Techniques of Materials Characterization
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Lecture - 47
Intensity of Diffracted Beam

Welcome everyone to this NPTEL online certification course on techniques of materials characterization and we are now in module 10. We are discussing about x-ray diffraction and this is the second lecture. Until up to now about x-ray diffraction we have discussed in the first in 9th module basically that was the first module dedicated to x-ray diffraction. We discussed about the generation of x-ray.

How x-rays are generated? What exactly is characteristic radiation? What is continuous spectrum? And how the x-rays is generated when a through target when an electron beam is hitting the target metal? All of these aspects we have discussed so far. And in the last class, we were continuing that discussion a little further and we were discussing about the absorption x-ray absorption we discussed about the absorption edge and so on.

And now from this week onwards where this lecture onwards we will start discussing about the intensity of the diffracted beam.

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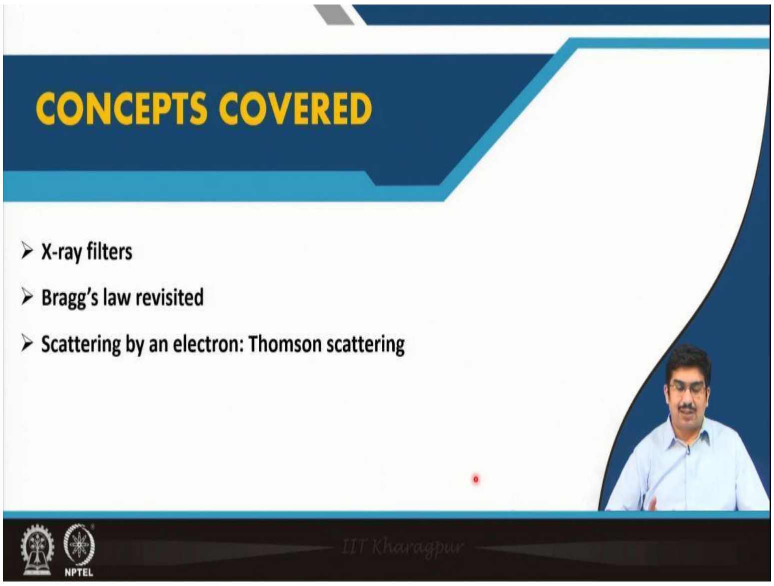

CONCEPTS COVERED

- X-ray filters
- Bragg's law revisited
- Scattering by an electron: Thomson scattering

And before that we will just left one small topic in the extra absorption and generation of extra instrumentation of an x-ray tube that is x-ray filters. So, we will just discuss about that briefly. And then we will just start our discussion about the intensity of x-ray beam intensity of the diffracted beam. And first we will discuss about the Bragg's law revisited from this intensity perspective.

We will discuss about why we need to separately discuss the intensity of x-ray diffraction even if we have the Braggs law. And then we will discuss about x-ray scattering by an electron. And there are two defects basically in x-ray scattering we will try to discuss about one in this class and the next one we will discuss another. So, that first effect we will discuss in this class is Thomson scattering.

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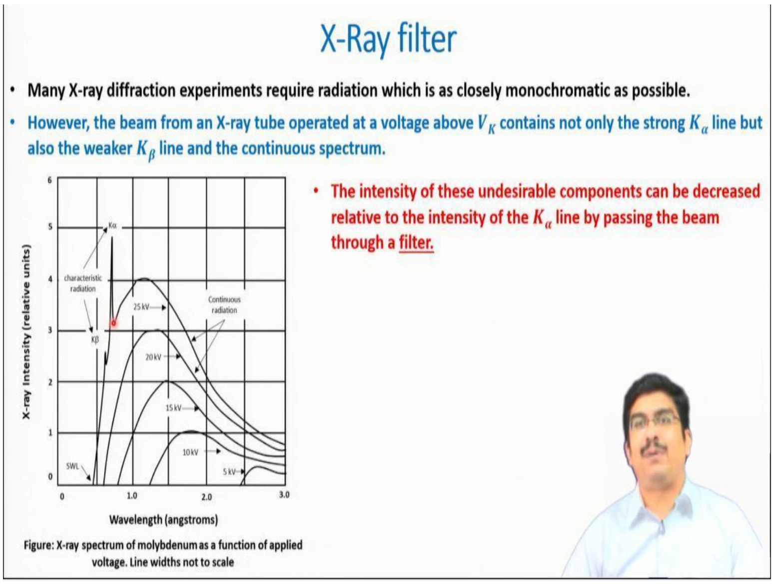

X-Ray filter

- Many X-ray diffraction experiments require radiation which is as closely monochromatic as possible.
- However, the beam from an X-ray tube operated at a voltage above V_K contains not only the strong K_α line but also the weaker K_β line and the continuous spectrum.
- The intensity of these undesirable components can be decreased relative to the intensity of the K_α line by passing the beam through a filter.

