Indian Institute of technology Madras
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Lecture - 39
Materials Characterization

Fundamentals of Transmission Electron Microscope

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Hello everyone welcome to this material characterization course in the last class we just started looking at the TEM amazing techniques and then we have seen that there are two theories which explains the diffraction contrast and fringe contrast and so on namely the kinematical and dynamic theory and we have also seen the concept of extinction distance where the dynamic theory explains and then which is very useful in understanding the fringe contrast as well as how it can explain the contrast which arises because of the interfaces and other effects in the crystalline material.

And also it we have seen that this extinction distance is very useful parameter in order to obtain a thickness of the foil and so on, so we have just we will continue that discussion about the diffraction contrast so if you look at what we have what we have discussing in the last class.

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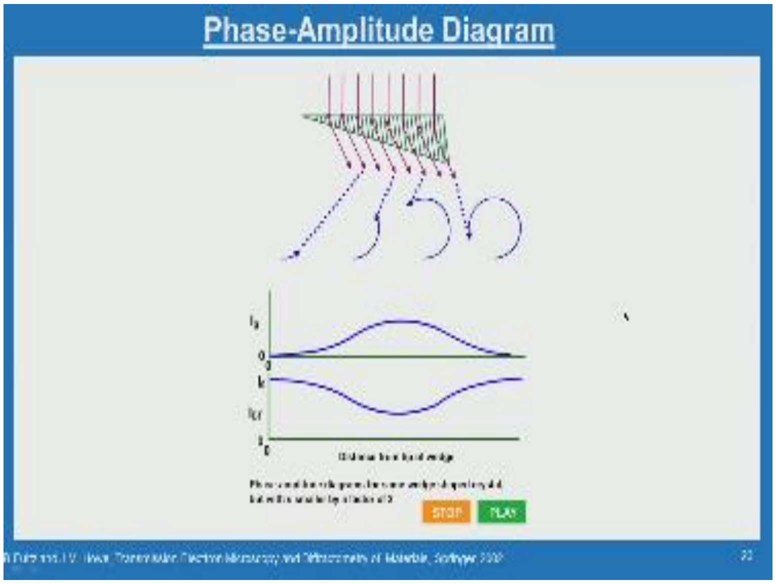

Phase-Amplitude Diagram

That we were looking at the contrast which arises from the wedge type crystal using the phase amplitude diagram, so the diagram with a schematic diagram what you are seeing here is a sample which is correspond to with a smaller s compared to the previous illustration we have seen, so what is that we are seeing now with the smaller s that means you are getting the amplitude phase diagram circle becoming bigger and you see that the intensity oscillation I would say that the diffracted intensity you can see like this.

And then this is a transmitted intensity so that the fringe pattern I would say that the distance between the fringe the dark and bright oscillation also increases. So that is one information we are getting from this we will now look at a an actual example for this

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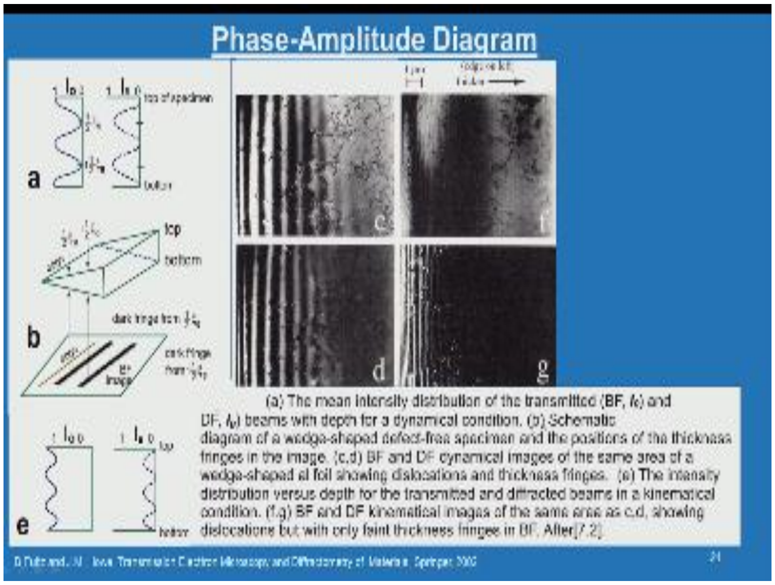

