objective aperture.

In a by field image you see that all the diffracted beams are stopped and only direct beam is allowed to pass through the objective aperture, on the other hand you have the direct beam it stopped by the objective aperture only diffracted beam is allowed to pass through the objective aperture and that is how the both the bright field filed and the dark filed and just saw wave allow to form at this operation can be done through beam tilt.

This again we have seen it when we discuss about diffraction phenomenon this is just for the recall.



And now we will look at this a typical a bright field transmission electron micrograph and this micrograph belong to the thin foil of molybdenum showing a light grains a dark grains, fringes and sub grains, so now our idea is why do we see this is a contrast why certain positions are certain grains are appearing darker and why certain portion regions appear in grey color and why particular grains appear in bright. So that is where we have to look at the theories and how we understand this.

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We should also understand why we see these fringes in the bright field image and we will go back to this image and then first try to explain why this Kane is appearing dark, so if you can go back and then look at this schematic.



What you are seeing here is, here is a specimen and this is the electron being passing through and this is the direct beam and this is the diffracted beam just assume that this diffracted beam which is coming out of particular region or a set of planes or a set of grains being stopped by the objective aperture which is not reaching the image plane, so you can now correlate by looking at this schematic.



And this image that means this is a bi-field image I said so the region which is appearing very dark or diffracting very strongly and they are not getting entered into the objective aperture for example the complete set if plane in the grains at the region and this region they are strongly diffracting but they are not entered Into the objective aperture so that regions will appear dark and the region which are appearing very bright that means they are not diffracting strongly or the diffracted beam also entering into the objective aperture we can consider that way also.

So you can either consider it is not diffracting strongly or the diffracting beam also entering into the objective aperture, basically this contrast arises because of the orientation of the grains so you can see that a strong diffraction not diffraction or individually, some diffracted beams are entered something is not entered into the objective aperture, so you can apply all the knowledge if you got in the diffraction phenomenon.

Including the deviation parameter and so on you can put together to understand this to give some perspective why you get this contrast so you can also look at some of the other very integrated features like a fringes, okay I know that a grain which is very dark we can say that the particular grain is strongly diffracting but when you have the fringes like this why I get this a very systematic absence of a dark and bright systematic absence of intensity, why do we are that, that we will look at with this example.

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For a schematic illustration of the origin of fringe contrast if the image of the crystal, so look at this schematic you have the thin foil which has got a whole inside after electrolytic polishing typically you produce a foil like this in a metallic foil that means you create a nice edge by ejecting, assume that your whole is prepared I mean this manner and as I just showed in the two slides before this is the you know the electron beam enters the sample in this direction.

And then you have the transmitted beam that are direct beam or the diffracted beam they have the intensity oscillation like this or the thickness of the foil is T from top surface P to the bottom surface P prime and the please note that the diffracted beam is out of phase by  $\Pi/2$  with respect to the direct P so the diffracted beam is out of phase of  $\Pi/2$  in this okay so now you just see that the bottom portion schematic this Beam which shows some kind of a bright dark line.

They are called fringes here also it is the fringes are in circular shape here it is a vertical lines they are all fringes so now the question is, how to account for this intensity oscillation in this fringes, so if you look at this transmitted intensity oscillation it is a diffracted intensity oscillation this periodicity is  $T^0$  prime the  $T^0$  prime which is again equivalent to  $\phi_g$  or an extension distant we can say that.

So look at this suppose if you have a boundary like this in the material and then you can just look at the how the diffracted beam will travel through this a boundary then because of that how the intensity variation will appear in a bright field that is what we are interested suppose if you have the boundary incline like this here the you see that you have the diffracted maxima here it comes on the bright field it will appear like a dark line wherever the diffraction maxima is the you see in a bright field image it will appear darker because the diffraction maxima is corresponding to minima in the direct been intensity oscillation so that is where the dark lines are see remember that intensity is not 0 there.

But it is minima so when you have this kind of a diffraction intensity which oscillation which come across any inclined boundary like this or this you will have this intensity oscillation that is called a fringes let us go through the a caption so this is the illustration of the origin of the fringe contrast the image of the crystal section through the crystal showing the kinematical intensity oscillation of the direct and diffracted of this the depth periodicity  $D_0$  fine is equal to 1/S and AB the brain boundary or it could be a sticky fault CD sub wedge and E is a whole D is a section normal through beam corresponding to the bright field image showing.

