Texture in Materials
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Module - 05
Principles of texture measurements by X-ray diffraction
Lecture - 24
Characteristic X-ray, Absorption and Filters

Good afternoon everyone and today, we will continue with module 5 that is Principles of texture measurement by X-ray diffraction and today is lecture number 24, where we will try to understand Characteristics X-rays, Absorptions and Filters for the these characteristics X-rays.

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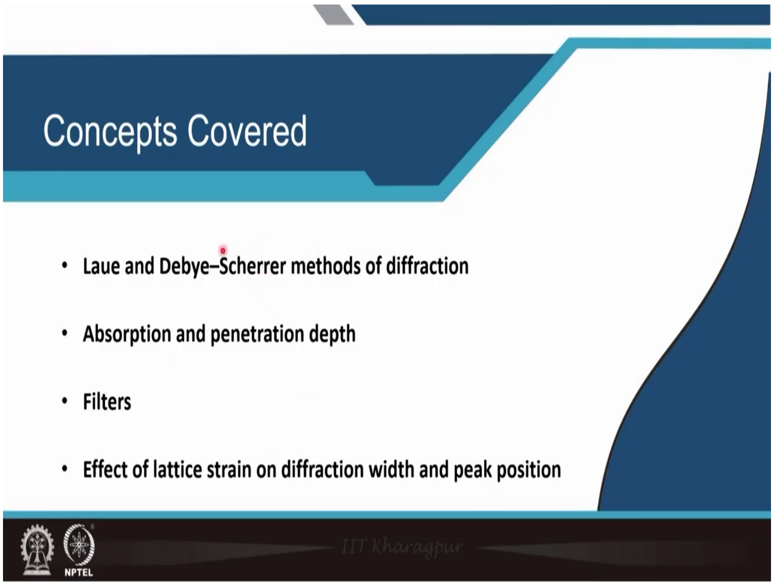

Concepts Covered

- Laue and Debye–Scherrer methods of diffraction
- Absorption and penetration depth
- Filters
- Effect of lattice strain on diffraction width and peak position

the concepts that will be covered in this lecture will be Laue method of diffraction, Debye-Scherrer method of diffraction, absorption and penetration depth, filters that are required to get monochromatic X-rays, effect of lattice strain on diffraction width and the positions of the peak.

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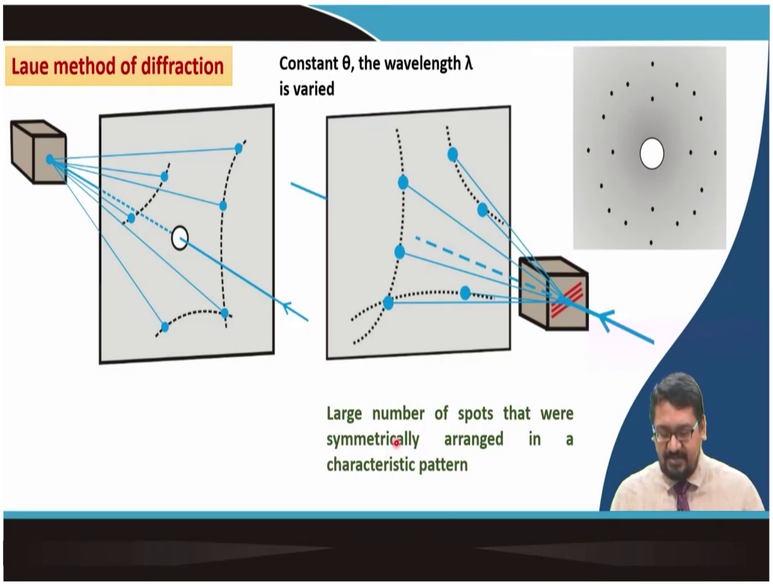

Laue method of diffraction

Constant θ, the wavelength λ is varied

Large number of spots that were symmetrically arranged in a characteristic pattern

let us start what is Laue method of diffraction. You see that when the X-rays, were initially invented and then, there were you know continuous X-rays and then, with the further invention the monochromatic X-rays were found out. Now, the point is that during that time when the X-ray is just came into existence, known to the mankind.

Then you see Laue used a method, where he used an x-ray to diffract you know single crystal and this diffraction was done in such a way that the wavelength of the x-ray is varied and that is why it is not monochromatic, it is a continuous X-rays. And the theta is kept constant and in this way, you see both in the reflection mode and in the you know transmission mode the hkl planes, different hkl planes that could diffract at the same theta angle.

Because the lambda of the wavelength of the X-rays are varying and it may at some theta will support the Braggs law and therefore, such kind of pattern, a pattern schematic of a pattern of a single crystal of a certain single crystal is shown here. you see in this manner a large number of spots that is that are means symmetrically arranged in a characteristic pattern could be observed and this is known as the Laue method of diffraction.