Phase-Amplitude Diagram

(a) The mean intensity distribution of the transmitted (BF, I_0) and DF, I_g) beams with depth for a dynamical condition. (b) Schematic diagram of a wedge-shaped defect-free specimen and the positions of the thickness fringes in the image. (c,d) BF and DF dynamical images of the same area of a wedge-shaped al foil showing dislocations and thickness fringes. (e) The intensity distribution versus depth for the transmitted and diffracted beams in a kinematical condition. (f,g) BF and DF kinematical images of the same area as c,d, showing dislocations but with only faint thickness fringes in BF. After[7.2]

D.Fultz and J.M. Howe, Transmission Electron Microscopy and Diffractometry of Materials, Springer 2002

We will go through this diagram little more carefully what you are seeing in schematic a is the oscillation of a transmitted intensity beam and this is an diffracted intensity beam through a sample thickness T this is a top of the specimen this is the bottom of the specimen and this is the EQ been shaped crystal which we are talked about and you see that a corresponding a bright field image where you see a bright and dark fringes you can see.

And what we are now seeing here is we will first go through this figure caption and then we will discuss the mean intensity distribution of the beam that is I_0 and a dark field IG beams with the depth of the dynamical condition that is schematic a schematic beam shows the wedge-shaped defect-free specimen and the position of the thickness fringes in the image and c and d is a bright field and a dark field dynamical images of the same area of the wedge-shaped aluminum foil showing the dislocation and thickness fringes that is C and D are images form with the dynamical images e the intensity distribution versus depth for the transmitted and diffracted beam in the kinematic condition.

So this is the kinematic recondition figure f and g bright field and dark field kinematical images of the same area as c and d showing the dislocations but with only a faint thickness fringes in right field what the schematics try to explain and this micrograph shows is that you have the 2's conditions that means the images are obtained with the 2's values and how the dynamical conditions that means where you have the transmitted beam and diffracted beam completely interacts and then what kind of image it produces are you have the kinematical conditions where you have a diffracted beam intensity and transmitted intensity I mean being they do not interfere.

So that is the assumption and now these two have at two guess values we will see how we can understand from this so here you see that the intensity oscillation comes with some kind of periodicity where you see that the transmitted intensity is minimum at half size g or one and a half size g, and so on and are you can say that the diffraction intensity is maximum at you know off sigh gr4 and half size g so you have the integral multiple of size g produces a Maxima in the bright field image like this where the dark pink intensity is minimum.

So it is other way around so you can see that the image producing a dynamical conditions where you see that the fringes are far apart so which is belong to the phase amplitude diagram of this kind that means s is smaller here and you can see that the fringes are widely spaced and you are able to see the dislocation details also in this, so this is a bright field image and this is a dark field image or complementary to each other and it is for a small s and this is for a same crystal obtained at the dynamical condition and you can see that the fringe pattern is very close.

And you see that the details of the dislocations are not clearly seen in this diagram as we see in a dynamical condition. So it clearly shows that you know this is the edge and we are traveling towards the thickness direction in this way, so this is an edge you see that so it is becoming a wedge here like this. So this is an edge and this is the thickness we are looking at so we are looking at from the top.

So you can clearly see this the fringe contrast and which explains by this dynamical theory and kinematic theory this is a classical example how this fringe contrast is understood and I hope you have some idea by looking at this images and schematics how the fringe contrast erases and please remember you have to keep that the initial slide which I showed in a perfect crystal how the electron beam goes and then how the intensity oscillation takes place for the transmitted beam and a diffracted beam and also look I am remember the size G the extinction distance then when it that is for a perfect crystal then when you have any defects like a wedge or a boundary or interface or a pit or a hole then how that intensity is affected is being explained by this two theories.

So this is where you have to be very clear about it and then you will be able to appreciate with this simple schematic and animations you will be able to appreciate this variation in the I mean intensity oscillation are the variation of the fringes as a function of yes whether if you have a small yes whether it is a largest you know the meaning of yes and then you will be able to see this and understand and appreciate please assume that one last point you have to appreciate before we move on to the next contrast mechanism.