Figure: X-ray spectrum of molybdenum as a function of applied voltage. Line widths not to scale

So, x-ray filter I think we already discussed about the x-ray absorption, how the absorption happens? True absorption what happens in a true absorption? And then what is the fluorescence radiation and photoelectric generation? All of these things we discussed. So, we know basically what an absorption phenomenon is all about and towards the end we discussed that why we are looking for an absorption what is the main implication of a characteristic radiation.

And an absorption and there one thing was told that the diffraction experiments basically require an x-ray radiation which is as closely monochromatic as possible. Because what happens is if you and that is what the importance of having this characteristic spectrum over the continuous spectrum, because if you look at here this is the characteristic spectrum and this is the K alpha line.

And this is a K beta line and this K alpha line we discussed that it is very sharply defined the lambda value the wavelength at which this characteristic radiation occurs. It only depends on the atomic number and it does not depend on the voltage. Applied voltage whereas continuous spectrum; does depend on applied voltage short wavelength limit and then maximum in continuous spectrum.

All of these things depend on the wavelength of or the voltage of used to accelerate these electrons. But characteristic radiation is what their number one their wavelength is very well-defined a and b they are very narrow. So, the spread of this characteristic spectrum is also very narrow. So, we tend to use such monochromatic beam for any diffraction experiment because that also we discussed in diffraction experiment you basically need N lambda = 2 d sin theta.

So, there is a Bragg's law and there lambda we want it to be constant as constant as possible. So, that we can keep at least one of these constant so there are like four variables N lambda d and sin theta. So, out of that if we can use a monochromatic beam at least one of these variables we can keep it constant and we can then discuss about the rest of the other variables how to keep them constant so on.

We have seen this when discussing about WDS we saw how we can keep vary the lambda and then we can keep the d constant and so on. But for x-ray diffraction of course we would like to keep the lambda to be constant and this is possible when we use a monochromatic radiation. So, that is fine. Now point is the monochromatic radiation. In order to generate a monochromatic radiation let us say I want to generate a K alpha radiation.

So, even if it is very fine and very well defined, but we have an instrumentation limitation. So, what happens is even though I want a K alpha pure K alpha radiation from my x-ray tube. So, like I want, an electron beam is hitting the target metal and then it is character. I am above a critical excitation voltage everything is fine; I am generating a K alpha but how I ensure that I am getting when it the x-ray beam is coming out of this extra tube.

How I will ensure that I am getting a pure x-ray K alpha x-ray. I can obviously use some kind of beryllium window is there just to make sure that I cut off most of the other wavelengths. But still normally what happens when an extra tube is operated at a voltage above this critical excitation voltage. Not only K strong K alpha but we also have a very weak we also have a weak but significant presence of K beta radiation.

And since K alpha and K beta these are very close wavelengths, that is why it is almost impossible to separate them out in a regular situation. And there will be also be a continuous spectrum which we already discussed mostly forms the background. So, all these things will be mixed together K alpha K beta and the continuous spectrum. Out of all of this as we understood, K alpha is the most desired one.

And the rest of this k beta or the continuous spectrum is undesirable one. K separating continuous spectrum is not that difficult. So, we can use this beryllium window and we can basically by absorbing most of the absorption edge is such that for the beryllium window the absorption is such that it will mostly it will eat all the other continuous spectrums. And remember one thing, this continuous absorption not only the absorption edge.

But other parts the absorption coefficient is pretty high and continuous spectrum the energy of the continuous spectrum is also pretty low in any other place except for this near to the short wavelength region. The intensity of continuous spectrum is pretty low. So, this beryllium window is quite well to separate out or to filter out the normal continuous spectrum.

Now the only difficulty that we have is how to separate the K beta line or K beta intensity from K alpha intensity, so, that K alpha characteristic emission so that we can get a pure monochromatic beam.