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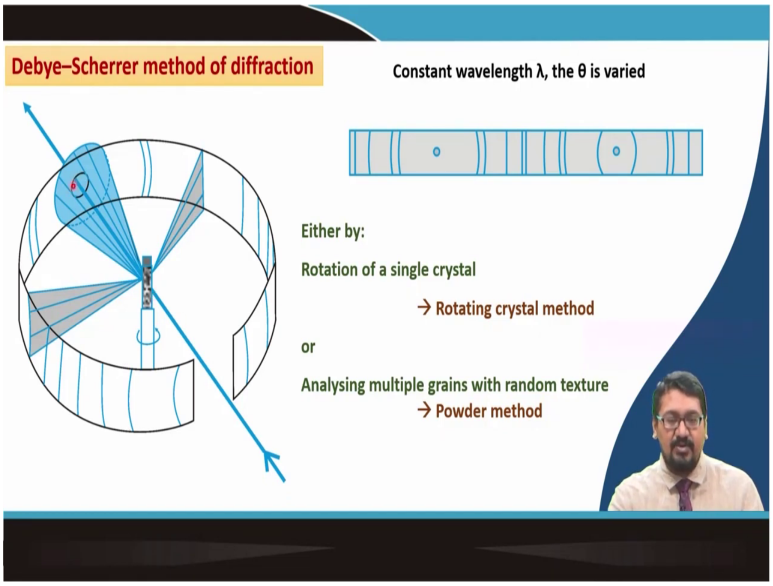

Debye–Scherrer method of diffraction

Constant wavelength λ, the θ is varied

Either by:

Rotation of a single crystal

→ Rotating crystal method

or

Analysing multiple grains with random texture

→ Powder method

Later, Debye-Scherrer you know modified this method of diffraction and used characteristic X-rays that is monochromatic X-rays to diffract different hkl planes at different Bragg's angle theta right.

you see here is the schematic of the construction of this kind of an experiment, where a small sample may be a single crystal or a powdered poly crystal is placed at the center. It is surrounded by you know the detector of phosphor screen which is rounded down to you know capture the intensities observed at various Bragg angles. You see either it is you know rotation of a single crystal and then, it is called the single crystal rotation method or it is an analysis of multiple grains using random texture; how we can obtain that? By making a powdered, a keeping a powdered sample with lots and lots of you know powdered particle having you know multiple grains in even in a single powder. This is known as powder diffraction method. What happens? If you look into this schematic, there is this phosphor screen and the X-ray beams falls on the sample, where the sample is being rotated. You see that when the x-ray beam falls on the sample, various hkl planes they will diffract at various theta angles which is basically the angle theta defined by the Bragg's law that is l lambda equal to 2 d sin theta right.

You can see that various diffraction, you know Debye-Scherrer lines or Debye-Scherrer curves could be obtained right. If we straighten this strip, we can see that this strip will will look something like this, where the direct X-ray beam also passes through and there is a hole, a small hole here, where it passes right.

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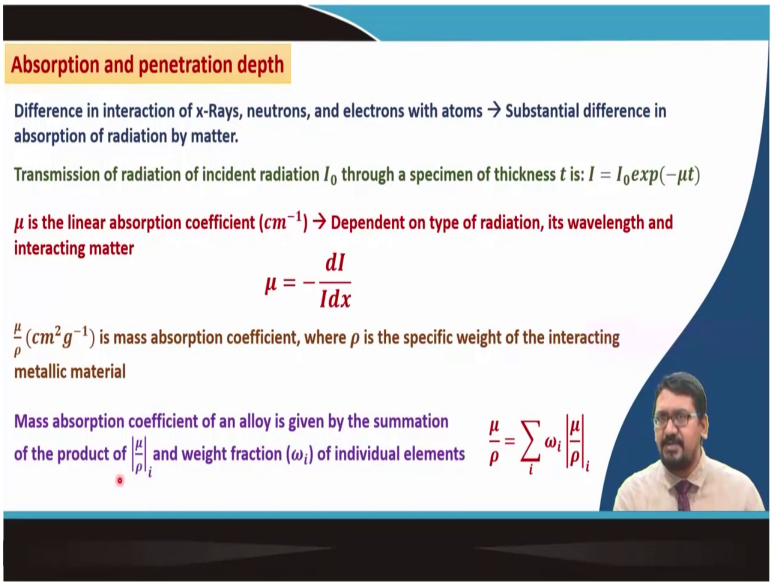

Absorption and penetration depth

Difference in interaction of x-Rays, neutrons, and electrons with atoms → Substantial difference in absorption of radiation by matter.

Transmission of radiation of incident radiation I_0 through a specimen of thickness t is: $I = I_0 exp(-\mu t)$

μ is the linear absorption coefficient (cm^{-1}) → Dependent on type of radiation, its wavelength and interacting matter

$$\mu = -\frac{dI}{Idx}$$

$\frac{\mu}{\rho}$ ($cm^2 g^{-1}$) is mass absorption coefficient, where ρ is the specific weight of the interacting metallic material

Mass absorption coefficient of an alloy is given by the summation of the product of $\left|\frac{\mu}{\rho}\right|_i$ and weight fraction (ω_i) of individual elements

$$\frac{\mu}{\rho} = \sum_i \omega_i \left|\frac{\mu}{\rho}\right|_i$$

you see in the x-ray diffraction, absorption and penetration depth is a very important factor. It decides whether the radiation not only the x-rays, the radiations that is you know x-Rays, neutrons, electrons whether they are you know how much they are interacting and how deep it is going into that matter. Because of the nature of the interaction of the x-rays, neutrons and electrons, there is a substantial difference between the absorption of the radiation of these radiations with the matter.