Dark fringes F or a thickness extinguish contours so this particular fringe contrast is related to S that it is inversely related to S 1/S T = T not prime is equal to 1/S and then we will show some more examples for this and so now this is accounts for this fringes what we are seen any fringes you have seen.



So not only that even if you look at the that is locations but what we are see here the sub boundaries sub boundaries are dislocation whatever we are see here it clearly related to the surface Anivuns which will have ups and downs when a such a crystal will interact with the, the diffracted beam and I mean a electron beam I would say which undergoes here intensity oscillation as we have seen in this schematic then this kind of contrast is possible is that is a very simple qualitative explanation more can get without getting into any complication.



You can see that thickness extension contours and the bend contours in the samples for a molybdenum crystal the grain boundary is lie normal to the plane of the foil here you can see that the boundaries or you can say that contours bright and dark line because of the it is orientation and the ups and downs or it could be ups and downs so if it is inclined the surface your bound have this intensity oscillation due to the we are because it is intersecting with the diffracted and the transmitted intensity oscillation.

So that is what it is the this micro graph shown here this foil of aluminum you have some features mark ABCD and so on, you have A two low orders strong reflections hkl and -H - K - L the single contours BCD belongs to a higher orders of hkl so you have this particular band this because of the strong reflection from particular hkl plane and there all belong higher order HKL this count dark lines comes from at least note that no he may consider the whole band as a strong a diffraction or whatever it is appearing bright and dark where the kinematical theory explains.

There is a variation within this band bright and dark which is explained only by the dynamical theory where the transmitted and the diffracted beam inter acts and produces this is oscillation.



And if you apply this concept of kinematical theory and we can look at the some of the fits and wholes which forms the specimen and they also produces a fringes so look at the schematic this is consider there a thick specimens which has got a the whole in it so this is the intensity oscillation of your diffracted beam and this is a transmitted so on and this is the extension distance and since the extinguish distances three times of the thickness so we can write this T = T  $\psi g$  and you can look at them.

Fringes from the this is a that full thickness after that you form their fringe atom like this circular fringe and it clearly shows that the distance between these two fringes is related to extinguish distance like this so if you simply count the distance the number of fringes will be able to calculate the a thickness of the foil this is of course returned to that particular location where you are seeing their a pick or whole in the foil so you will be you are seeing this the fringes then you can relate this.

Fringes and it is count to the total thickness of the foil so this is geometrical description of formation of image pattern at wholes or a similar thickness irregularities in thin foils dark fringes correspond to the intensity minima in the bright field so whatever the dark fringes your see as I

mention in the previous slide it is not 0 but is intensity minima in a bright field we mention so you can see that a typical example which is shown in this.



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Diagram this is not sorry this not a diagram this is not a micro graph excel micros graph you see that whole which is formed in a foil as a got a fringes like this and here again another schematic which illustrates the bend extinguish contours so you have the foil of this nature is a bend and this is the electron been entering into the foil and to produce the contours of this nature and this suppose if you assume that this is the a bright field image and this is the intensity oscillations and if it is a dark field image.

This dark will appear brighter and this will appear dark current so on. So this is the geometrical origin of a bend contours for the idealized case within foil section observe in the bright field illumination and this is again another example of the fringe contrast and etched pits in a steel so you will get the a symmetrical contours like this because you can identify the symmetrical nature of the contours by the a CD pattern which is given as I insert here so the point which I want to.

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Emphasis here is if you understand the electron beam interaction with the a perfect crystal and then if you assume that the intensity oscillation of transmitted being under diffracted beam and any irregularities or a thickness variation or any of the micro structural features they are going to raise to intensity oscillation are called a fringes or a bend conduce and so on. (Refer Slide Time: 41:20)



So here is another example where the where you have the sample with we just got incident boundary and you have this intensity oscillation of the diffracted beam, so the geometrical origin of the fringe contrast at the phase shift or interface plane. The number of fringes depends on the extinction distance for the operating reflection g. so please understand one thing very important thing this extinction distance is a characteristic of an operating reflection g that is for a given bad condition the extinction distance is valid.