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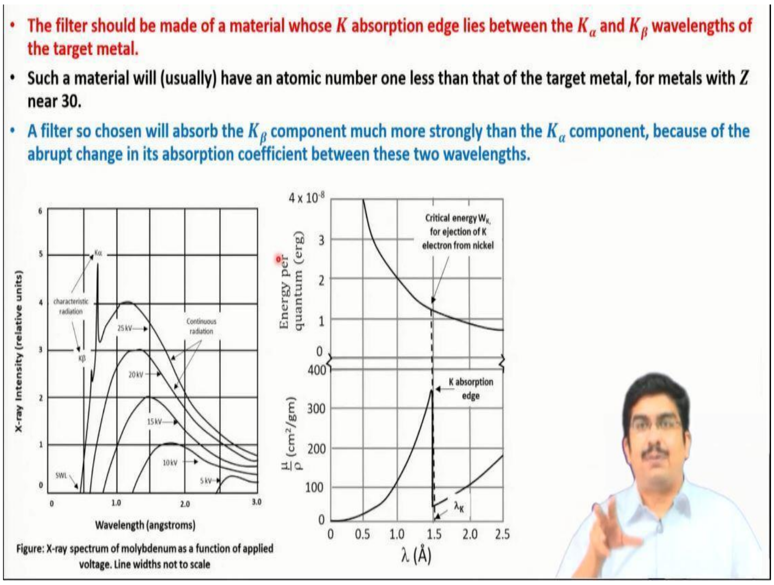

- The filter should be made of a material whose K absorption edge lies between the K_α and K_β wavelengths of the target metal.
- Such a material will (usually) have an atomic number one less than that of the target metal, for metals with Z near 30.
- A filter so chosen will absorb the K_β component much more strongly than the K_α component, because of the abrupt change in its absorption coefficient between these two wavelengths.

Figure: X-ray spectrum of molybdenum as a function of applied voltage. Line widths not to scale

So, that is where these x-ray filters the concept of x-ray filter comes in. So, we can use basically a filter to separate out the K alpha and K beta primarily. Of course, so the filter and here we will take the advantage of x-ray absorption. We understood that x-ray absorption also is very much specific; it happens at a specific wavelength. So, that also we have seen and it is related to the characteristic radiation only.

So, fluorescence radiation and characteristic radiation is basically the same thing we discussed about that in the last class. So, if we look at this absorption edge, we know that absorption K absorption h is happening over a very fixed wavelength. And the absorption coefficient also increases by many fold in this near this absorption edge and that is quite good enough to sort of stop any kind of extreme coming x-ray radiation.

If the wavelength is very close to, we have seen this in the last class if the x-ray the intensity of x-ray wavelength just close to this absorption edge is many fold just after this absorption. So, there is a huge possibility of absorbing the incoming x-ray if I am operating very close to this absorption h wavelength. And that is what we will try to take advantage of. So, we will put a filter so we imagine that an x-ray beam is coming out of the x-ray tube.

And this has K alpha and K beta radiations mixed. Now we put a filter we put a material in the path of this incoming x-ray beam in such a way and the choice of the material we make it in such a way that the K absorption h. So, this k absorption h is intensity of this material this lies between the K alpha and K beta line of or K beta characteristic radiation of the material target metal in this case.

So, target metal is generating a K alpha and K beta and I am using a filter material in the path of that beam which is coming out of the of the extra tube, which has its K absorption edge exactly between the wavelength I am talking in terms of wavelength exactly in between this K alpha and K radiation K beta radiation. The thumb rule normally, for such material to find out exactly a material.

So, I have to basically for filter I have to basically find out another metal which has an absorption edge atom wavelength between K alpha and K beta of the target metal. So, the thumb rule normally is that close to near atomic number of 30 so those transition elements basically they are mostly the transition elements which are typically used as a target metal copper cobalt and so on.

Thumb rule is that we use the filter material which has one atomic number less than that of the target metal. So, in for example, if the target metal has a atomic number of 30 we can use copper something like that so we can use copper with 29.

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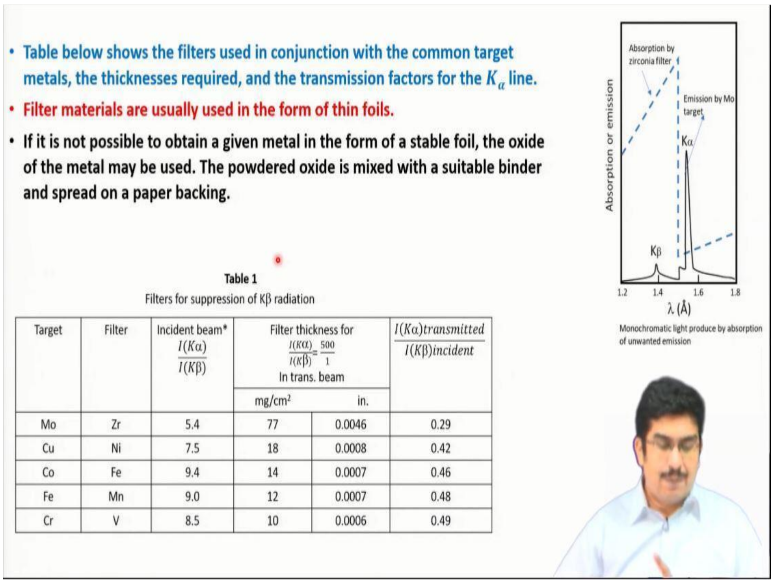