You see if a radiation of an intensity I 0 is incidented on the surface of a particular polycrystalline metal, then the intensity I, if it you know of a certain thickness say the thickness of that particular specimen or sample is t and then, the intensity I comes out. Then the relationship between the final intensity I and the you know incident intensity I 0 is I equal to I 0 exponential minus mu t, where mu is the linear absorption coefficient and t is the thickness of the sample.

Now, you see the linear absorption coefficient has an unit of per centimeter and depending upon the type of the radiation, its wavelength and its way of interaction with the matter. This linear absorption coefficient varies and this linear absorption coefficient can be given by mu equal to minus of differentiation of the intensity by the intensity the delta x. delta I by delta x and by the intensity. this is just the you know different form of showing the same equation, where we can find out the linear absorption coefficient.

Now, you see usually the linear absorption coefficient is not shown in terms of mu rather it is shown in terms of mu by rho, where mu by rho is the mass absorption coefficient. Here it is and is given by the unit centimeter square per gram. Now, where rho is the specific weight of the interacting material right.

Now, you see that if that interacting material is not a single polycrystalline metal, but a mixture of one or two metals that is it is kind of an alloy. Then you see then the summation of the you know the mass absorption coefficient mu by pi and the weight fraction of that particular individual element present in that alloy is used to calculate the total mass absorption coefficient for that particular you know polycrystalline material, which is not a single metal, which is not made up of single element; but multiple elements right that is an alloy.

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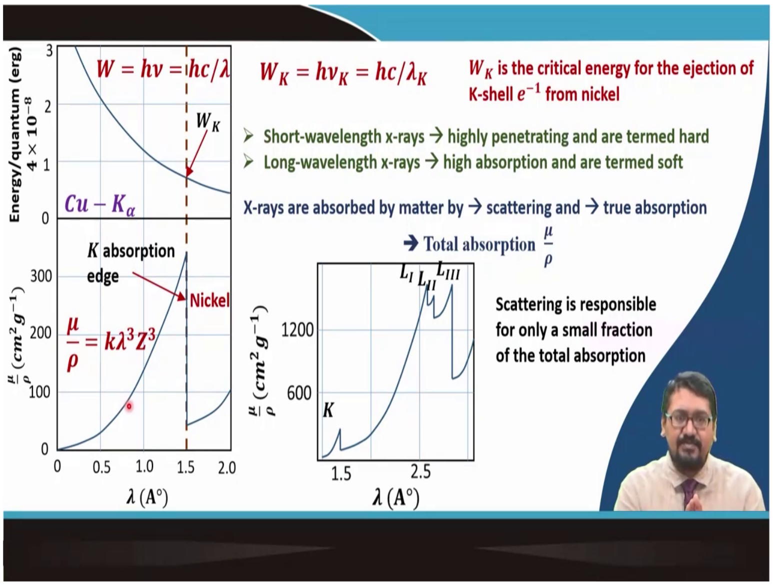

$W_K = h\nu_K = hc/\lambda_K$

W_K is the critical energy for the ejection of K-shell e^{-1} from nickel

- Short-wavelength x-rays → highly penetrating and are termed hard
- Long-wavelength x-rays → high absorption and are termed soft

X-rays are absorbed by matter by → scattering and → true absorption

→ Total absorption $\frac{\mu}{\rho}$

Scattering is responsible for only a small fraction of the total absorption

Now, if we look into mass absorption coefficients relationship with wavelength. You see that let us take a copper you know radiation. Let us take a copper radiation ok. this radiation say that it is coming, it is a continuous radiation coming from you know copper anode and now, you see that if we plot the lambda which is in the x-axis versus the energy per unit quantum, then you will find out that as the lambda is decreasing the energy of this radiation is increasing.

Because you see that the relationship of energy that is, W is equal to h mu right; h is the Planck's constant and mu is the frequency and is equal to hc by lambda. you see that as the energy of the incident radiation is increasing, the lambda is decreasing. Now, if you look closely, we can see that the if this you know continuous radiation coming from copper is incidenting on say for example, a nickel which has a little lower you know an atomic number lower; nickel has an atomic number of 29; whereas, copper I think is 29.

If we look into this curve, this is the curve which is between you know the you know x-axis is lambda and the y-axis is the mass absorption coefficient that is mu by rho and you see that as the incident X-ray radiations you know energy is increasing that is the lambda is decreasing, the mass absorption is decreased right. the penetration depth is increased. However, if we look at this whole curve, the if we look here, there is another curve which has the same trend that as the wavelength is decreasing from point from 2 to 1.5, the mass absorption coefficient is decreasing. But at a certain you know energy of this incident X-ray, there is a large peak and this peak corresponding to you know the energy W K is seen and this, this this peak is said to be you know the K absorption edge or the K excitation voltage, K excitation energy.