Suppose if you assume that you have a perfect crystal and then electron beam passes through it and you do not have any defect and what you will see in a screen is only a bright illumination you do not see any features the moment you have any defects or inclined surface then your diffracted intensity will get affected by this and then you will see different intensity coming out of it or theoretically you can also see that you if you look at the first image what I have shown in the last class in order to explain the why particular grains are appearing a darker and grey and white and so on.

Theoretically you can assume that if suppose if your objective aperture is able to collect all the diffracted beam including the transmitted beam then also you will not see any contrast in the in the fluorescent screen then you will be then you can understand how this contrast arises by that way also you can assume for a perfect crystal but you will you will always produce a sample with lot of surface irregularities or non-uniformpneus or a taper section.

So you are bound to see these kind of a fringes or a contours or I mean any kind of fringes what we have seen so far so that is very clearly explained by this kinematical and dynamic theory now we will move onto the next contrast mechanism is called a mass thickness contrast first look at the initial remarks then we will get into the discussion.

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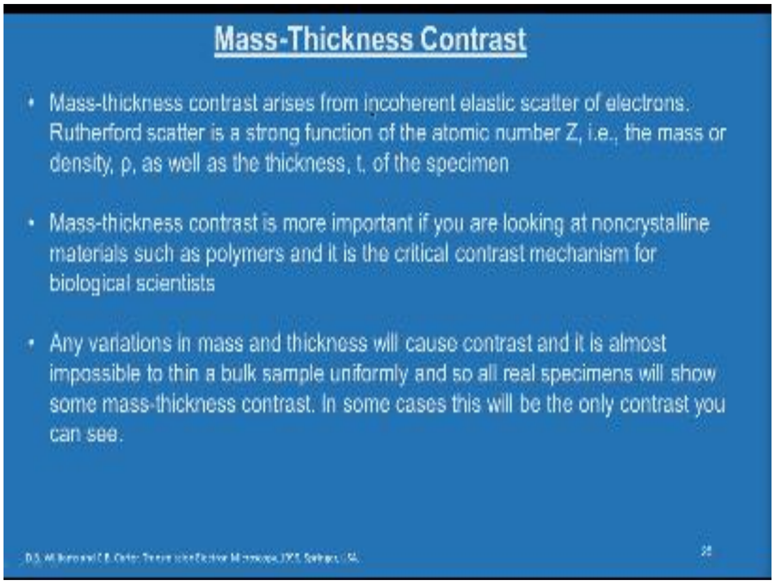

Mass-Thickness Contrast

- Mass-thickness contrast arises from incoherent elastic scatter of electrons. Rutherford scatter is a strong function of the atomic number Z, i.e., the mass or density, ρ, as well as the thickness, t, of the specimen
- Mass-thickness contrast is more important if you are looking at noncrystalline materials such as polymers and it is the critical contrast mechanism for biological scientists
- Any variations in mass and thickness will cause contrast and it is almost impossible to thin a bulk sample uniformly and so all real specimens will show some mass-thickness contrast. In some cases this will be the only contrast you can see.

Mass thickness contrast arises from an incoherent elastic scatter of electrons do therefore scatter is a strong function of atomic number z that is the mass or density Rho as well as the thickness T of the specimen mass thickness contrast is more important if you are looking at the no crystalline materials such as polymers and it is the critical contrast mechanism for biological scientists any variation in mass and thickness will cause the contrast and it is almost impossible to thin a bulk sample uniformly.

And so all trail specimens will show some mass thickness contrast in some cases this will be the only contrast you can see so this is very important point some mastic miss contrast is related to the a mass and density and the thickness and so on as I just mentioned in the previous contrast mechanisms you will never be able to produce a perfect samples with the uniform thickness so in general you will have whatever the contrast you are going to see whether it is going to I mean you are going you are looking at a diffraction contester a specifically mass thickness contrast every specimen will have some contribution of the mass thickness contrast.

So you can only say that a diffraction contrast is the maximum are dominating contrast mechanism which is operating but we cannot say that a mass thickness contrast is zero there because your sample preparation will never be perfect and you will have a mass or a density variation because of your non uniform surface on the top and bottom of the oil or any specimen so this is very important point to remember you will have a contribution from the mass thickness for the contrast always.