- Table below shows the filters used in conjunction with the common target metals, the thicknesses required, and the transmission factors for the K_α line.
- Filter materials are usually used in the form of thin foils.
- If it is not possible to obtain a given metal in the form of a stable foil, the oxide of the metal may be used. The powdered oxide is mixed with a suitable binder and spread on a paper backing.

Table 1

Filters for suppression of Kβ radiation

Target	Filter	Incident beam* $\frac{I(K\alpha)}{I(K\beta)}$	Filter thickness for $\frac{I(K\alpha)}{I(K\beta)}=\frac{500}{1}$ In trans. beam		$\frac{I(K\alpha)transmitted}{I(K\beta)incident}$
			mg/cm^2	in.	
Mo	Zr	5.4	77	0.0046	0.29
Cu	Ni	7.5	18	0.0008	0.42
Co	Fe	9.4	14	0.0007	0.46
Fe	Mn	9.0	12	0.0007	0.48
Cr	V	8.5	10	0.0006	0.49

Monochromatic light produce by absorption of unwanted emission

So, if it is, I think the relationship goes if I am not wrong yes, its copper and nickel. So, if nickel is there if the target is copper, I can use a nickel filter.

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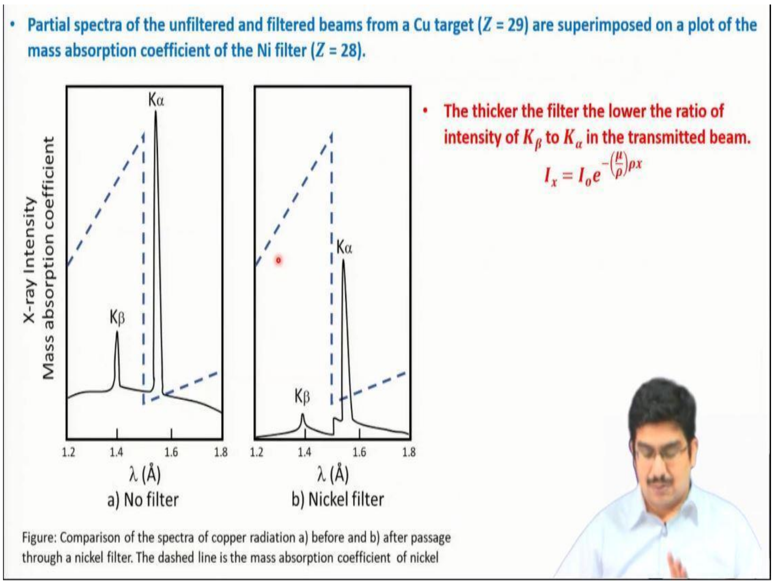

- Partial spectra of the unfiltered and filtered beams from a Cu target ($Z = 29$) are superimposed on a plot of the mass absorption coefficient of the Ni filter ($Z = 28$).

a) No filter

b) Nickel filter

- The thicker the filter the lower the ratio of intensity of K_β to K_α in the transmitted beam.

$$I_x = I_0 e^{-\left(\frac{\mu}{\rho}\right)\rho x}$$

Figure: Comparison of the spectra of copper radiation a) before and b) after passage through a nickel filter. The dashed line is the mass absorption coefficient of nickel

The most the most important example here; so, if I have a copper 29 as the target metal, I will be using a nickel filter 28 atomic number as the filter material. So, now what happens is if I think of about this then what this filter material does is it will absorb the K beta component because what is happening is if we can we can think of this one basically. So, we have the K alpha and K beta radiation coming out of copper.

And we have a nickel filter whose absorption edge is exactly in between that. So, wait for K alpha radiation the absorption it is before the absorption a where the absorption coefficient of nickel is pretty low. But right after this absorption is for K beta line the absorption coefficient it is after the absorption edge and this is now pretty high the absorption edge is or the absorption coefficient is very high for K beta line.

Whereas for K alpha line the absorption coefficient of nickel filter is pretty low. So, what will finally happen is that the K alpha radiation of copper can go through almost without much of an absorption; it can go through the nickel filter. But K beta now because of this absorption age is there the absorption coefficient of nickel will be sufficiently high to capture the K beta. So, K beta will be absorbed but K alpha will not.

