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Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-25

by…

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Advanced Characterization Techniques

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Hello everyone I am Dr. Nilesh Prakash Gurao an assistant professor with the Department of material science and engineering at Indian Institute of Technology Kanpur, while recovering a small module in the course advanced characterization techniques. So the different things that we are going to cover in this module are as follows first and foremost we are going to have a brief introduction to x-rays.

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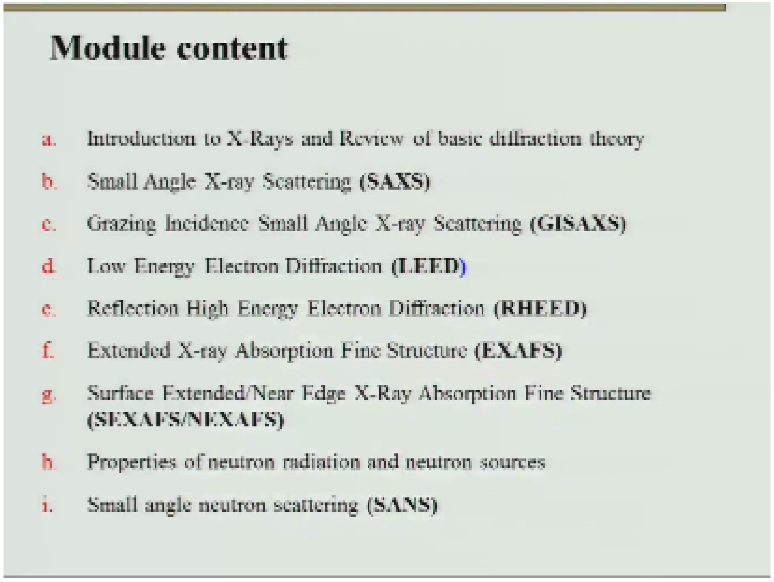

Module content

a. Introduction to X-Rays and Review of basic diffraction theory
b. Small Angle X-ray Scattering **(SAXS)**
c. Grazing Incidence Small Angle X-ray Scattering **(GISAXS)**
d. Low Energy Electron Diffraction **(LEED)**
e. Reflection High Energy Electron Diffraction **(RHEED)**
f. Extended X-ray Absorption Fine Structure **(EXAFS)**
g. Surface Extended/Near Edge X-Ray Absorption Fine Structure **(SEXAFS/NEXAFS)**
h. Properties of neutron radiation and neutron sources
i. Small angle neutron scattering **(SANS)**

Which I am sure you would have already had in preliminary courses on characterization of materials having done that we are going to have a basic diffraction theory review in a couple of slides after that, however we will look at advanced techniques involving scattering and diffraction like small-angle x-ray scattering and grazing incidence small-angle x-ray scattering after understanding these advanced techniques we are then going to move to electron diffraction and study low-energy electron diffraction as well as reflection high-energy electron diffraction.

After this we are going to cover extended x-ray absorption fine structures and surface extended near at x-ray absorption fine structures, after covering x-ray and electron diffraction we will go and have an understanding about neutron diffraction and essentially study small-angle neutron scattering. I will like to bring to your notice that what all techniques that we have enumerated over here are pretty advanced techniques and our techniques which we do not use on a day-to-day basis in our labs having said that the basic purpose of this course is to acquaint you with this state-of-the-art scattering and diffraction techniques which you will be using for specific research related problems. So let us first go back and have a look at the basics of x-ray diffraction.

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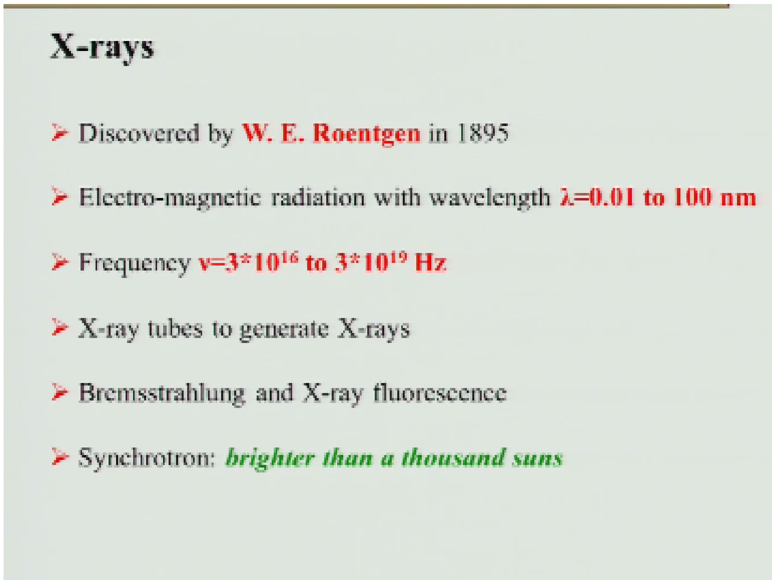

X-rays

- Discovered by **W. E. Roentgen** in 1895
- Electro-magnetic radiation with wavelength **λ=0.01 to 100 nm**
- Frequency **$\nu=3*10^{16}$ to $3*10^{19}$ Hz**
- X-ray tubes to generate X-rays
- Bremsstrahlung and X-ray fluorescence
- Synchrotron: ***brighter than a thousand suns***

So all of us are aware that x-rays were discovered by W.E Roentgen in 1895, x-rays as we are aware are electromagnetic radiation with wavelength of the order of point zero one to 100 nanometer and frequency of 3 x 10^{16} 3 x 10^{19} Hertz all of us are aware that x-ray are generated using x-ray tubes and which produce Bream styling as well as characteristic x-ray peaks we use optics to separate the brain styling x rays from the characteristic x-rays and do conventional x-ray diffraction studies, having said that another important tool I am going to talk about different sources of x-rays and how they are generated as well as the optics in the due course of this lecture.

But before that we are going to study how exactly diffraction or for that matter how do x-rays interact with matter who give us sufficiently important information.

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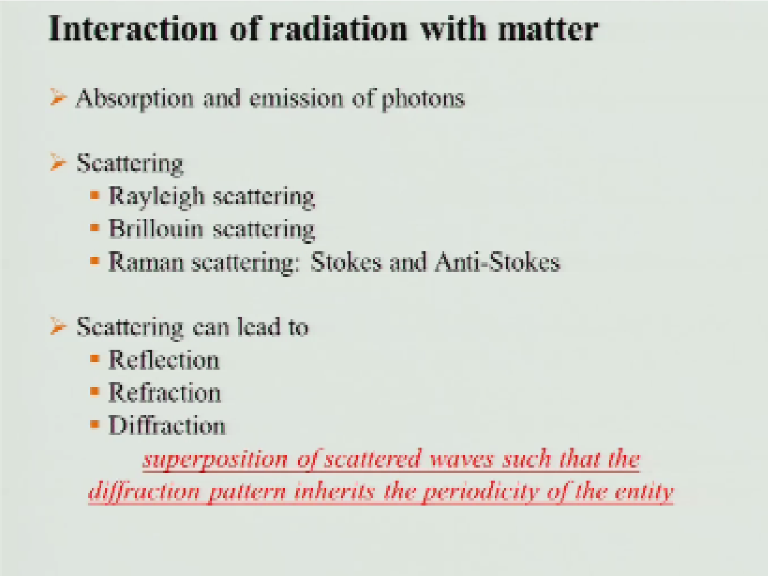

Interaction of radiation with matter

- Absorption and emission of photons
- Scattering
  - Rayleigh scattering
  - Brillouin scattering
  - Raman scattering: Stokes and Anti-Stokes
- Scattering can lead to
  - Reflection
  - Refraction
  - Diffraction

superposition of scattered waves such that the diffraction pattern inherits the periodicity of the entity

So let us first try to understand the interaction of radiation with matter we know that whenever a radiation I mean an electromagnetic radiation interacts with the matter it can lead to absorption as well as emission of photons, at the same time it can lead to what is known as scattering. Scattering can be classified as released scattering or where the energy is conserved brilliant scattering which occurs for liquids as well as Rayleigh scattering part of these scattering events which I have mentioned here will be covered in the course of this module as most of the characterization techniques to be introduced in the later part of this course are dependent on various scattering events.

We are also aware that scattering can lead to reflection, refraction, and diffraction, the most important of it is diffraction which is essentially defined as superposition of scattered waves such that the diffraction pattern inherits the periodicity of the entity. Having said that I am going to spend a couple of slides later focusing on what exactly is diffraction, but before we do that let us try to understand how do x-ray interact with matter.

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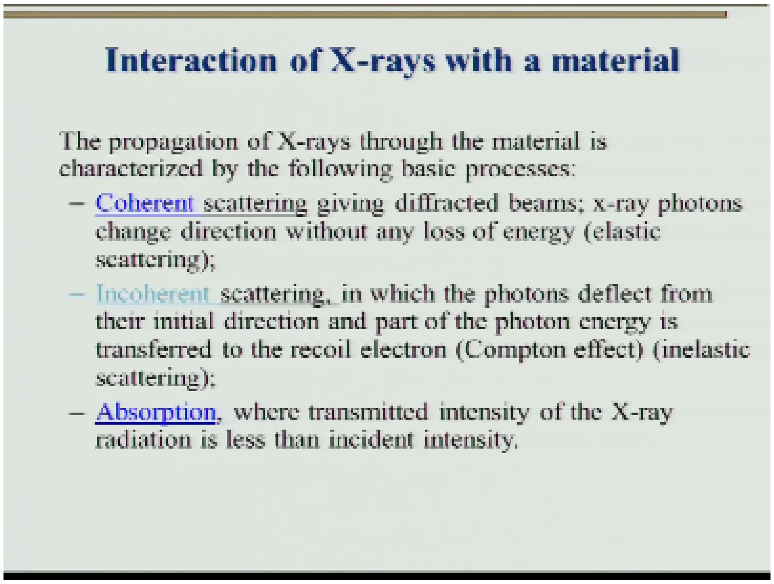

Interaction of X-rays with a material

The propagation of X-rays through the material is characterized by the following basic processes:

- Coherent scattering giving diffracted beams; x-ray photons change direction without any loss of energy (elastic scattering);
- Incoherent scattering, in which the photons deflect from their initial direction and part of the photon energy is transferred to the recoil electron (Compton effect) (inelastic scattering);
- Absorption, where transmitted intensity of the X-ray radiation is less than incident intensity.