With respect to this, you know formulation that the energy is equal to h mu is equal to hc by lambda, one can say that for this particular you know K absorption edge or K excitation energy. We can find out W K, the energy for that is equal to h times mu K is equal to hc by lambda K. At this particular lambda, when the energy is increased and the lambda is decreasing slowly and slowly and it is coming to this lambda, the energy becomes so high that this at this critical energy, the K-shell electron from the nickel could be ejected. an energy of such level W K level, when it incidents on the nickel, it is capable enough to eject a K-shell electron from nickel and when it is done, the nickel goes to a very high energy state right. When the L-shell atom come of the nickel goes to the K-shell atom of the nickel to you know reduce its energy to come to the ground state. Then, another characteristic wave X-rays of characteristic radiation of a certain wavelength is released also from nickel, we will talk about that later. However, you see that even though as the X-ray wavelength is shortened, reduced the penetration depth reduces because the mass absorption coefficient is reducing.

But, at certain critical excitation energy or critical excitation voltage, what happens that it is enough to eject a K-shell electron from nickel and this we are talking about if the radiation is from say for example, a copper and it is falling on nickel and then, it is able to absorb energy so that the K-shell electron is you know excited and goes away from the atom and then, that excited atom that is known as the K absorption edge right.

You see that X-rays are absorbed by matter by you know this scattering and true absorption and you see that the total absorption including scattering plus true absorption is equal to mu by rho. Now, scattering is though responsible for a small fraction of this absorption, but most of the total absorption is basically based upon the true absorption.

Now, here, you see we will show we are showing another you know schematic and you see what we are showing here is a schematic, where the lambda that we have shown up to 2.0, we are extending it to a higher lambda and you see the x-axis is basically lambda and the y is again mass absorption coefficient. What we mean to say here is that when the K excitation you know energy is incidenting a radiation of sufficient energy which is equivalent to that critical K absorption edge or K excitation energy is incidenting on the on another material say nickel in this case as an example. Then, the K absorption edge is always there which shows a higher you know mu by rho at a certain wavelength, but also there will be the L absorptions all together.

Because the L absorption that occurs because of the you know the critical energy that is required to eject an L-shell electron and there are three L levels; L I, L II and L III say, then there could be three you know peaks of mass absorption coefficient. you see that while we get a K absorption edge, it will not be divided of L absorption edge because to obtain L absorption edge, the excitation energy required by the incident radiation is quite low. K absorption edge is always it includes the L absorption edge also. it happens all together.

Now, very importantly one very important thing you see that while the wavelength is reduced from for this curve and you see that there is a high absorption coefficient here and when the lambda is reduced, the energy W is basically increasing more and more than the W K right and thereby, it can always cause the K excitation and therefore, there will be always a K absorption edge. But you see that the absorption is reducing as the lambda is in decreasing; sorry the absorption is yes reducing as the lambda is decreasing and therefore, the penetration depth is basically increasing.

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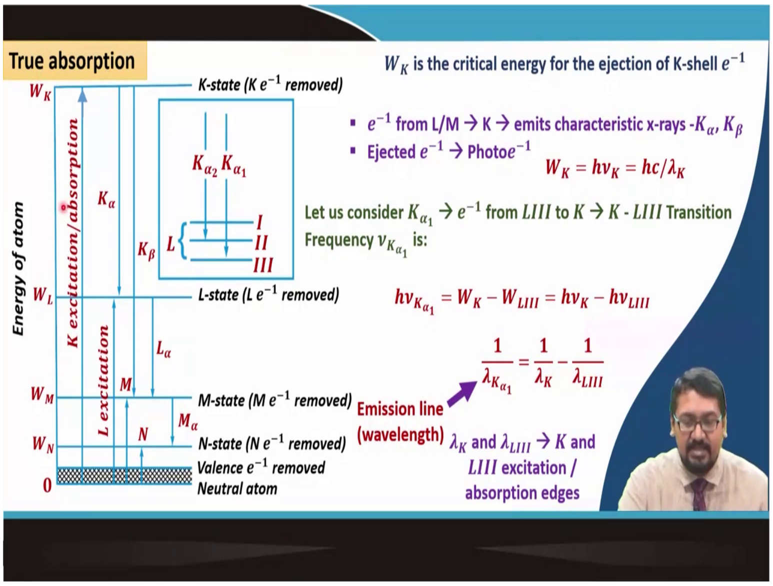

True absorption

W_K is the critical energy for the ejection of K-shell e^{-1}

- e^{-1} from L/M → K → emits characteristic x-rays -K_α, K_β
- Ejected e^{-1} → Photoe^{-1}

$$W_K = h\nu_K = hc/\lambda_K$$

Let us consider $K_{\alpha_1} \rightarrow e^{-1}$ from $LIII$ to $K \rightarrow K$ - $LIII$ Transition

Frequency $\nu_{K_{\alpha_1}}$ is:

$$h\nu_{K_{\alpha_1}} = W_K - W_{LIII} = h\nu_K - h\nu_{LIII}$$

$$\frac{1}{\lambda_{K_{\alpha_1}}} = \frac{1}{\lambda_K} - \frac{1}{\lambda_{LIII}}$$

Emission line (wavelength)

λ_K and λ_{LIII} → K and $LIII$ excitation / absorption edges

Now, you see that what we are talking about regarding this you know true absorption. What we mean to say is that say look into this you know schematic and you see that if 0 is the position of the energy for the neutral atom and you see that this is basically the energy for the metals which are present in a polycrystalline material.