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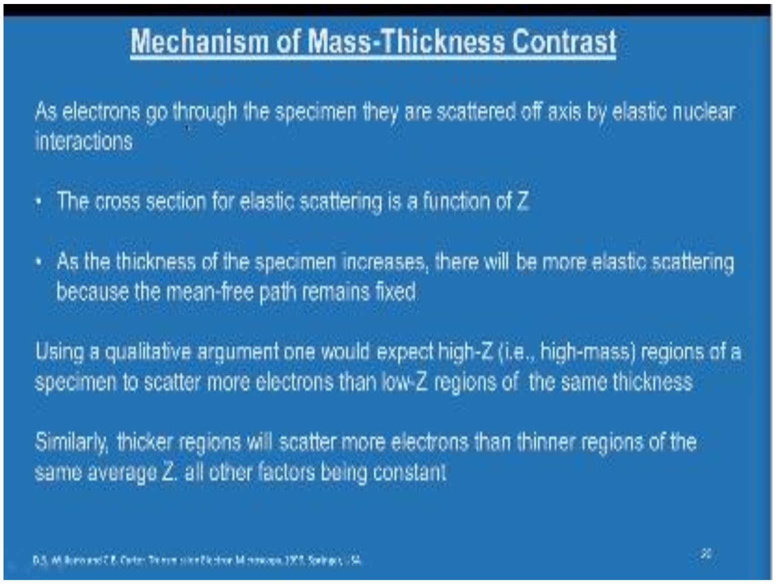

Mechanism of Mass-Thickness Contrast

As electrons go through the specimen they are scattered off axis by elastic nuclear interactions

- The cross section for elastic scattering is a function of Z
- As the thickness of the specimen increases, there will be more elastic scattering because the mean-free path remains fixed

Using a qualitative argument one would expect high-Z (i.e., high-mass) regions of a specimen to scatter more electrons than low-Z regions of the same thickness

Similarly, thicker regions will scatter more electrons than thinner regions of the same average Z. all other factors being constant

As electron go through the specimen they are scattered off axis by elastic nuclear interactions the cross section for a elastic scattering is a function of Z that is atomic number as the thickness of the specimen increases there will be or elastic scattering because the mean free path remains fixed using a qualitative argument one would expect high Z that is high mass regions of a specimen to scatter more electrons than the low see regions of the same thickness.

So a regions of high atomic number will scatter the electron beam more or are compared to the low atomic number regions similarly a thicker region will scatter the more electrons than the thinner regions of the same average Z all other factors being constant. So this is again an important factor now we will look at the schematic.

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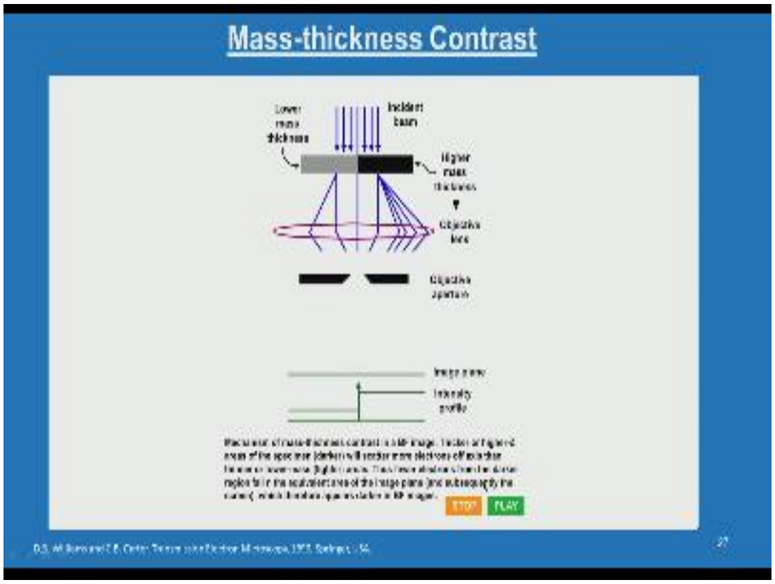

Mass-thickness Contrast

And then what I was trying to say in the previous slide also I will make it clear so look at this specimen which contains the lower mass thickness and this is a higher mass thickness electron beam pass through this and this is a the ray diagram for this and what you are seeing in the bottom is the intensity profile which for the two respective regions.