So the number of fringes as I just mentioned depends up on the extinction distance for the given operation reflection. So here is a typical micro graph given for a grain boundary so you see that the fringes in the inclined boundaries intersecting the polycrystalline section is shown here see the boundary is not straight here it is an inclined plane for this orientation, suppose if you change thee orientation you will able to see such a slope in this boundary also.

So that is where the tilting exercise is very sensitive in TEM so you see that the kind of fringes it produces that clearly shows that kind of a tapper section that boundary has and then we can identify this boundary and the fringes comes because of the what we have seen in the schematic, the reason for the obtaining a fringe contrast explained quantitatively by the kinematical theory, and dynamical theory.

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And we can also explain this diffraction contrast through something called phase amplitude diagram, what you are seeing here is let me first start with the polar the presentation of a complex number which is being used to construct the phase amplitude diagram. This is for a unit circle on a unit circle exponential  $i\theta$  this is a polar representation of complex number and what you are seeing here in this schematic is the wavelets diffracted from a unit cells at increasing depth rg in the expression diffracted intensity  $\psi s=\Sigma$  over rg into fg exponential  $+i2\pi s.rg$ .

We call that s is lying the vertical z direction, so this the diffracted wave which travels inside the crystals so that is how the unit cells are represented by this because the total diffracted intensity is always  $\Sigma$  of the individual diffracted wavelets like this so we will just look at that which more detailed using this phase amplitude diagram.

The diagram which is shown and they tell here are vectors representing the individual terms that is nothing but your complex exponential how this each diffracted wave is considered like this. So this animation clearly shows that suppose if you have if try to sum up the individual diffracted terms like this then the total intensity of diffraction is given by this  $\Sigma$  of all this exponentials complex exponential in this fashion, so that is how the diffraction intensity is appreciated or I would say is understood by this phase amplitude diagram.



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We will now see that how this phase amplitude diagram is useful in understanding the contrast what we see in a bright field image, so this is the again representing the intensity  $\varphi \varphi^*$  where you have the real part and image in real part of the total intensity, and you have this phase amplitude diagram for two conditions one is for S is greater than 0 or S is much, much greater than the 0 that means you have the phase amplitude diagram for two values of S.

Phase amplitude diagrams of diffracted intensities for two deviation parameters S. The eight short vectors each have the same length but different orientation so the very important information which you obtain from this phase amplitude diagram is, so you will be able to say what kind of the contrast we are going to see just by looking at this s values or how this the circle the diameter of the circle the phase amplitude diagram circle how it varies by just looking at this you will be able to command on how the intensity is going to vary.



So we will see an example like that, so here is an schematic where you have wedge kind of a sample and you see the electron beam coming and then how the diffracted wave vectors will form, how exactly it get you know rotated I mean I would say that all this simple diagram shows that how the phase shift occurs as a function of the specimen thickness and then what you see here is the intensity of the transmitted beam and intensity of the diffracted beam.

You can see that it is very close to the edge of this specimen you see that you know the intensity is high in a transmitted beam but as you go up higher the thickness you are able to see the diffracted intensity goes to the maximum and then as it comes down further thicker side that means you have that distractive interference contributes and then it comes down and then again it increases so you can see that clearly the phase amplitude diagrams for increasing thickness across a wedge shaped crystal showing a origin of thickness fringes in the diffraction contrast.

So all the contrast whatever we have discussed in the whether it is a grain or a fault or boundaries whatever we have just discussed in the fringes they are all coming under diffraction contrast or amplitude contrast imaging and this particular illustration clearly shows that the thickness variation will have a significant influence on the fringe contrast what you see.



So here you see a similar example, where the phase amplitude diagram shows for the same wedge shaped crystals but with us S smaller by a factor of 2 that means you reduce the S that means what kind of changes it will show, if you go back to the introduction where I showed here that means my phase amplitude diagram circle is going to be bigger, if I reduce the S the phase amplitude diagram or the circle is going to be bigger.

So this is what I am seeing so the phase shift which is represented by this phase amplitude diagram is bigger compared to the different at the S vale. So similar effect we can see it in one of the examples we can take some examples and see what we have gone through is true or not and we can look at actual fringes in a metallic samples and then we will continue this discussion in the next class, thank you.

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