We know that the propagation of x-rays through matter is characterized by a below mentioned three basic processes namely coherent scattering wearing the x-ray photons do not change their energy why there is also what is known as incoherent scattering in which the photons reflect from their initial direction and part of the photon energy is transferred to the recoil electron and is classically known as the Compton effect of due to elastic scattering.

Another important phenomenon which occurs with x-rays interaction with metal is x-ray absorption where the transmitted intensity of the x-ray radiation is less than the incident intensity having said that our major interest lies in the fraction.

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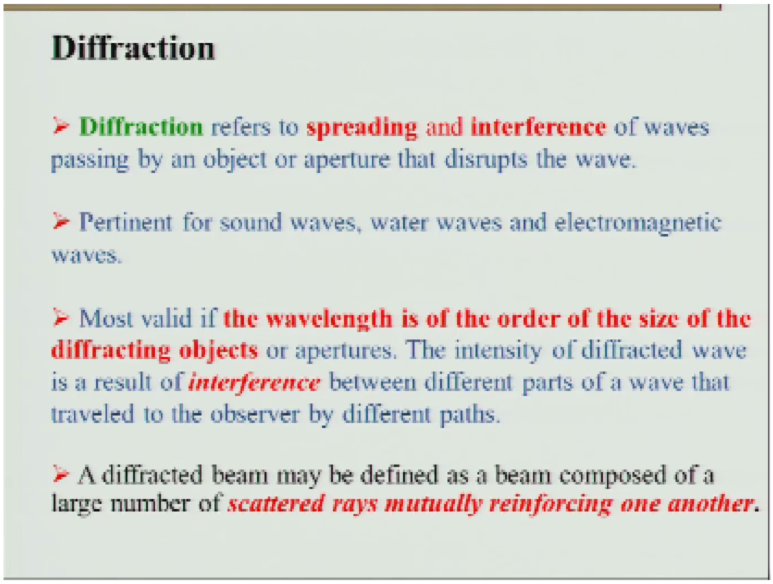

Diffraction

- **Diffraction** refers to **spreading** and **interference** of waves passing by an object or aperture that disrupts the wave.
- Pertinent for sound waves, water waves and electromagnetic waves.
- Most valid if **the wavelength is of the order of the size of the diffracting objects** or apertures. The intensity of diffracted wave is a result of ***interference*** between different parts of a wave that traveled to the observer by different paths.
- A diffracted beam may be defined as a beam composed of a large number of ***scattered rays mutually reinforcing one another***.

The fraction I would like to mention refers to spreading and interference of waves passing by an object or aperture that disrupts the view, it is pertinent for sound water as well as electromagnetic waves if you want to study diffraction the best way is to go out any reservoir of water where the water is stable and try to throw two pebbles and see how the two waves are formed and how do they interact with each other.

Another important point that is we mentioned regarding diffraction is that the diffraction effect is most valid if the wavelength is of the order of the size of the diffracting objects, these deflecting objects may be apertures or maybe other objects which lead essentially to spreading or for that matter scattering of waves it is to be mentioned that a diffracted beam may be defined as a beam composed of a large number of scattered rays which are reinforcing one another the most important point to be noted in the above discussion is that the scattered rays which mutually reinforce one another that means their amplitudes add up leads to diffraction.

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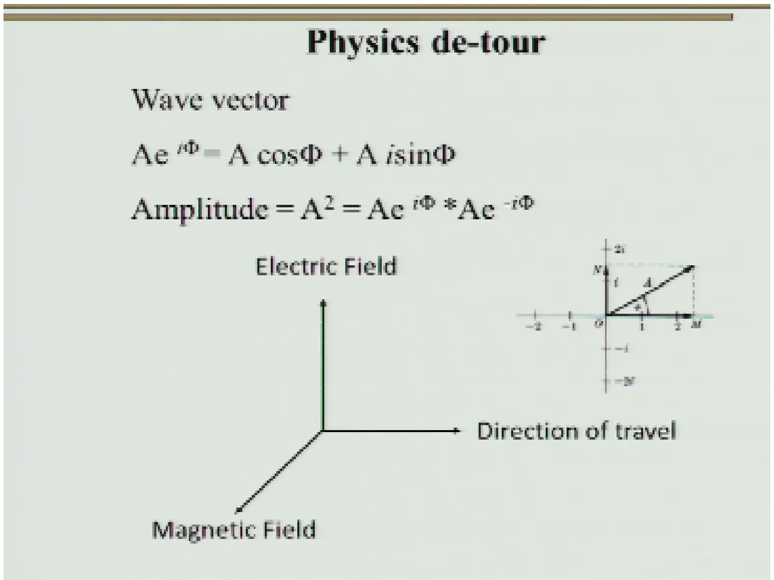

Physics de-tour

Wave vector

$Ae^{i\Phi} = A\cos\Phi + A\, i\sin\Phi$

$\text{Amplitude} = A^2 = Ae^{i\Phi} * Ae^{-i\Phi}$

So before we go ahead with diffraction let us spend some time on understanding the wave particle duality of electromagnetic radiation, we all know that light or any electromagnetic radiation can be considered as a particle with energy H nu which is a photon if you talk about quantum mechanics while in terms of classical physics we can always consider it as a wave and define it by of a vector which is a complex vector which can be given by AE I $^{\pi}$ where which can be decoupled amplitude which is given by the product of the complex amputation of the wave and its complex conjugate and is directly proportional to a square.

We also know that electromagnetic radiation including light rays as well as x-rays comprised of electric field and magnetic field which are mutually orthogonal to each other and that the direction of travel of light is normal to the electric field as well as the magnetic field will be using these concepts in understanding how does diffraction occur in the course of this module.

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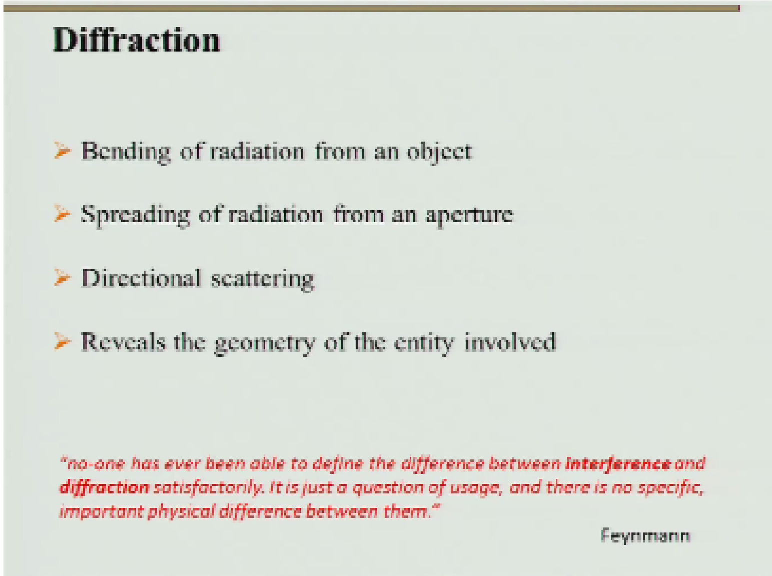

Diffraction

- Bending of radiation from an object
- Spreading of radiation from an aperture
- Directional scattering
- Reveals the geometry of the entity involved

*"no-one has ever been able to define the difference between **interference** and **diffraction** satisfactorily. It is just a question of usage, and there is no specific, important physical difference between them."*

Feynmann

So coming back to diffraction has had already mentioned diffraction is bending of radiation from an object or spreading of radiation from an aperture it is also defined as directional scattering and is very important for revealing the geometry of the entity involved, because the diffraction pattern essentially inherits the geometry of the entity that leads to diffraction having said that there is another important interesting phenomena which we have studied during the course of our high school physics which is interference from apertures and there is a very nice similarity between diffraction and interference.

And therefore in this module we are going to understand diffraction through interference and I would like to quote and import a very nice saying by none other than filming himself who said that no one has ever been able to define the difference between interference and diffraction satisfactorily, it is just a question of usage and there is no specific important physical difference between them.

Therefore we are going to fall an approach wherein we try to understand diffraction through interference but before we do that let me just throw in a bit of history pertaining to x-ray diffraction as I had already mentioned it was in 1896 that x-rays were discovered by Ron chair however it was not until 1912 when von Laue Friedrich and nipping passed x-rays through crystal of zinc sulfide.