So what is shown here is mechanism of mass thickness contrast in a bright field image thicker or higher Z areas of the specimen will scatter more electron of axis than the thinner or lower mass areas thus fewer electrons from the darker region fall in the equivalent area of the image plane and subsequently on the screen which therefore appears darker in a bright field image see you have to be very careful here when you say that that higher atomic number region will scatter the electron more here in this particular ray diagram.

It is shown in the form of how much the scattered electrons are pushed half the axis that is move away from the axis compared to the a low-mass thickness region so that is that is one way of looking at it so you should not get confused with that so the more number of electrons are taken half axis are moved away from the axis so you have very few electrons reaching the image plane, so that means the intensity will be very different intensity profile will be very different for the both the cases. So thus the fewer electrons from the darker regions fall into the equivalent area of the image plane which therefore appear darker in the bright field images.

So that is a idea and the finally we will discuss about a phase contrast imaging so contrast in tem images can arise due to the differences in the face of electron wave scattered through a thin specimen this particular imaging is very difficult to interpret as it very sensitive to many factors the appearance of the image varies with the small changes in the thickness orientation or scattering fact of the specimen and variation in the focus or astigmatism of the objective lens its sensitivity is the reason phase contrast can be exploited to image the atomic structure of this thin specimen.

The primary distinction between the phase contrast image and other imaging in TM is the number of beams collected by the objective aperture or electron aperture the more beams collected the higher the resolution so the basic difference between the diffracted or amplitude contrast image or diffraction contrast image here is we are going to select a single diffracted beam are a many affected beam to interfere with the transmitted beam please understand the as we discussed in the dynamical theory that we talked about a two beam interaction.

So there is a possibility in phase contrast image whether you whether you can allow the two beam to interfere to form an image or you can select many diffraction rays or refracted beams to interact with the transmitted be in order to obtain the a phase contrast image, so let us look at the origin of the lattice fringes we can understand the origin of the lattice fringes by allowing two beams that is transmitted beam and the diffracted beam to interfere we can write the total wave function as sy is equal to V 0 Z Exponential 2 pi I ki dot R plus phi g z exponential 2 pi k d dot R this is for a transmitted and the diffracted beam interference a total intensity function.

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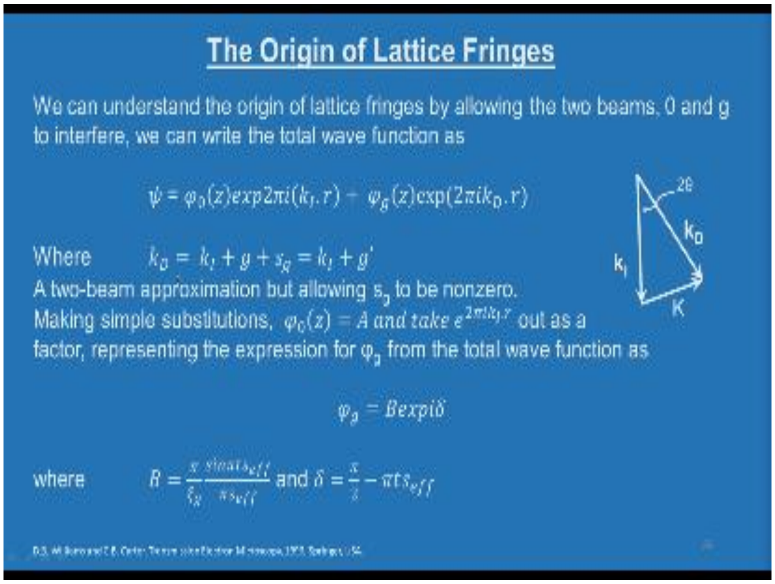

The Origin of Lattice Fringes

We can understand the origin of lattice fringes by allowing the two beams, 0 and g to interfere, we can write the total wave function as

$$\psi = \varphi_0(z) \exp 2\pi i(k_I.r) + \varphi_g(z) \exp(2\pi i k_D.r)$$

Where $k_D = k_I + g + s_g = k_I + g'$

A two-beam approximation but allowing s_g to be nonzero.