Because you see that the metals in a polycrystalline in a polycrystalline metal the atoms are you know are present in the C of electron right. the valence electron is basically removed from the atom and then it is there. it is slightly in a higher excitation state and then, you see that whenever the you know energy of a sufficient level that is easy W K is enough to eject a K-shell electron, the energy say for this is this much from 0 to here.

this much energy if it is given, then it is able to eject a you know a K-shell electron and then, you see that when this energy, when the atom is in such energy state that is the K state, where the K electron is removed and then, the electron from the L-shell goes to the K cell right and it is shown like this.

there is a reduction of energy from W K to W L or the electron from the M-shell goes to the K-shell that is the reduction of energy from here to here right. this is the you see this is the N state, M N electron removed; M state, the M electron removed; the L state, the L electron removed.

If we look into this schematic properly, we can say that in order to remove the K-shell electron, we need an energy W K. In order to you know remove the L-shell, we need an energy W L. To remove M-shell, we need just W M and then, W N for the N-shell.

Now, the point is that you see that when the W K is given the K-shell electron is removed and the probability that M-shell atoms sorry M-shell electron will fill the K-shell is lower than the probability that the L-shell electron will fill the K-shell. Therefore, the K alpha is usually have a very high intensity as compared to the K beta and K beta is from electron from M to K and K alpha is the electron from L to K right.

Now, you see L has three you know sub-shells and that is L 1, L 2 and L 3 and it is shown here and you see when K falls from K to L 3, it is K alpha 1 and when it falls to K, L 2, it falls from L 2, it is K alpha 2 right. Now, what happens here is that when these you know as I said that electrons from L produces K alpha electrons, from M produces K beta and these are the characteristics wavelength and this you know wavelength will have a characteristic you know particular wavelength a particular wavelength.

You see that when W K is given; W K is the excitation energy which is equal to h mu K which is equal to hc by lambda K. this particular critical excitation energy or critical excitation lambda K, lower lambda K as we are lowering it, it allows the electron from the K-shell to be removed. Let us say electron from the L 3 is reaching the K-shell and then, it is emitting a characteristic radiation and this characteristic radiation as I was saying in the last slide althis characteristic radiation say it has a frequency mu K alpha 1 or 1 by lambda K alpha 1 means basically lambda K alpha 1 is this wavelength right.

Using this equation, you see the energy of that radiation is h times mu K alpha 1 and then, this is basically the energy corresponds to this K a and this K a is basically corresponding to W K minus W L 3 right. W K minus W L 3 is equal to h mu K from this minus h mu L 3 right. If we get a relationship between the X-rays emitted from that particular element which is radiated say copper X-ray is used to radiate nickel as I said earlier and then, the radiated wavelength of this characteristic radiation has a wavelength of lambda K alpha 1 right and which is equal to 1 by lambda k minus 1 by lambda 3. this is basically the you know absorption edge or the emission line wavelength right. lambda K and lambda 3 is the K and the L 3 excitation or absorption edge. thus, this is by the way the absorption in a metal takes place absorption of X-rays.

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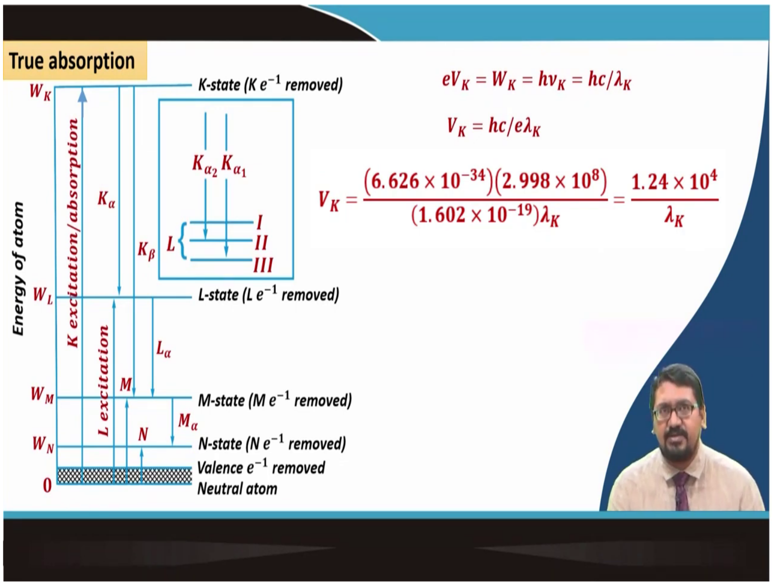

True absorption

$$eV_K = W_K = h\nu_K = hc/\lambda_K$$

$$V_K = hc/e\lambda_K$$

$$V_K = \frac{(6.626 \times 10^{-34})(2.998 \times 10^{8})}{(1.602 \times 10^{-19})\lambda_K} = \frac{1.24 \times 10^{4}}{\lambda_K}$$

if we look into this you know energy that is W K the x K excitation energy, this is equal to you see the in terms of electron volt eV right. the critical excitation energy which is equal to h mu K that is equal to hc by lambda K is the energy of the X-ray beam which is generated because of the you know the in the X-ray tube, the electrons from the cathode hits the anode and in this case the anode is the copper anode right.