So, filter material basically they are chosen in such a way that they will selectively absorb certain wavelengths from the incoming x-ray that is the purpose that is why it is called a filter material. So, this example again as I was talking, we are showing this for a copper target and for a nickel filter and this is what the initial. Initially this is how the spectrum looks like the characteristic spectrum of copper looks like K alpha and K beta.

They have quite K beta is quite large as compared to K I mean K alpha lines its considerable intensity is there for K beta. Now once I use this filter material K beta line is almost K beta characteristic radiation is almost gone. So, I am not getting much of a K beta here, so this shows the effectiveness of such filtering material and it takes advantage of the x-ray absorption. Now we will discuss couple of characteristics of this filter metal.

The first thing is we have seen from this equation absorption x-ray absorption equation is that the absorption of the material or the absorbing power of any material depends not only on its density definitely it depends on its density but also on the thickness of this material. So, the thicker this filter the higher will be the absorption of K beta radiation and the lower will be the ratio of the intensity of K beta to K alpha in the transmitted beam.

So, basically that means that if I use a thick filter, I will be able to absorb K beta much more efficiently and then in the final beam the K alpha radiation to K beta radiation this ratio will be much higher.

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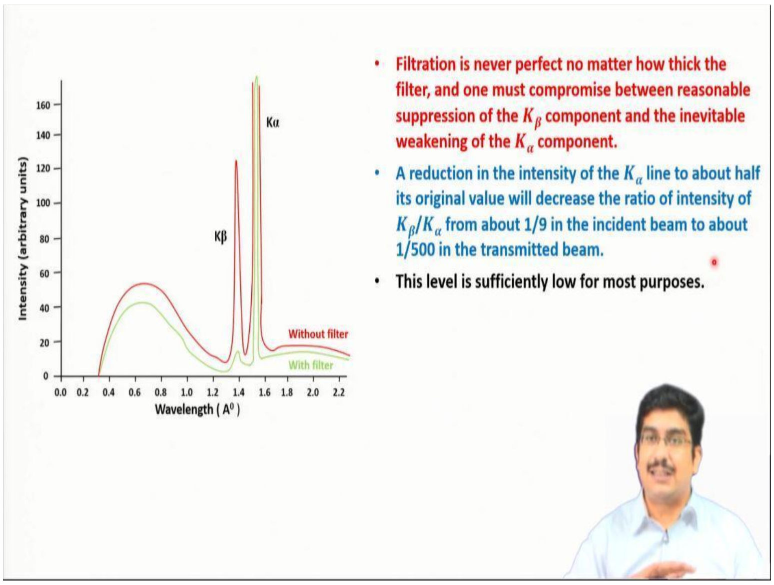

- Filtration is never perfect no matter how thick the filter, and one must compromise between reasonable suppression of the K_β component and the inevitable weakening of the K_α component.
- A reduction in the intensity of the K_α line to about half its original value will decrease the ratio of intensity of K_β/K_α from about 1/9 in the incident beam to about 1/500 in the transmitted beam.
- This level is sufficiently low for most purposes.

The problem in this process is that this filtration is not 100% perfect, what I mean is that even if the absorption coefficient of nickel is pretty low for K alpha but it is not completely it cannot be ignored. It is still quite significant to bring down the intensity of the K alpha radiation as well. So, that means the overall intensity of this x-ray beam will come down both for K alpha and K beta will almost be cut off.

But in the process K alpha radiation or intensity of the K alpha radiation will also be coming down. So, this filtration process is good to give me a K alpha radiation pure K alpha radiation but at the same time it will be sort of cutting down the intensity of the K alpha. So, this is sort of a trade-off in the process of suppression of this K beta component we are weakening the K alpha component.

So, that we have to accept that and depending on this our intended purpose we have to just we have to sort of very reasonably we have to use this filtering material. So, if you take some kind of a example any reduction in the intensity of K alpha line about half of its original value. So, if the filter material is such that it will reduce the K alpha value by almost half 50% of K alpha radiation is cut off in the filter.

But that will increase the ratio of intensity of K beta by K alpha to about 1 9th which is the original. So, this is if you look at this, this red one is the original line where K beta to K alpha ratio is 1 is to 9. So, for a pretty high amount of K beta is there from there after filtering this radiation this ratio will become almost like 1 is to 500. So, beta lines are almost completely removed and alpha line is still there at a ratio of 1 is to 500.

But what happens in the process is the intensity the absolute intensity of K alpha light is almost becoming 50% of what it was in the original material. So, but point is if we starting in the starting itself if, we use if you start with sufficient intensity for K alpha. So, even after the filtering the K alpha intensity used to be there, you have to make sure there is sufficient amount of K alpha intensities is there it remains even after filtering.