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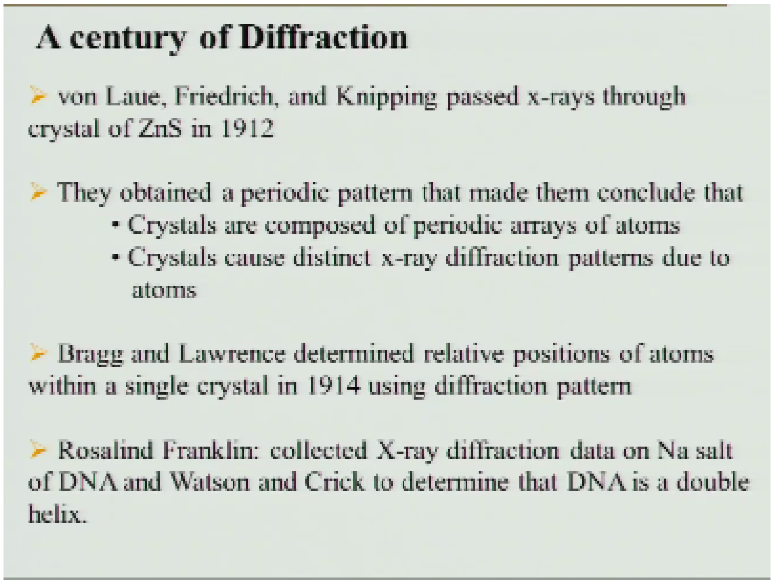

A century of Diffraction

- von Laue, Friedrich, and Knipping passed x-rays through crystal of ZnS in 1912
- They obtained a periodic pattern that made them conclude that
  - Crystals are composed of periodic arrays of atoms
  - Crystals cause distinct x-ray diffraction patterns due to atoms
- Bragg and Lawrence determined relative positions of atoms within a single crystal in 1914 using diffraction pattern
- Rosalind Franklin: collected X-ray diffraction data on Na salt of DNA and Watson and Crick to determine that DNA is a double helix.

The obtained a periodic pattern that made them conclude that crystals are composed of periodic arrays of atoms and crystals cause distinct x-ray diffraction pattern due to atoms brag ms Bragg and Lawrence determine the relative positions of atoms within a single crystal in 1914 using x-ray diffraction for this the Bragg father-son duo also won a Nobel Prize it was much later when the you know diffraction was used to decipher the structure of DNA and the well-known very famous double helix structure of DNA was deciphered first firstly using x-ray diffraction.

Thus we see that exceed diffraction has come a long way over the last century and the development of state-of-the-art characterization techniques involving x rays has led to a fundamental path-breaking discoveries over the last century. So before we go ahead and talk about diffraction let me just talk about interaction of light of waves for that matter any electromagnetic waves with matter or for that matter with an aperture.

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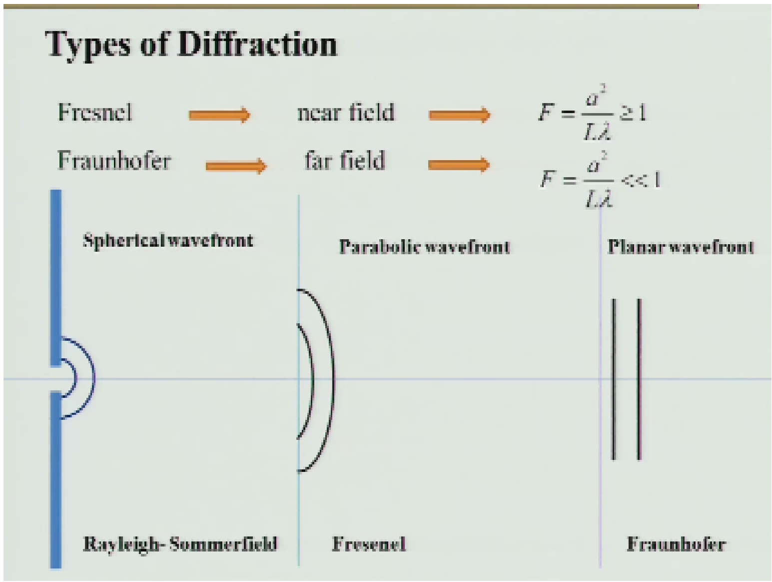

Types of Diffraction

Fresnel → near field → $F = \frac{a^2}{L\lambda} \geq 1$

Fraunhofer → far field → $F = \frac{a^2}{L\lambda} << 1$

Spherical wavefront

Parabolic wavefront

Planar wavefront

Rayleigh- Sommerfield

Fresenel

Fraunhofer

So for this we are going to look at interaction of light waves in the visible spectrum with an aperture, so we know that whenever a light wave interacts with an aperture each and every point in the aperture acts as a source of spherical wave front according to Huygens view principle. This is what has been shown in the first part wherein we can see how a spherical wave front is from the aperture.

However as we move away far from this the distance increases from the aperture we see that the wave front becomes parabolic this is the regime of what is known as trundle diffraction the condition for which is given right over there we're in the $f = a^2 / L \lambda$ that is greater than or equal to 1 where a is the size of the aperture and L is the distance between the aperture and the image plane having said that what we are going to mostly deal with is the front of a diffraction in which the wave front which started a spherical then became parabolic at very large l it essentially becomes planar. And this is what we are going to deal with mostly during the course of x-ray diffraction.

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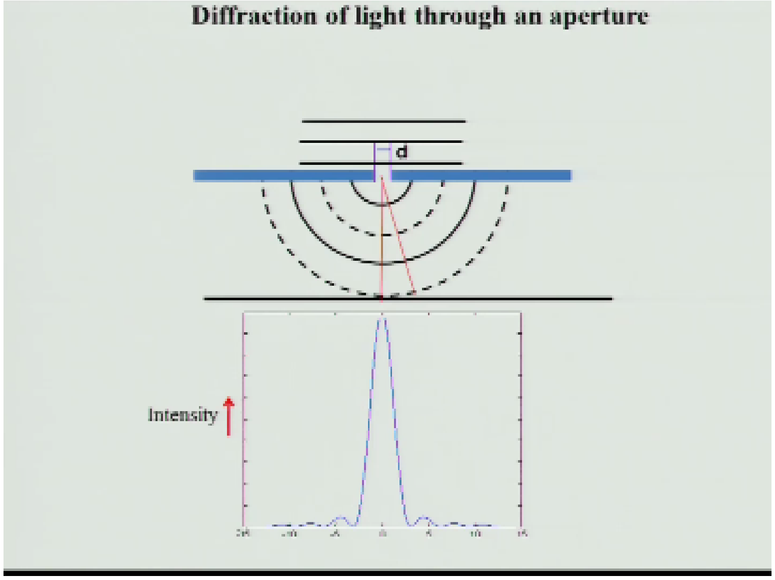

Diffraction of light through an aperture

Having said that as I have already mentioned we are going to try and understand diffraction in terms of interference, so whenever we have a wave a planar wavelength of light interacting with an aperture we see that instead of getting one high intensity we do not we get a spread of intensity as shown in the figure below.

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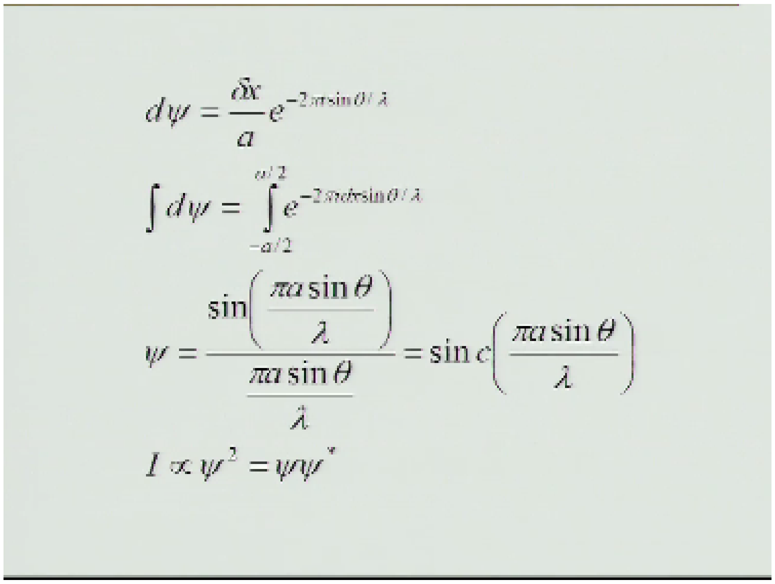

$$d\psi = \frac{\delta x}{a} e^{-2\pi x \sin\theta / \lambda}$$

$$\int d\psi = \int_{-a/2}^{a/2} e^{-2\pi dx \sin\theta / \lambda}$$

$$\psi = \frac{\sin\left(\frac{\pi a \sin\theta}{\lambda}\right)}{\frac{\pi a \sin\theta}{\lambda}} = \sin c\left(\frac{\pi a \sin\theta}{\lambda}\right)$$

$$I \propto \psi^2 = \psi\psi^*$$

Just to give you an idea of how we can understand the mechanics of wave I have just noted down the wave equation and integrated it to show again that the intensity is directly optional 2 ^{2} of the z and that the wave function itself that you get on an interaction with the aperture is a function of φ of π a φ θ / λ / π a φ θ / λ which is nothing but a φ c function of π φ θ / λ let us kind of neglect the mathematics for the time being and see what is the physical significance of it.