Making simple substitutions, $\varphi_0(z) = A$ and take $e^{2\pi i k_I.r}$ out as a factor, representing the expression for φ_g from the total wave function as

$$\varphi_g = B \exp i\delta$$

where $B = \frac{\pi}{\xi_g} \frac{\sin \pi t s_{eff}}{\pi s_{eff}}$ and $\delta = \frac{\pi}{2} - \pi t s_{eff}$

Where KD is equal to K I plus G Plus SG this is a deviation parameter please understand this comes from that factorial notation of the diffraction this K can be represented as a G refraction vector allowing a two beam approximation but allowing sg2 to be anon zero here making simple substitutions like V 0 Z = a and take this exponential term out as a factor representing the expression for Fiji from the total wave function as Fiji is equal to be exponential i delta we can define this term separately b is equal to π by φ g times sine pi t s effective by sign s effective and δ = π / 2 - PI T s effective.

So the dynamical theory also recommends to use yes effective instead of s which is nothing but s effective is equal to square root of s square plus 1 by sighs square so where s IG is the extinction distance we will not get into the details for at this moment so this effective is nothing but square root of s square plus 1 by sy z square, for the reasons where it takes care of other parameters like absorptions and so on which we will not discuss it here so you should not just confuse that what is s effective s effective is in another parameter where it also takes care of the absorption which is given by that expression what I just mentioned.

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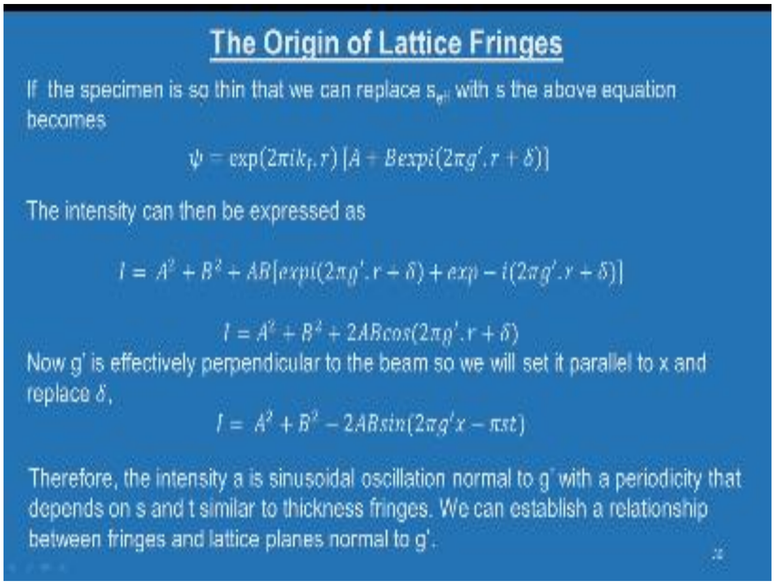

The Origin of Lattice Fringes

If the specimen is so thin that we can replace s_{eff} with s the above equation becomes

$$\psi = \exp(2\pi i k_I . r)\,[A + B\exp i(2\pi g'.r + \delta)]$$

The intensity can then be expressed as

$$I = A^2 + B^2 + AB[\exp i(2\pi g'.r + \delta) + \exp - i(2\pi g'.r + \delta)]$$

$$I = A^2 + B^2 + 2AB\cos(2\pi g'.r + \delta)$$

Now g' is effectively perpendicular to the beam so we will set it parallel to x and replace δ,

$$I = A^2 + B^2 - 2AB\sin(2\pi g'x - \pi st)$$

Therefore, the intensity a is sinusoidal oscillation normal to g' with a periodicity that depends on s and t similar to thickness fringes. We can establish a relationship between fringes and lattice planes normal to g'.

So if the specimen is so thin that we can replace s effective with s the above equation becomes sigh exponential 2 pi ki dot r into a plus B exponential I 2 pig prime dot r plus delta so the total intensity can be expressed in this fashion now g prime is effectively perpendicular to the beam so we will set it parallel to the x and replace δ in this fashion. So the intensity is as in soy del oscillation normal to G’ with a periodicity that depends on s and T similar to the thickness fringes we can establish a relationship between fringes and the lattice planes normal to g’.