And as the energy of this electrons are increasing, the voltage means we have to increase the voltage to increase the you know energy of the electron so that the electrons that is hitting the anode can produce more and more intensified you know X-rays. W K is related to electron volt right; electron into voltage is the energy of the electron beam, which is converted into copper K alpha radiation and the other continuous radiation. as the W K is basically related to V K. if we find out the voltage of that in the of the X-ray tube, V K is equal to hc by e which is the electronic charge lambda K right.

V K is equal to h, which is 6.626 into 10 to the power minus 34 Planck's constant; c is the speed of the light 2.998 into 10 to the minus 34 divided by the charge of the electron, which is 1.602 into 10 to the power minus 19 times lambda K. It comes up to be 1.24 into 10 to the power 4 by lambda K. the voltage required to produce a certain K excitation, you know wavelength can be calculated by this method.

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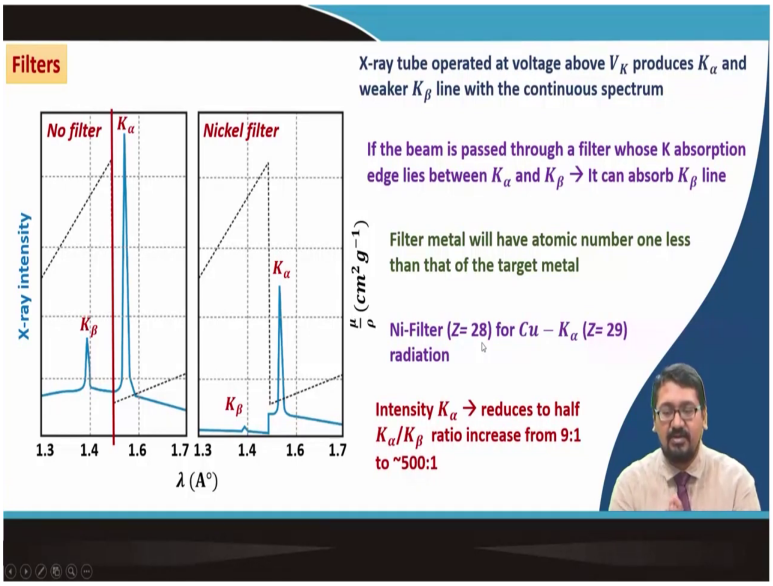

Filters

X-ray tube operated at voltage above V_K produces K_α and weaker K_β line with the continuous spectrum

If the beam is passed through a filter whose K absorption edge lies between K_α and K_β → It can absorb K_β line

Filter metal will have atomic number one less than that of the target metal

Ni-Filter (Z= 28) for $Cu - K_\alpha$ (Z= 29) radiation

Intensity K_α → reduces to half
K_α/K_β ratio increase from 9:1 to ~500:1

this method of mass absorption of X-ray radiation can be used to filter the continuous X-ray to obtain monochromatic radiation because monochromatic radiation can be used for diffraction experiments to obtain information in a much more detailed manner. you see that if a X-ray tube is operating at certain voltage V K and it is producing K alpha and K beta radiation and it is also producing definitely the continuous spectrum.

But as I said earlier, in the earlier classes that as the voltage of the X-ray tube containing cathode and anode is increased, the electrons from the cathode reaches the anode and the you know the intensity of the continuous radiation keeps on increasing as the voltage is increased right. And at certain voltage K alpha and K beta radiation starts to fall and as the voltage of that X-ray tube is increasing the lambda SWL that is short wavelength limit is also decreasing, the intensity of K alpha and K beta is also keeping on increasing. But it is important to know that to do you know diffraction experiments only a single monochromatic radiation that is K alpha is required which has the highest intensity; whereas, the K beta has to be removed.

Now, if the beam containing this K alpha, K beta and other continuous radiation is passed through a filter, the filter is a metal which should have a K absorption edge which lies between the K alpha and K beta. It can absorb the K beta line and excites its K-shell means the K beta line that this radiation can give enough energy to that filter metal. So that its K electron is ejected out and the if any electron from the L or M-shell is falling on this K-shell atom of this filter metal, then that characteristic radiation is dispersed everywhere and it will not go to the detector. It will absorb the K beta as well as a part of you know continuous radiation. Therefore, you see if copper you know X-ray tube is used to produce the K alpha of copper, then the nickel which has an atomic number lower than copper can be used to filter out the K beta and this helps the material to this helps us to obtain you know characteristic K alpha radiation.