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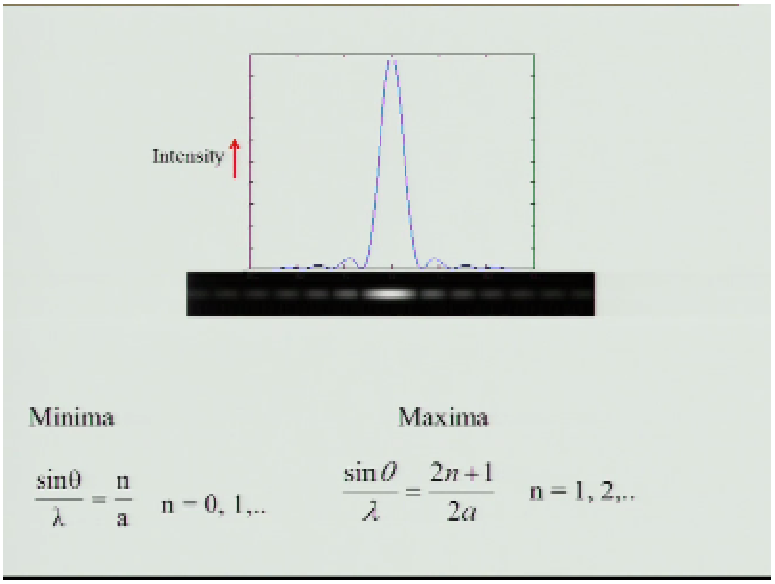

Minima

$$\frac{\sin\theta}{\lambda} = \frac{n}{a} \quad n = 0, 1,..$$

Maxima

$$\frac{\sin\theta}{\lambda} = \frac{2n+1}{2a} \quad n = 1, 2,..$$

And we all know that whenever optical or visible light passes through a small aperture we do get a pattern like this wherein there is one single Maxima at the same time there are some secondary maxima and the condition for Maxima as well as minima is given below. So you can see that whenever φ θ / λ = n / a, we do get a minima and there is no intensity however whenever φ θ / λ is 2n +1 / 2a where is the length of the aperture we do get a Maxima.

Now this is for only one aperture what if we are having two apertures which is the classical Young's double slit experiment this experiment and the results of it we have covered in our high school days and we all know that ends double slit experiment leads to an interference pattern which can be explained on the basis of constructive interference and destructive interference.

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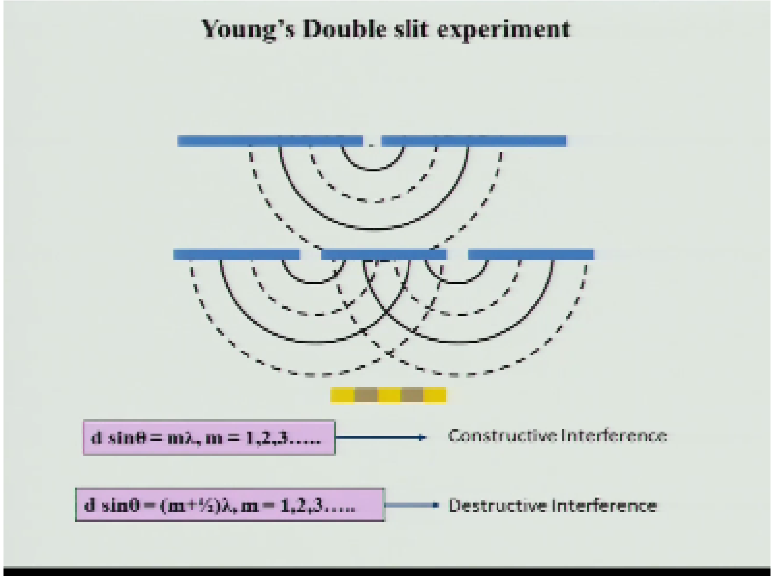

Young's Double slit experiment

$d \sin\theta = m\lambda, m = 1,2,3.....$ → Constructive Interference

$d \sin\theta = (m+½)\lambda, m = 1,2,3.....$ → Destructive Interference

It is explained that whenever two waves meet in phase their amplitudes get added up and leads to constructive interference, however if the two waves need out of phase then their amplitudes get canceled and if they are having the same amplitude you get complete or total destructive interference and there is no intensity the condition for constructive interference is that $D \varphi \theta = n \lambda$ which essentially means that the path difference between the two waves is an integral multiple of λ.

Therefore the two waves are meeting in phase and their amplitudes get added up, however if the phase difference is not an integral multiple of λ and in fact if it is M plus half λ the two phases are the two waves rather are completely out of phase and therefore their amplitudes cancel each other and we get total destructive interference. Well having understood the eggs double slit experiment completely opposite experiment.

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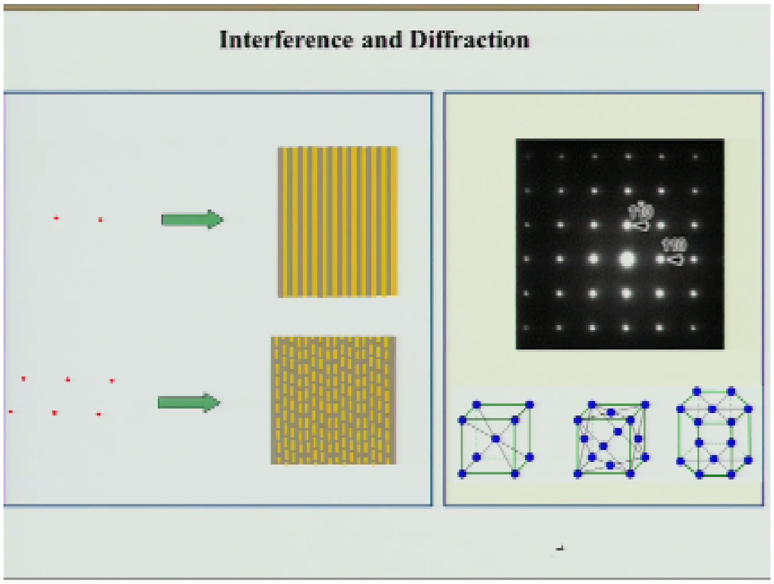

Interference and Diffraction

I like to show you how the yanks double slit experiment can be extended to 2d. Just to view an entire and understanding for interference and diffraction I have shown how we can get a diff interference pattern from two slits as observed as observed in the figure above add however if we are having an array of slits we do get a pattern comprising of dark spots which are shown here in yellow rather gray and bright spots which are shown here in terms of yellow color.

Similar thing we get when there is an interaction of electrons with matter and it lead it leads to what is classically known as a electron diffraction pattern which is quite routinely seen in seen in a transmission electron microscope. And this is because of diffraction occurring not from occurring from atoms here in we can clearly see the similarity between diffraction of electrons from atoms and interference of visible light with apertures.

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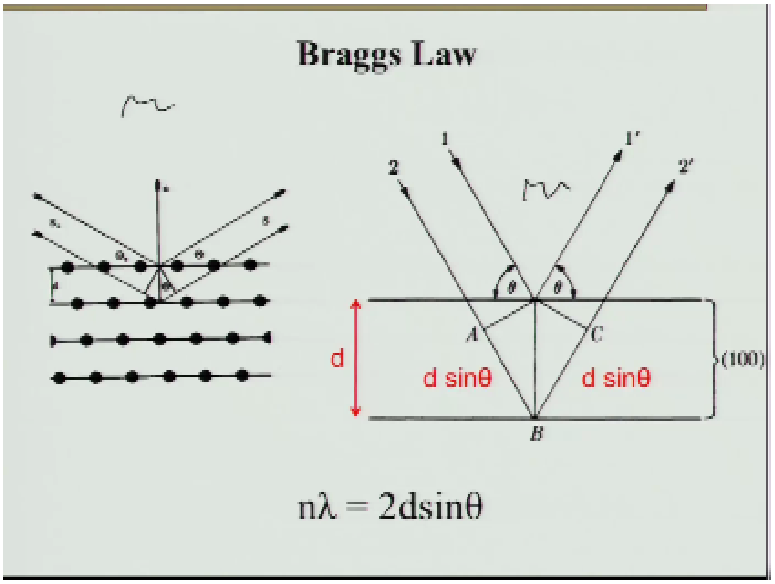

Braggs Law

$n\lambda = 2d\sin\theta$

And we all know from the basic course on characterization that we have undertaken that diffraction follows what is known as Bragg's law in order to understand what exact I will just repeat what is Bragg's law and we know that whenever there are two waves which are coming over a crystalline material the wave say one and two. The path difference between these two waves is essentially d φ θ + T φ θ which is nothing but a b + DC we know that if the overall path difference between waves 1 and 2 is an integral multiple of λ that is if n λ is equal to 2 d φ-λ the two waves one and two are going to meet in phase and their amplitudes are going to get added.

And hence we will say that the fraction has occurred, so this is the generalized definition of Bragg's law where n is known as the order of diffraction λ is the wavelength d is the interlunar spacing and λ is essentially the angle of incidence, having said that I like to mention that many a times refraction and reflection are used intermittently though these two are two different phenomena.