And to ensure that we start with a very high intensity of this K alpha line and this level 1 is to 500 is generally what is sort of recommended for any kind of diffraction experiment. So, the

K beta the filtering material, the efficiency of the filtering material is chosen in such a way or the thickness of this filtering material is chosen in such a way that it will be able to sort of bring down the ratio of K beta to K alpha at 1 is to 500.

Then if this is what the standard in or rather, I would say x-ray manufacturers, they follow this kind of a standard.

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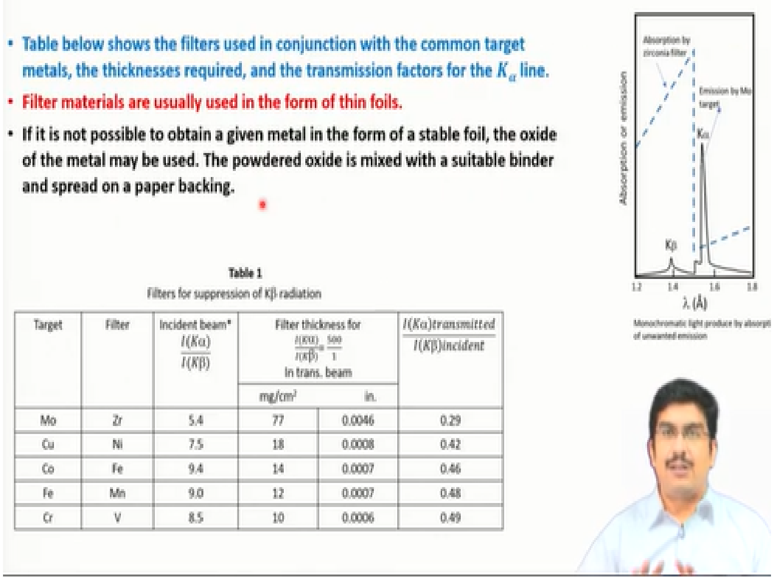

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Monochromatic light produce by absorpti of unwanted emission

So, now we will just quickly glance through some of these examples of these filter materials. And below this is a table which shows that what are the typical filters that is used for what kind of, so this is like a combination. Filtering material filter material and the target metal these two comes in a combination. You cannot just simply arbitrarily use any filter material for any target material.

If it is for example if it is molly you have to use something like zirconium. If it is copper you have to use nickel. If it is cobalt it is Iron so on and so forth. Now you can see the efficiency of this filtering material for example again we will take the copper nickel example. So, the incident beam has a K alpha intensity of K alpha versus intensity of K beta which is around 7.5.

Now if you use something like a nickel filter with a thickness of only 0.008 inch. That is the thickness very thin nickel filter you use and this is the kind of density that you use, this is the rho value. That you use there in nickel density and you will be able to now cut down the beta K alpha intensity of K alpha to intensity of K beta you will be able to maintain it at a ratio of 500 is to 1 which is as I just now explained that it is sufficient for any diffraction.

And that is the kind of gold standard for any diffraction experiment. So, only using a nickel filter of this much of thickness point very thin. And the density of this you can cut down most of the beta and if you look at now in the process if you look at the intensity of the ratio of the K alpha which is transmitted versus the K beta which is incident this ratio is coming down to be all only 0.42 from 7.5 it is coming down to 0.42.

So, remember this that how much and this tells how much K alpha radiation in the process of suppressing the K beta how much K alpha radiation is cut down. So, here we are taking the K alpha intensity of K alpha in the transmitted beam versus intensity of K beta in the incident beam. And compared to the incident beam now most of the K alpha, K beta is gone most of the K alpha also is sort of absorbed.

So, you are suffering a huge amount of decrease in the intensity but as I said this is a trade-off so you have to leave with that.

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Intensities of Diffracted Beams

So, we will stop our discussion about this x-ray filter here and we will start our discussion about the intensities of diffracted beams.