Now, the intensity of K alpha to K beta is of the range of 500 is to 1, after the filtration takes place and it reduces and before the filtration; it is in a range of 9 is to 1 and you see that nickel which is used as a filter has an atomic number of 28, one lower than the copper which has an atomic number of 29.

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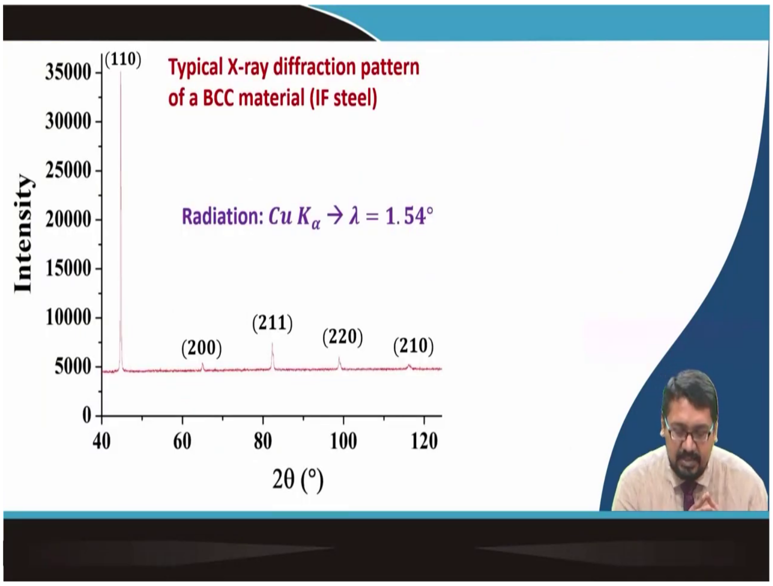

Typical X-ray diffraction pattern of a BCC material (IF steel)

Radiation: $Cu\ K_\alpha \rightarrow \lambda = 1.54°$

Using this technique, one can obtain you see characteristics copper alpha radiation and this copper alpha radiation of characteristics lambda equal to 1.54 can be used to obtain diffraction pattern from various you know metals and alloys and you see here is an example of the diffraction pattern obtained from this kind of copper alpha radiation and this you can see that we have obtained a peak at 110, another peak at 200, another at 211, 220, 210 and this this peaks shows that this is a you know BCC material and thus, this shows the importance of characteristics radiations.

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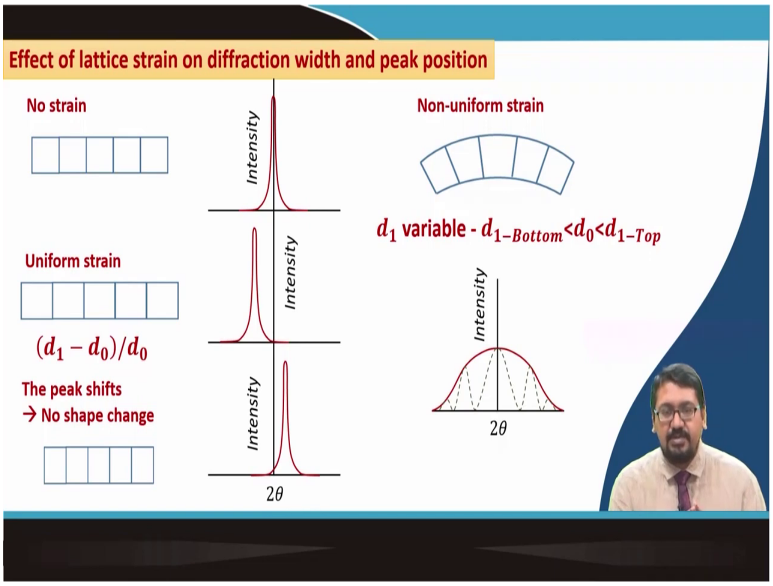

Effect of lattice strain on diffraction width and peak position

No strain

Uniform strain

$(d_1 - d_0)/d_0$

The peak shifts

→ No shape change

Non-uniform strain

d_1 variable - $d_{1-Bottom}<d_0<d_{1-Top}$

Now, you see that whenever we are doing diffraction and at a particular theta a peak is coming and that theta that theta angle, the Braggs angle theta is represented of representation of that particular hkl plane right. You see when the metal has no strain at all and it is in the nascent state and then, that the d that is the inter lamellar distance between the distance between the plane d is not is equal to what? It should have, it should come for that particular theta right. n lambda equal to two d sin theta. this d and theta is basically known that ok for a particular material and if the you know the is I am going to the previous slide and if the material is diffracting from a certain you know hkl plane say 110 plane, the inter planar spacing between this is already known.

The theta is already decided right and this is decided for you know 110, for 200 and 211 and it is fixed at certain theta. But you see that when the material is under some kind of a stressed condition, say if there is a uniform stress in such a way that this d, the inter lamellar spacing for that particular plane is increased.

Then, what happens that n lambda equal to 2 d sin theta equation if we use, that is the Braggs law. When you use it, we will find out that theta that is sin theta becomes equal to n lambda by 2 d right. As d is increasing, the theta has to decrease. you see that when there is no strain, the intensity of the theta is obtained at the exact theta peak as per the Braggs law for that particular hkl plane.