Now let us try to understand the geometry of Bragg's law before we proceed we know are the most important thing that is very essential for Bragg’s condition to be followed is that the incident beam and the normal of the reflection plane.
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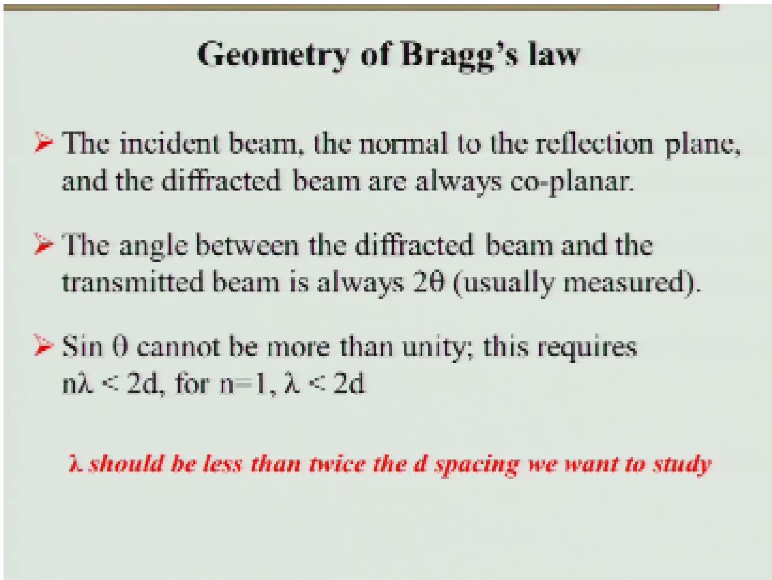

Geometry of Bragg's law

- The incident beam, the normal to the reflection plane, and the diffracted beam are always co-planar.
- The angle between the diffracted beam and the transmitted beam is always 2θ (usually measured).
- Sin θ cannot be more than unity; this requires $n\lambda < 2d$, for $n=1$, $\lambda < 2d$

λ should be less than twice the d spacing we want to study

Ss well as the diffracted beam how to be coplanar the angle between the diffracted beam and the transmitted beam is always 2 λ and this is what we measure if you recollect a normal x-ray diffraction pattern essentially comprises of i vs 2 λ and another important thing regarding the choice of radiation for the kind of study that you are doing is determined by the wavelength very you know that φ λ cannot be more than unity and therefore it requires that λ should be less than twice the d spacing we want to study.

There are other conditions which are laid down on the selection of wavelengths, but we will cover them as an in the due course of the module, another important thing which we would like to locate in the Bragg's law is that is what is known as the order of reflection.

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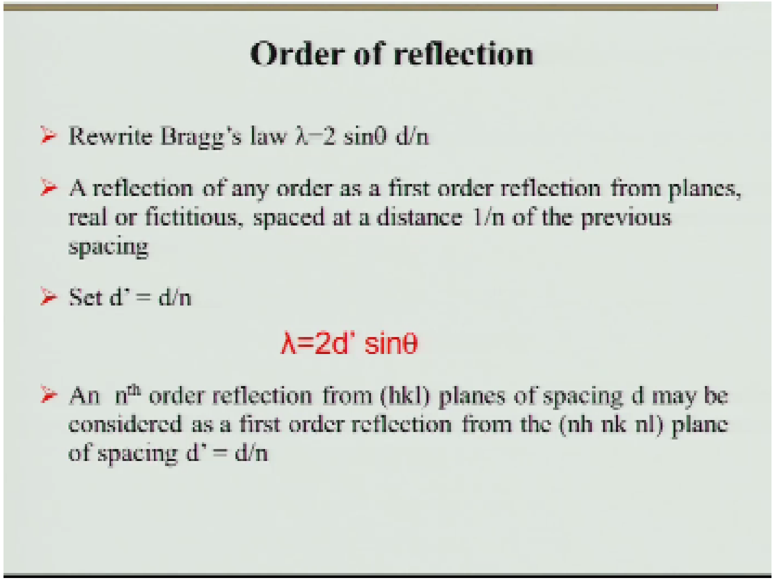

Order of reflection

- Rewrite Bragg's law λ=2 sinθ d/n
- A reflection of any order as a first order reflection from planes, real or fictitious, spaced at a distance 1/n of the previous spacing
- Set d' = d/n

$$\lambda = 2d' \sin\theta$$

- An n^{th} order reflection from (hkl) planes of spacing d may be considered as a first order reflection from the (nh nk nl) plane of spacing d' = d/n

So if you if you look at the branch log which is nothing but n λ or 2 d φ-λ and rearrange the terms we do get λ = 2 φ λ x D / N. And therefore we can say that a reflection of any order as a first-order reflection from planes real or fictitious spaced at a distance of 1 by n of the previous spacing, therefore an n order reflection can always be replaced with a first-order reflection from a real or fictitious planes spaced at a distance of 1 /n of the previous spacing.

Therefore if we set D' as d over n we do get λ = 2 d ^{a} φ λ put in other it means that an nth-order reflection from htl planes of spacing deal may be considered as a first-order reflection from the NH NK NL plane of spacing d' wherein the spacing d'= d / n.

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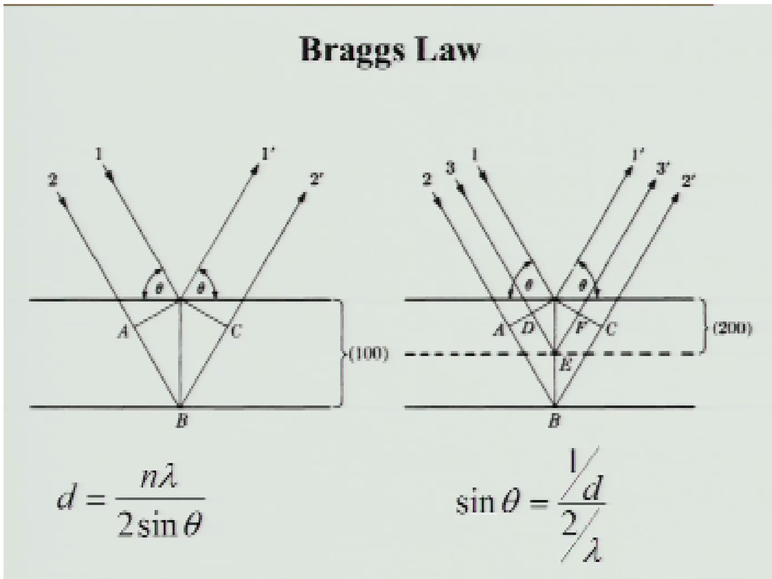

Braggs Law

$$d = \frac{n\lambda}{2\sin\theta}$$

$$\sin\theta = \frac{1/d}{2/\lambda}$$

The reason I am visiting Bragg's law again and again because is to emphasize the importance of what is going to be what is essentially known as reciprocal space so again here I rearranged the Bragg's law and jet d on one side and then I essentially separate out φ λ on the other side wherein I can write φ λ = 1 / d or 1 /d' / 2 / λ what is that one the basic advantage of writing the Bragg's law in this way will be enumerated in the next couple of slides.

The easiest way of approximating it is to look at the value of n, so if we look at 100 set of planes we get the first-order reflection wherein λ = 2 d φ-λ, however if you look at the second order reflection we go back and we see that the plane is supposed to lie right in between the two planes which we have shown already. Similarly we can imagine that the 300 or the third order reflection is going to lie at one-third the distance between the two planes.

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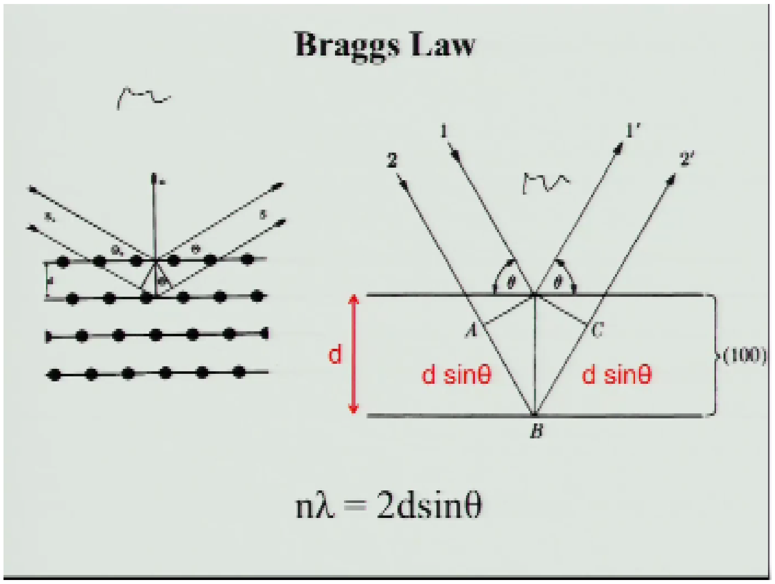

Braggs Law

$$n\lambda = 2d\sin\theta$$

Now if you go back to the two slider the last slide we see that if you go in between two set of planes we do see that there are no atoms so there is going to be no interaction between the x-rays and the atom which is absolutely essential for diffraction to occur. So how do we understand diffraction if at all there are no atoms in this plane that is where the concept of reciprocal lattice comes into comes for our help?

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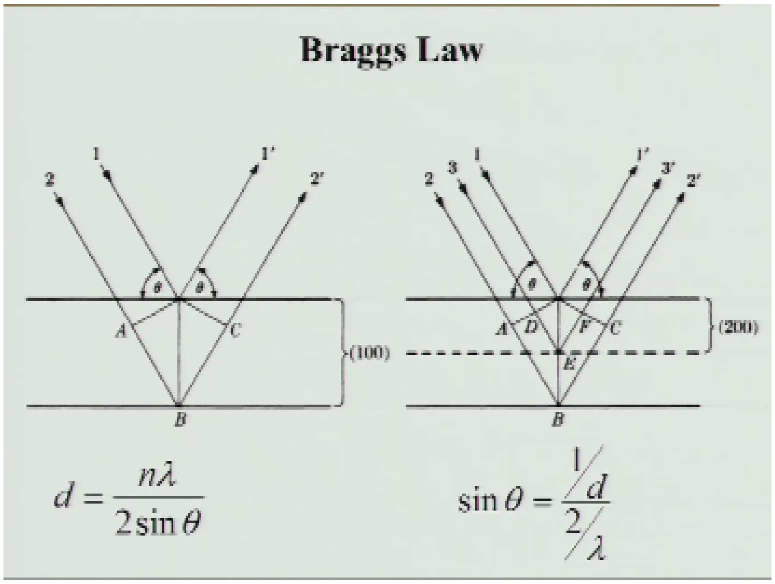

Braggs Law

$$d = \frac{n\lambda}{2\sin\theta}$$

$$\sin\theta = \frac{1/d}{2/\lambda}$$

If we look at the right the bottom right equation φ λ = 1 or D / 2 or λ you can hope that the fraction occurs when this to over λ which is the wavelength of choice that we have chosen correspond to any one of the point lying on 1 / d.