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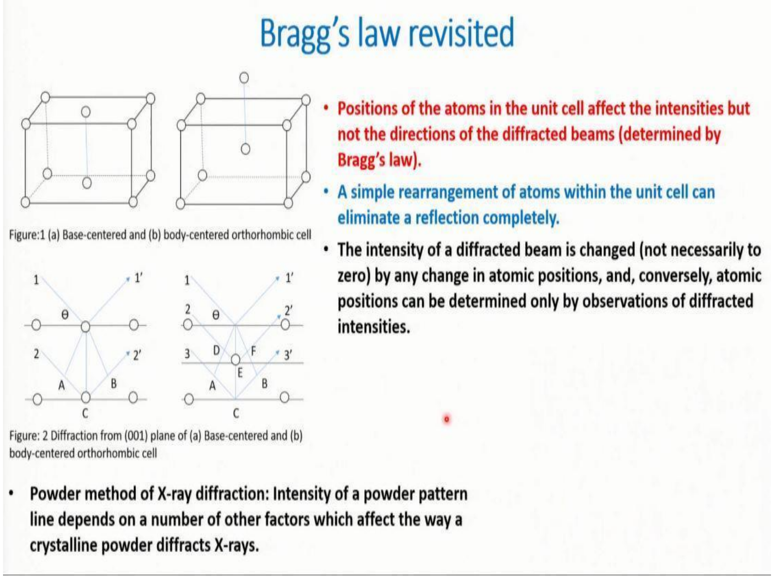

Bragg's law revisited

Figure:1 (a) Base-centered and (b) body-centered orthorhombic cell

Figure: 2 Diffraction from (001) plane of (a) Base-centered and (b) body-centered orthorhombic cell

- Positions of the atoms in the unit cell affect the intensities but not the directions of the diffracted beams (determined by Bragg's law).
- A simple rearrangement of atoms within the unit cell can eliminate a reflection completely.
- The intensity of a diffracted beam is changed (not necessarily to zero) by any change in atomic positions, and, conversely, atomic positions can be determined only by observations of diffracted intensities.

- Powder method of X-ray diffraction: Intensity of a powder pattern line depends on a number of other factors which affect the way a crystalline powder diffracts X-rays.

First thing we have to discuss is Bragg's law, as I said we already discussed about Bragg's law and we saw that Bragg's law is good enough to tell you exactly when the diffraction phenomena will happen. So, we all know that okay fine this is a Bragg's law, the typical way of defining Bragg's law n lambda = 2 d sin theta and we have proved it in geometrically as well.

That if the lambda is such that x-ray intensity is such and that when it falls on the atomic planes. And this lambda that wavelength is a basically integral multiple of the path difference which is defined by 2 d sin theta, then there will be and constructive interference and we should get a considerable or there will be constructive interference between the x-ray beams which are diffracted, which are scattered and we should get then considerable intensity.

So, this is what we have already seen. Point is this is half of the story. There is rest half. How? Basically, what happens is that position of the atoms in the unit cell that also affect the intensity x-ray intensity. Basically, Bragg's law gives you only the direction of the diffracted beam which direction, means how the x-ray beam should fall on an atomic plane and after diffraction after scattering in which direction.

These atomic planes will or these x-ray beams that is coming out of these atomic planes in which direction they will have the constructive interference, that is it. So, Bragg's law will tell you only up to that. The direction of the diffracted beam or the direction in which the; constructive interference will happen basically sin theta. If you know the 2 d that is your material and lambda that is your x-ray intensity.

Then which direction or which of the atomic planes in which direction this diffraction should happen. It does not talk anything about the intensity of the diffracted beam. So, in those direction the beam that is finally getting diffracted the beams which are extremes which are going through this constructive interference. What should be the intensity of that beam? That is another very important question.

And that question is directly related to the position of the atoms in the unit cell of any material. Basically, we express we will come to know that if we derive these conditions of intensity for a unit cell by the basic definition of the unit cell. That it is a repetitive unit it is the smallest unit which has all the crystallographic information about a material. Just by repeating the unit cell we can construct the entire material.

So, by virtue of that if we define how the intensities are depending on the atomic positions in an unit cell basically we can derive for the entire material, that is what. So, this is something that we will be going to discuss. How the position of the atoms in the unit cell are affecting the intensity of the x-ray diffractions. Even if Bragg's law is satisfied for them and this is simply because the arrangements of atoms within an unit cell can eliminate certain diffracted beams completely.

You will not if you do a real diffraction pattern. Even if Bragg's law is satisfied for those atomic planes still, you will not be getting any kind of an intensity because of some other kind of consideration. So, intensities that is why and those considerations are mostly related to the atomic arrangement in a unit cell. So, that is why what I am what we are discussing here is basically how this intensity of a diffracted beam depends on the atomic arrangement basically.

So, this intensity of the diffracted beam is changed by changing these atomic positions. If the atomic positions in unit cell changes the intensity of the diffracted beam also changes. So, if we measure this intensity of the diffracted beams by back calculation, we can find out the atomic arrangement. We can get an idea about the atomic arrangement within the unit cell. So, that means the determination of crystal structure of a material is not only sufficient.