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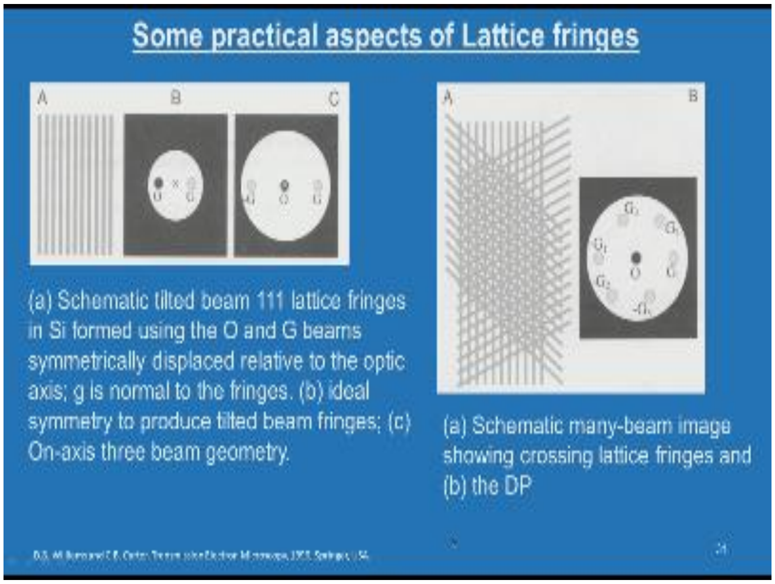

Some practical aspects of Lattice fringes

(a) Schematic tilted beam 111 lattice fringes in Si formed using the O and G beams symmetrically displaced relative to the optic axis; g is normal to the fringes. (b) ideal symmetry to produce tilted beam fringes; (c) On-axis three beam geometry.

(a) Schematic many-beam image showing crossing lattice fringes and (b) the DP

Suppose if you look at the how the lattice fringes are formed this to schematic will illustrate that point you have the schematic tilted beam 111lattice fringes in a silicon formed using a transmitted beam and a defector means symmetrically placed displaced relative to the optic axis G is normal to the fringes so G is normal to the fringes like this, so this is the typical a fringe pattern you get when you use a tilted beam configuration where you see that this is an optical axis and this is a transmitted beam this is a diffracted beam.

So we are allowing only one diffracted beam to the objective opportune to interfere and then you will get this kind of fringes and what you see is an ideal symmetry for to produce a tilted beam fringes like this where you have the transmitted beam and the optic axis are or you can say that the planes what we are I mean diffracting are looked up looked edge on, so in that case you will you will see with only g you will not produce sis equal to 0 that is exact condition you need to consider minus g also to establish 0 you will be able to produce this kind of a fringes.

And if you use many beam diffracted beam you will be able to produce a cross lattice fringes in a diffraction pattern please note that though you may have the relation between the distance between the diffracted spot which will have some relation with the lattice fringe spacing but this is nothing to do with the atom positions in the crystal that is why you have to be very careful this high resolution images they are not actually a lattice planes they are only a lattice fringes which has some relation with the spacing atomic plane spacing in a real lattice.

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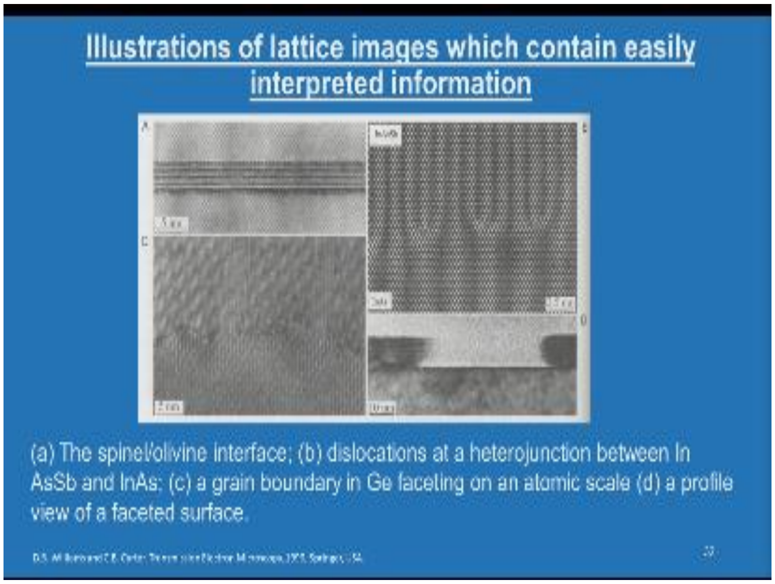