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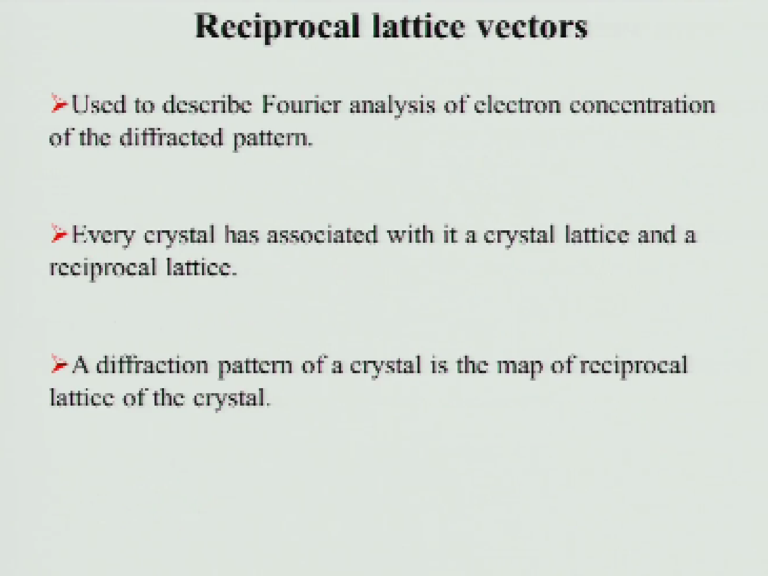

Reciprocal lattice vectors

- Used to describe Fourier analysis of electron concentration of the diffracted pattern.
- Every crystal has associated with it a crystal lattice and a reciprocal lattice.
- A diffraction pattern of a crystal is the map of reciprocal lattice of the crystal.

We know that as I mentioned or rather I you must be aware that all the diffraction or interference that we studied can be essentially described in terms of Fourier analysis of electron or electron concentration in the diffraction diffracted pattern. Every crystal therefore has a crystal lattice at the same time a reciprocal lattice associated with it a diffraction pattern of a crystal is nothing but the map of the reciprocal lattice of the crystal.

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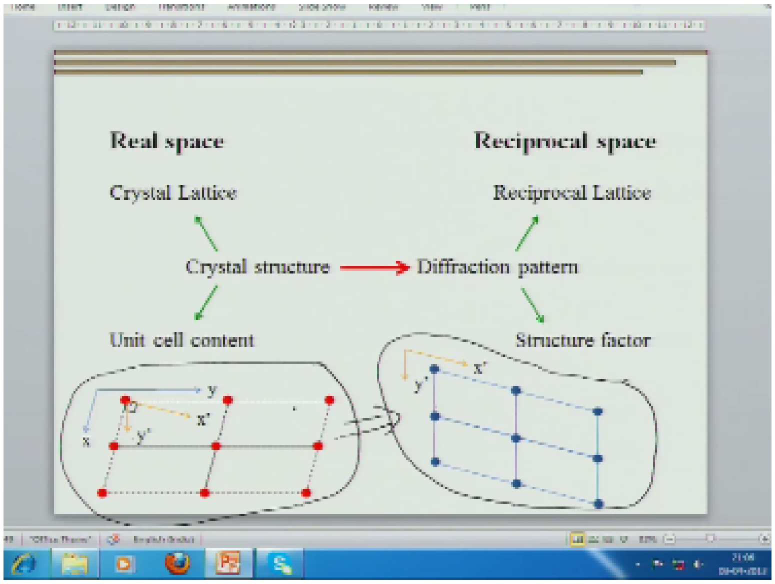

Real space

Reciprocal space

Crystal Lattice

Reciprocal Lattice

Crystal structure

Diffraction pattern

Unit cell content

Structure factor

In order to decipher what exactly is a reciprocal lattice, let me first say that let me first tell you that in a real space we do have crystals which has a particular crystal structure now the crystal structure essentially comprised of a lattice or a crystal lattice and a unit cell content while the diffraction pattern that we get using x-rays or for the matter electrons is essentially comprising of the reciprocal lattice. And the structure factor which determines the intensity of diffraction in order to decipher how how we construct reciprocal lattice does.

Let me not go into all the mats of it but just start with a simple 2d presentation wherein I have shown you a plane lattice which is which can be seen over here and you can see that this lattice which I have over here is characterized by basis vectors x and y okay. Now if I were to draw on normal to these two vectors which I have shown over here as y prime as well as X' where y prime is parallel to Y and X' is parallel to x and plot 1 over the distance so you can see over here that along why the distance between the two atoms is very large by then I go to reciprocal space I not only go 90^0 to Y but reduce the distance.

And that is where you see that my Y' over here is smaller than X' while in real space my x is smaller than Y so not only have we gone change the directions but also we have changed the length the magnitude of the directions therefore we can define y' has been normal to why but the distance between any two reciprocal points along y' is 1 over the distance along Y in the real space. Similarly we can define X and X' the whole concept that I am trying to convey is that if you have a real lattice which is shown over here it leads to a reciprocal lattice shown over here and there is a mapping function or we can go from the real lattice we can go to the reciprocal lattice which we see using x-ray diffraction.

So if we can understand how we go from real space to reciprocal space we are going to measure actually the reciprocal space by diffraction, so we can always come back and get some idea about the kind of real lattice that is existing.

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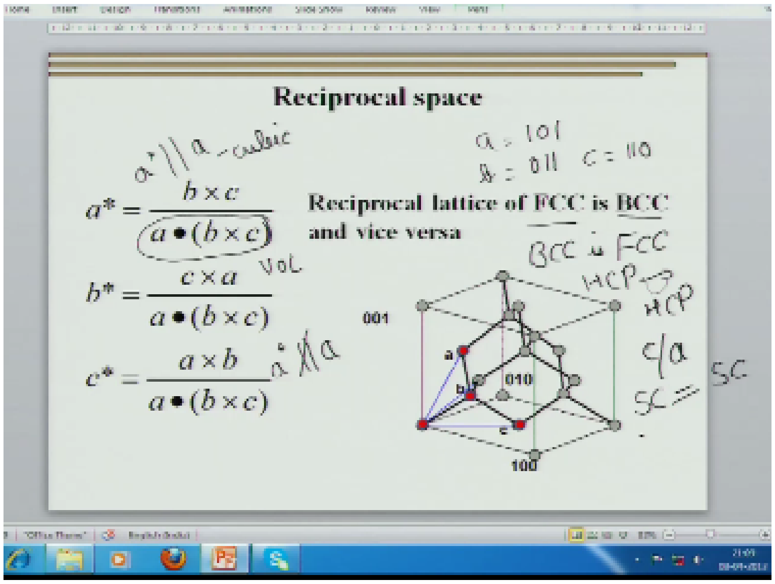

Reciprocal space

$a^* \parallel a$ - cubic

$$a^* = \frac{b \times c}{\boxed{a \bullet (b \times c)}}$$

vol

$$b^* = \frac{c \times a}{a \bullet (b \times c)}$$

$$c^* = \frac{a \times b}{a \bullet (b \times c)}$$

$a^* \nparallel a$

$a = 101$

$b = 011$ $c = 110$

Reciprocal lattice of FCC is BCC and vice versa

BCC is FCC

HCP → HCP

c/a

SC =

SC

So just a few words about reciprocal lattice if we go from 2d to 3d as had already mentioned that Y prime here was normal to Y, similarly if I start with say a cubic or orthogonal lattice I can start with the you know with ABC as the lattice parameters I can always get a star in the reciprocal space which is nothing but B x C /a dot B x C where a dot B x C essentially gives me the volume of the unit cell, and B x C gives me a vector which is normal to the two vectors B and C in case of cubic lattice my a star is parallel to A but it is not a necessary condition and for other lattices this condition may not be true.

So this is true only for cubic however East or may not be parallel if the lattice is non cubic however what are a rather a B and C if you are looking at FCC BCC and HCP structure I would like to mention that the ABC that we have chosen here are not the general ear general you know lattice parameters that we use like a = b = c and α = β = γ = 90 for any cubic lattices instead it is the primitive lattice are the basis vectors that we have to choose.

Therefore for FCC the A B and C that we choose are essentially the three different types of 101 we're in a is 101 b is 0 1 1 and C will be 110, so these are the three basis vectors that we have to choose another interesting thing which you must be aware is that the reciprocal lattice of FCC is BCC while that of BCC is FCC for HCP lattices we do have another HCP lattice, but the c by a ratio is changed one then we go from real space to reciprocal space for simple cubic the real lattice and the reciprocal lattice are the same okay.