However, if it is under uniform strain that is if d is higher, then the theta decreases and if the you know the peak shifts towards the lower, you know 2 theta value right. As if it is trained in such a way that the d is you know the inter lamellar spacing is decreased, then the theta basically increases. the peak shifts at a higher 2 theta You.

This peak shift as I said depends upon the as the Braggs law has to be followed and the d spacing alters when there is a uniform strain. So there is a peak shaped either below that particular Braggs angle theta be for that particular hkl plane or increases depending upon whether the uniform strain is stretching the distance between the plane or it is compacting it. And in case of non-uniform strain, the strain is such a way that the hkl plane in one position, they are compressed and the d is reduced and in other place it is basically the d is increased.

If you look into this figure, we see that the d is a variable right. d 1 at the bottom is less than the d 1 at the top and it is basically in the bottom is less than the actual d 0 without strain. what happens that for the where the d is increased that is in the top, the peak shift occurs towards the negative direction, towards the lower side and you see what happens that the peak is shifting this side then right and when the d is decreased in the bottom, the peak is shifting towards this side right. Let me start with the laser pointer. When the d is decreased, the peak shifts towards this direction and if the d is increased, then the peak shifts towards this direction and leading to you know thickening of this peak and it becomes something like that. it is also important to understand that how d spacing and the changes in the d spacing may occur in the material under some kind of processing conditions which leads to change or shifts in the peak or you know increase the in you know the volume of the peak spreading of the peak that may occur.

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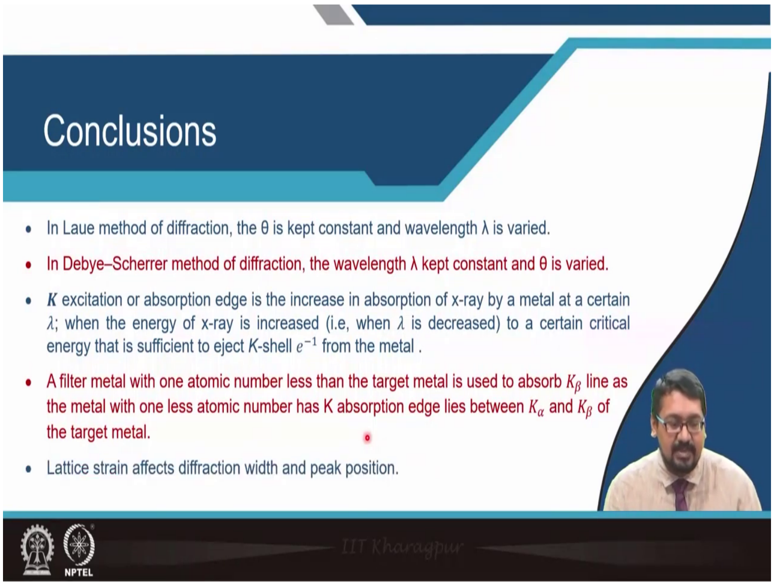

Conclusions

- In Laue method of diffraction, the θ is kept constant and wavelength λ is varied.
- In Debye–Scherrer method of diffraction, the wavelength λ kept constant and θ is varied.
- *K* excitation or absorption edge is the increase in absorption of x-ray by a metal at a certain λ; when the energy of x-ray is increased (i.e, when λ is decreased) to a certain critical energy that is sufficient to eject *K*-shell e^{-1} from the metal .
- A filter metal with one atomic number less than the target metal is used to absorb K_β line as the metal with one less atomic number has K absorption edge lies between K_α and K_β of the target metal.
- Lattice strain affects diffraction width and peak position.

What we understood from this today's lecture? We understood that the there are method which is called the Laue method of diffraction, where the experiment is done in such a way a continuous X-ray with variable wavelength is taken and the theta is kept constant to obtain diffraction from various hkl planes. On the other hand, with the advent of characteristic radiations and with the increase in the you know use of the filters like nickel filter for copper. We use we can use Debye-Scherrer method of diffraction, where lambda can be kept constant; that means, characteristic wavelength K alpha radiation can be obtained and the theta is varied to obtain you know various hkl diffraction information from the from various metals and alloys.

Now, importantly what you observe that K excitation or absorption edge is the increase in the absorption of x-ray biometal at a certain fixed lambda that is some critical lambda excitation edge lambda. Where the energy of the x-ray is increased and the lambda is decreased to a certain critical value so that it becomes sufficient to eject a K-shell electron from another metal from the metal. This you know mechanism can be used to make you know filter material to filter out K beta radiation from and some continuous radiation from the K alpha radiation so that a higher intensity of K alpha radiation could be obtained. a filter material is used is actually a one atomic number less than the target metal so that it can absorb the K beta line.

In case of copper, nickel is used as a filter. as the metal with one atomic number less has the K absorption edge in between the K alpha and K beta of the target metal. Thank you. That is ok; one more thing lattice strain affects diffraction width and peak position, we have seen that.

Thank you very much.