Illustrations of lattice images which contain easily interpreted information

(a) The spinel/olivine interface; (b) dislocations at a heterojunction between In AsSb and InAs: (c) a grain boundary in Ge faceting on an atomic scale (d) a profile view of a faceted surface.

So this is one typical example of the spinal 11 interface this is an a. a b is a dislocation at the hetero junction between fin DM arsenic antimony and arsenic here what you are seeing here see is a grain boundary in a germanium faceting on atomic scale d is a profile view of a faceted surface so this is a typical lattice images of different material, so these lattice fringes are not directly related to atomic positions but they have indirect relations like the spacing lattice spacing will have some relations. So I just want to introduce this pattern let us look at the other important point.

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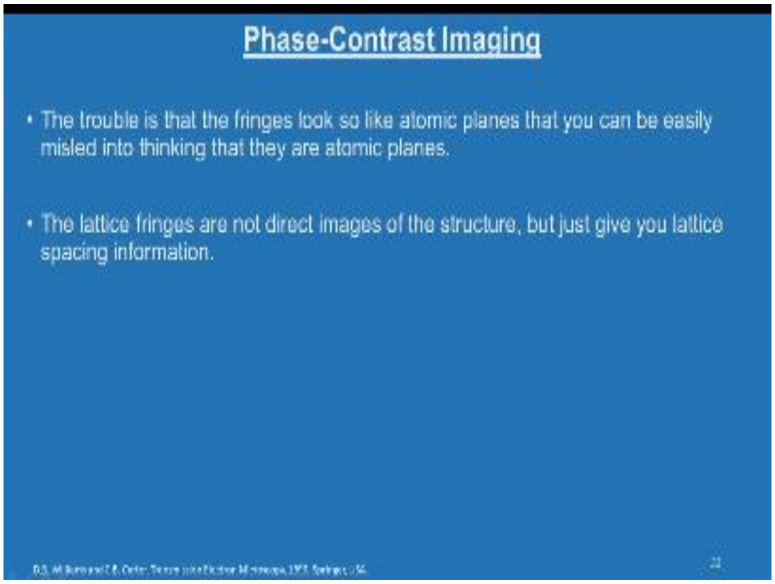

Phase-Contrast Imaging

- The trouble is that the fringes look so like atomic planes that you can be easily misled into thinking that they are atomic planes.
- The lattice fringes are not direct images of the structure, but just give you lattice spacing information.

The trouble is that the fringes look like look like atomic planes that you can be easily misled into thinking that they are all they are atomic planes the lattice fringes are not direct images of the structure but just give you the lattice spacing information. So just want you to know what is phase contrast image and what it gives and how this is looking like and what kind of information you can take that is all I wanted to give and in that particular explain I mean this case we have just taken a to beam interaction that is one diffracted beam and transmitted beam interaction.

And like what how we well an equation are described in a dynamical theory of to be me interaction, so like that you will be able to produce a lattice fringes and then and if you want to look at the much more detailed about anatomic position there are things and I mean bastes and means of doing it and which requires a lot of image simulation and so on which is not be a scope of this particular course so my idea is just to introduce and you should know what is phase contrast imaging to that extent II wanted to bring this imaging lecture to a conclusion and for a much more detailed analysis of this phase contrast imaging you need to go through a specialized and exclusive transmission electron microscopy course.

So I hope all that imaging techniques and the contrast mechanisms we have discussed in the last two classes must have given you some basic idea about how the contrast which we see in the TM is very I mean different from what you see in a in a conventional optical microscope and in scanning electron microscope. So you should be in a position to at least appreciate the differences between these three techniques after going through this courses I hope these lectures were useful to identify the fundamental ideas. Thank you.

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