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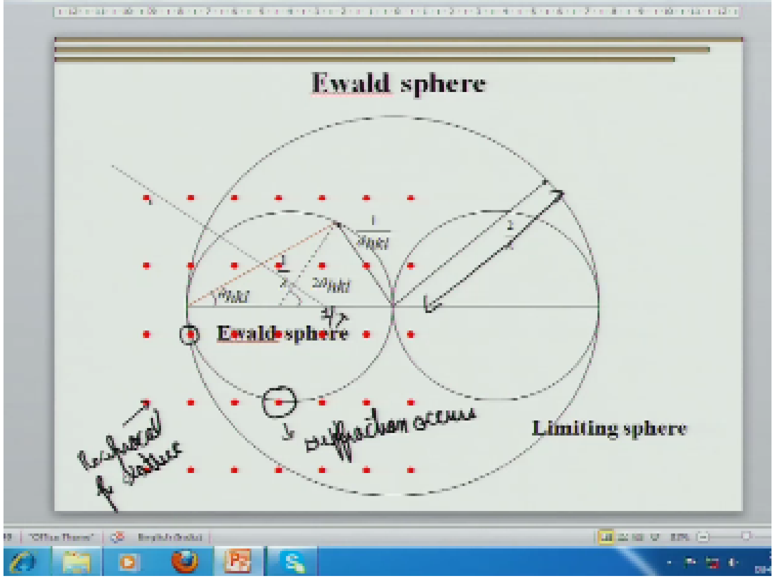

Ewald sphere

Now in order to understand diffraction as I had mentioned in the earlier slide that whenever it satisfies this particular condition of φ λ equal to 1 by D over 2 or λ to understand the geometry we can see how they actually diffraction occurs. So I can always draw a radius or rather a sphere with radius 2 over λ which is shown over here which is the limiting sphere and the eval sphere is essentially the small sphere that have shown over here with radius 1 / λ and diffraction has already mentioned occurs at particular point when the sphere or the circle as shown in this figure and any one of the point the reciprocal lattice points which are shown over here these are my receipt local lattice points interact.

So therefore you see that for this particular point where in this point as well as say something like this point is lying on the you are is lying on the Ewald sphere diffraction occurs, having said that you can now imagine that by varying either the reciprocal lattice or the Ewald sphere we can get the fraction in a material or in the crystal of our interest.

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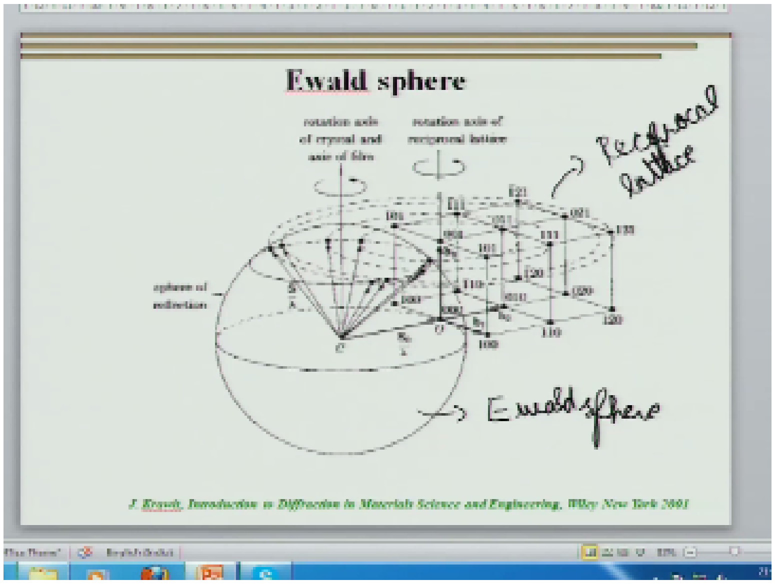

Ewald sphere

Reciprocal lattice

Ewald sphere

J. Krawitz, Introduction to Diffraction in Materials Science and Engineering, Wiley New York 2001

This is what has been shown in this slide and herein I have shown a 3d representation instead of a normal 2d representation and here we can see how exactly the Ewald sphere looks, so this is my Ewald sphere avoid sphere and this is how my this is my reciprocal lattice, so different x-ray diffraction techniques use different ways of either changing the Ewald sphere or the reciprocal lattice but in terms of crystal rotation. So that they come into coincidence and we can get diffraction.

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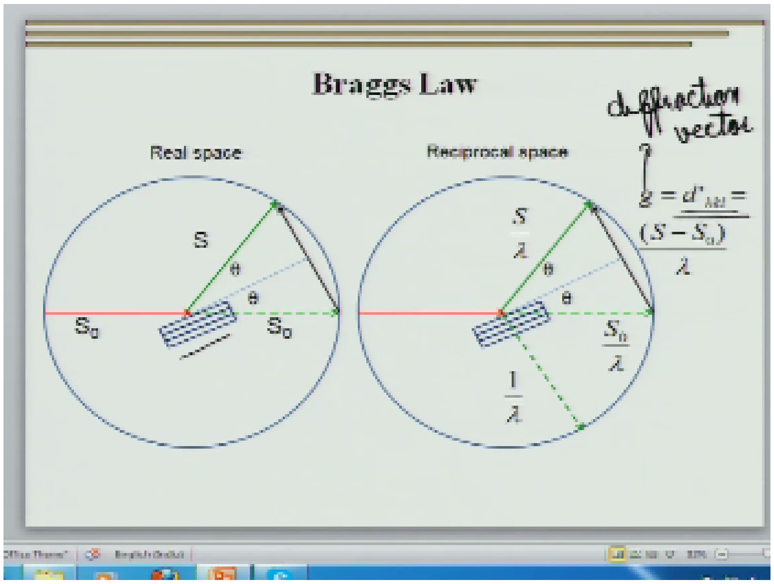

Braggs Law

diffraction vector

$g = d^*_{hkl} = \frac{(S - S_0)}{\lambda}$

So this is how I have shown Bragg's law in the real space on the left hand side the rain you can clearly see the s0 windy day which is incident on the crystal over here on the crystal which gets diffracted in the real space while in the reciprocal space the same thing instead of having talking about the lens we talked about the reciprocal lens and therefore we have this one over λ term coming over here and here we see how the incident wave is nothing but a zero by λ while the diffracted one is a s / λ.

And this leads to a path difference which is nothing but which is which is equal to the interlinear spacing which is shown over here, and this leads to the diffraction vector g. And therefore it can be defined as G = D' HDL which is given by s - s 0 / λ so this is the necessary condition for diffraction to occur in terms of in three dimensions.

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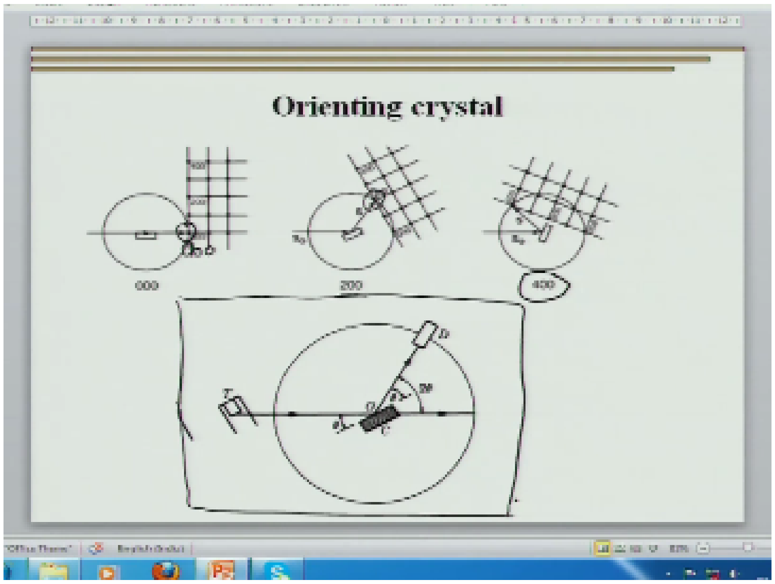

Orienting crystal

As I had already mentioned the entire exercise now evolves about how to orient the crystals so that at least one of the point lays one or more point lies on de vials clear so that diffraction occurs. So here in you can see that the 100 point lies and the 0 the 0 0 point lies and therefore you do not get diffraction however once you tilt the sample you do have 200 point of the reciprocal space which is lying on the Ewald sphere and we do get diffraction similarly the crystal can be oriented so that we do get diffraction from the 400 reflection.

So here in we can I hope you can appreciate that by changing the orientation of the crystal we can change the orientation of the reciprocal lattice that is associated with it and that diffraction I have shown a simple normal θ 2 geometry which is routinely used in our x-ray labs for analyzing poly line samples which comprised of multiple grains but I will cover the basics of rather the instrumentation part of it at a later stage.