If we just go by the Bragg's law, which will tell us that exactly which theta value which direction I will be getting a diffraction condition satisfied. We also need to measure the intensity of those beams at those 2 thetas. That will again tell you there about the atomic arrangement of any material. So, other than this definitely there are not only atomic arrangements of any material within the unit cell.

There are other factors which are also responsible for modifying the intensity of a diffracted beam. There are many other factors which affect the way a crystalline material basically diffract x-ray. And finally, the intensity of the x-ray beam diffracted beam that is coming out of the material. So, we will go through all, of this factors one by one, most of them most the important ones are mostly related to the crystallography of the material.

So, at this point I would assume that people who are doing this course they have a fairly good amount of knowledge about the crystallography of any material. And if you do not have that do not worry, please go through any basic book of crystallography because without crystallography this part particularly this part basically the entire diffraction. If you want to understand you should have a good knowledge about extra knowledge about the crystallography of material crystal.

The unit cell concept close packing and then symmetry and all other concepts you must have a very good knowledge about that. But particularly this part the intensity of a diffracted beam, to understand this part you must have a very good knowledge about crystallography, without that it is difficult to understand. So, we will take a simple example here how these atomic arrangements are basically influencing the diffracted intensity on and over this Bragg's law.

So, let us imagine this two crystallographic 2-unit cells here. Now one is the base centred orthorhombic another one is a Body centred orthorhombic base centre. Orthorhombic means you have so orthorhombic I hope it is clear to all. Orthorhombic means it is alpha beta gamma the angle between the directions all these lattice parameters is 90 degree but A B C the lattice parameters itself are not same that is orthorhombic.

So, in this orthorhombic the base centred one as it is showing that you have 1 1 extra atoms on the basis. And body centred means you have 1 extra atom in between here this is the body centred position. Now if we look at its 1 0 0 planes or 0 0 1 plane, whatever if we look at 0 0 1 planes, that is this plane, the side planes all the six side planes. If we look at this for this base centred one what we have is all the atoms are lying on these 0 0 1 planes.

I mean including this base centred atom at all. So, this may be the position or this distance is different from a base centre and a primitive one the distance is different. But basically, the base centred one is lying on 0 0 1 planes all of them fine. So, I have the 0 0 1 planes, here 0 0 1 planes here and so on and so forth. Now Bragg's law, if I look at the brag slope for this I have this diffracted beam from this plane, this set of planes.

I have this diffracted beam and I have another diffracted plane from another set of planes. The condition is basically here the lambda has to be an integral multiple of the path difference. And if it is satisfied these two will undergo and completely and constructive interference and let us imagine that that is what is happening. These two are in and this is the first order refraction so lambda this multiple the path difference is equals to lambda let us imagine here.

Now for this one the body centred orthodontic cell the point is you have again 0 0 1 plane here. Look at here, you do not have an atom here you do not here you have this atom. This atom is basically coming off this base centred 1, you do not have a base centring here. So, this atom is missing whatever it is. But still from other atoms you have again same thing this x-ray beam coming out of the first plane.

And x-ray beam coming out of this plane they are in phase; they are completely in phase. So, they will be diffracting no problem with the path difference here is exactly lambda. Point is if you look at these 0 0 1 planes you will have halfway between these 0 0 1 planes you have another set of atoms this is coming out of this. This is basically like I am seeing from the top I am seeing from the top.

So, I have this layer 1 layer this another layer and this I have another set of atomic layers which is in between these two 0 0 1 planes. Now from what happens is again the beam that is diffracted from here this will have exactly the path difference is lambda by 2 here. If we geometrically calculate the path difference of this one is exactly lambda by 2 that means instead of a constructive interference this one will cause this beam will cause a destructive interference with the first layer.

So, I will not get any intensity. If I consider only these two beams here, they will not undergo any constructive interference they will undergo a complete destructive interference. Similarly, there may be another atomic plane after this plane and that will undergo a destructive interference with this beam coming out of this atomic plane. So, ultimately what will happen is that all of these beams coming out of the 0 0 1 planes will nullify each other will undergo in destructive interference.

And finally, I will not be getting any intensity out of these 0 0 1 planes for body centred orthorhombic but I will be getting it for base centred at autonomic. Even if, the Bragg's law is satisfied for both of them just because of the atomic arrangements I will not be getting any diffracted beam from a particular plane for this one and I will get for that particular plane I will be getting a diffracted intensity for this one.

So, this is again basically tells you the same story that Bragg's law is not enough to have a diffracted beam there is atomic arrangement also plays a role in getting a diffracting intensity. So, we will stop here and will continue this discussion plus the Thompson's effect in the next class. Thank you.