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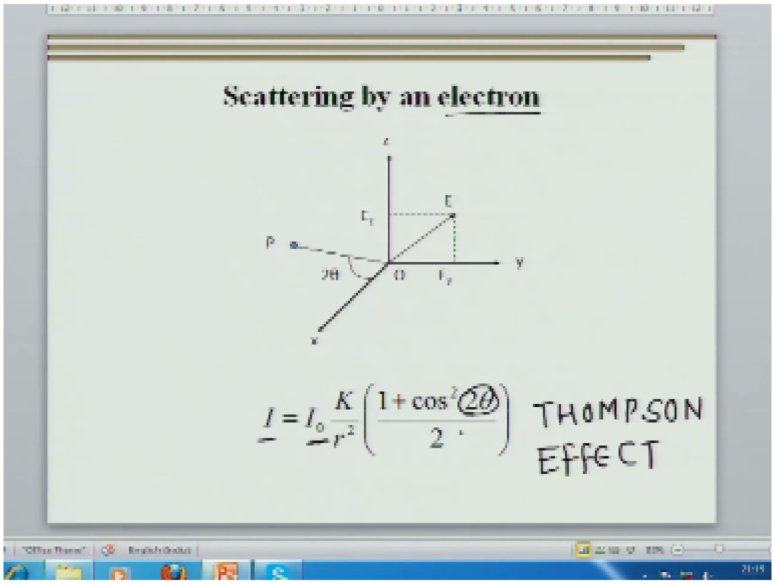

Scattering by an electron

$$\underline{I} = \underline{I_0}\frac{K}{r^2}\left(\frac{1+\cos^2 \boxed{2\theta}}{2}\right)$$

THOMPSON EFFECT

So before we go into the instrumentation part I will just like to go back and talk about you know we talked about a lot about how do waves interact and all those things, but that is in a very layman's language actually diffraction is also the weight builds up is essentially with interaction of the wave with the electron or essentially the scattering of the x-rays from an electron by what is known as Thompson effect what is known as the Thompson effect which essentially tells us how the intensity of the x-rays very when it is when it interacts with an electron at an angle λ.

So we can correlate that the intensity of the x-rays on interaction with electrons changes as a function of θ.

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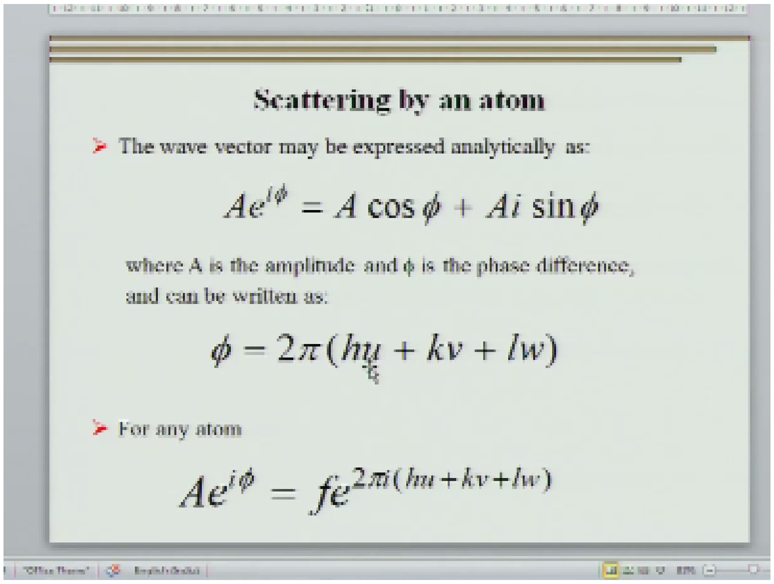

Scattering by an atom

- The wave vector may be expressed analytically as:

$$Ae^{i\phi} = A\cos\phi + Ai\sin\phi$$

where A is the amplitude and ϕ is the phase difference, and can be written as:

$$\phi = 2\pi(hu + kv + lw)$$

- For any atom

$$Ae^{i\phi} = fe^{2\pi i(hu+kv+lw)}$$

If you talk about an atom we know that an atom is an N symbol of electrons and it is directly from and the scattering by an atom is directly proportional to the atomic number however this is true only when there is direct incidence and as the incidence angle changes there is a decrease in the scattering atomic scattering factor of X series.

However we know that it can be expressed in terms of Ae vector which may be expressed analytically as AE $^{i\pi}$ which is equal to a cos 5 + AI φ π and π is nothing but the amplitude sorry the π is nothing but the phase difference and can be defined as 2 π x HQ + kv + LW for any atom AE I ^{5} is nothing but f of e 2 π h 0 + kv + l w but this is true as you must be aware as soon as i talk about these locations hu k v and LW this is true for an atom not single atom which is not isolated but is in a unit cell.

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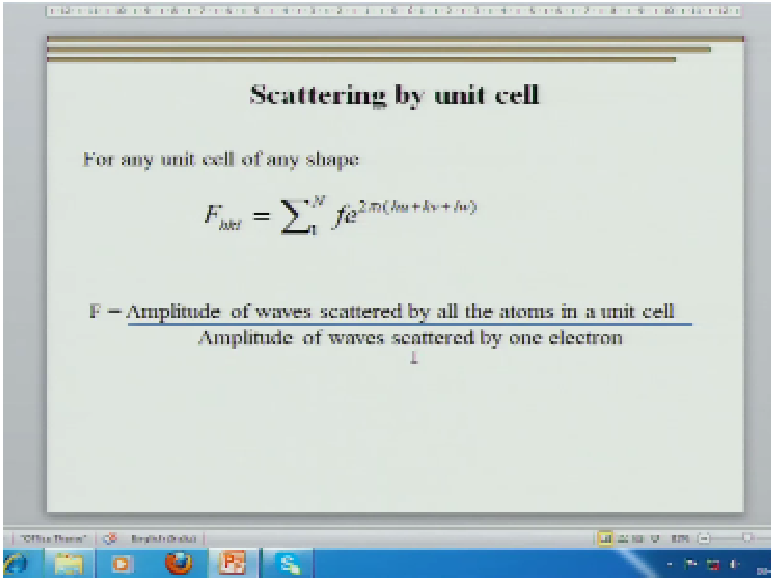

Scattering by unit cell

For any unit cell of any shape

$$F_{hkl} = \sum_{1}^{N} f e^{2\pi i(hu+kv+lw)}$$

$$F = \frac{\text{Amplitude of waves scattered by all the atoms in a unit cell}}{\text{Amplitude of waves scattered by one electron}}$$

Therefore if you look at the scattering by unit cell which comprises of more than one atom as in a FCC unit cell or a bcc unit cell the simplest example that I had mentioned earlier was the simple cubic case wherein there is only one atom per unit cell, however when we go to bcc or FCC unit cells with the two atoms and four atoms per unit cell the scattering by unit cell is given by summation of all the atoms in the unit cell and can be given as f of HK l is equal to summation over all summation 1 over n small f eh to 2 pi h u + kv + l w + f the capital f the amplitude is nothing but the amplitude of waves scattered by all the atoms in the unit cell / amplitude of waves scattered by one electron.

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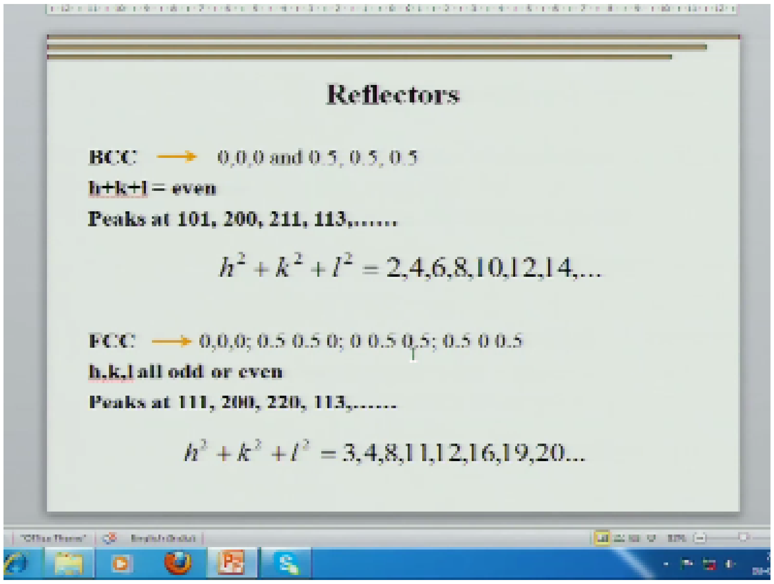

Reflectors

BCC → 0,0,0 and 0.5, 0.5, 0.5

h+k+l = even

Peaks at 101, 200, 211, 113,......

$$h^2 + k^2 + l^2 = 2,4,6,8,10,12,14,...$$

FCC → 0,0,0; 0.5 0.5 0; 0 0.5 0.5; 0.5 0 0.5

h,k,l all odd or even

Peaks at 111, 200, 220, 113,......

$$h^2 + k^2 + l^2 = 3,4,8,11,12,16,19,20...$$

And therefore we come to the most important part of diffraction as in what all are the different reflectors that are possible with x-ray diffraction for different crystal structures, so if we look at simple cubic crystal structure we see that all the reflections are possible. So that $H^2 + K^2 + L^2$ is equal to 1 2 3 up to 6 and then eight nine ten we do not get seven because knows some of three squares never add up to seven however when we come to bcc we have two atoms per unit cell which are located at 0 0 0 and 0 point five point five point five right at the center of the queue.

Therefore we do get a condition that diffraction occurs only when $H^2 + K^{2+} n^2$ is even that is 2 4 6 8 10 or 12, and therefore put in other words it essentially means that diffraction occurs only for peaks wearing h + k + L is even and we do get diffraction for 10 12 0 0 and 2 11 peaks however when you go to FCC which has got four atoms per unit cell at 0 0 0 0 point 5.50 0.5 0.5 0.5 0.5 we do get diffraction only if hkl are all odd or all even and therefore we get diffraction at peaks of 111 200 220 and 113 therefore the condition for diffraction can be summarized as $h^2 + K^2 + N^2$ is 3 for 8 11 and so on, so I think I am done with it.

(Refer Slide Time: 37:39)


Thank
You

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