

Techniques of Materials Characterization
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Lecture - 43
Characteristics X-ray Radiation

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. We are in module 9 and we were discussing about X-ray diffraction. So, previously we have discussed in X-ray diffraction last two lectures we were discussing about first history of X-ray diffraction, how it was discovered by Rontjen and then, Bragg's law and how Bragg's law was discovered, how it changes the entire scenario of X-ray characterization.

And we will also discussing about some similarities and differences between electron microscopy techniques and X-ray diffraction technique. And then we move on to some electromagnetic radiation in general and we saw that where does X-ray stand as an electromagnetic radiation. Then we discussed about the continuous spectrum X-ray generation, how X-ray generation happens.

And what is continuous spectrum, and what are the features of continuous spectrum like short wavelength limit and then, the shape of the curve with respect to the X-ray intensity continuous X-ray intensity with the wavelength and how it changes with the tube voltage and so on and so forth. All of these things we have discussed till until now, and in the third lecture, we will be discussing about the characteristic X-ray radiation.

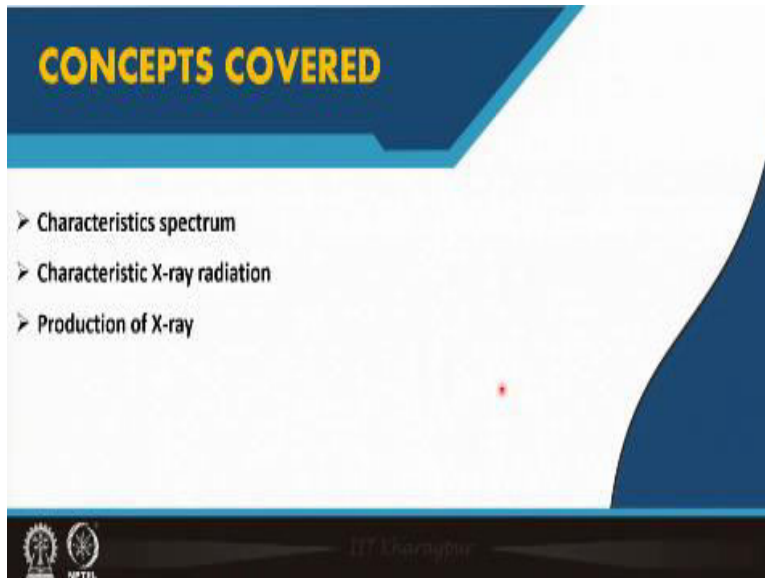
Now, one thing I must tell you before we begin is that characteristic X-ray radiation many of the things many of the mechanisms of characteristic X-ray generation and all, we have discussed when we were talking when we were in the scanning electron microscopy and there we have seen that how an electron beam as a result of elastic scattering the inertial excitation and then how it produces the characteristic X-ray signal, as a secondary effect and then how we can use that characteristic X-ray signal to for chemical analysis for doing chemical analysis.

We can use EDS detector, WDS detector and so on. So, in that sense some part of characteristic X-ray radiation that we will be going to discuss now, may appear almost similar and may appear that we are sort of duplicating we are repeating the same. One important difference you must understand at this moment is so, in electron microscopy when we were discussing about characteristic X-ray, their characteristic X-ray was a signal.

That is produced from the specimen and we were using it as a detection signal whereas in characteristic X-ray radiation in case of X-ray diffraction, we use characteristic X-ray as a source signal. So, we have this X-ray generation, mechanism of X-ray Generation, X-ray tube and so on. From there we generate characteristic X-ray spectrum and use it for further doing X-ray diffraction. In that sense, there is a little difference between the way characteristics X-ray are generated is pretty much the same.

But afterwards, why it is generated and various attributes of this is totally different in case of electron microscopy, where it is used as a source detection signal, rather than in X-ray, where it is used as a source signal. So, this important difference you must understand, and it will emphasize on that when we go through the rest of the part. But please in the right in the beginning you must understand, you must appreciate this difference between the characteristic X-ray in electron microscopy in the context of electron microscopy and characteristic X-ray in the context of X-ray diffraction.

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So, what we will be discussing today is basically characteristics spectrum X-ray spectrum in general and then characteristic X-ray radiation, how it generates and so on. And finally, we will discuss a little bit about the production of X ray, on the basis of whatever we discussed about the continuous spectrum and characteristic X-ray radiation, how the X-rays are produced which will be further used for X-ray diffraction.

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Characteristic spectrum

- While the continuous spectrum is caused by the rapid deceleration of electrons by the target, the origin of the characteristic spectrum lies in the atoms of the target material (anode) itself.
- Consider an atom consisting of a central nucleus surrounded by electrons lying in various shells, where the designation K, L, M, \dots corresponds to the principal quantum number $n = 1, 2, 3, \dots$
- If one of the electrons bombarding the target has sufficient kinetic energy, it can knock an electron out of the K shell, leaving the atom in an excited, high-energy state.
- One of the outer electrons immediately falls into the vacancy in the K shell, emitting energy in the process, and the atom is once again in its normal energy state.

The energy emitted is in the form of radiation of a definite wavelength and is the characteristic K radiation.

Labels in diagram: Specimen Atom, e-beam from SEM causes inner shell ionisation, Electron from Atom, e- from SEM.

So, characteristic spectrum, we know the continuous spectrum is caused by the rapid deceleration of electrons by the target. So electrons are produced from the cathode. And from that, there is a high potential is created between the cathode and the target metal which is working like an anode. There is a huge potential difference is there and then, this rapid this

electrons are accelerated under that large electron volt typically kilo electron volts in the range of kilo electron volts.

So, they are rapidly accelerated towards the anode and when they hit the anode, then it causes a rapid deceleration of the electrons and in that process, the energy of the electrons they lose that energy in the form of an extra photon. And some of them which are stopped in one impact or maybe one or two impact, give away all their energy in one shot one go and that creates the highest X-ray photon, highest energy X-ray photon corresponding the lowest wavelength and that is called short wavelength limit.

And some of the incoming electron signals they generate they stopped after many many impacts and which may include even some inelastic, elastic scattering process and so on and so forth. So, their plus point is, they are continuously they will be losing the energy and they will be emitting X-ray photons of lower energy and correspondingly higher wavelength. So, the continuous spectrum is a combination of many X-ray photons, many many X-ray photons.

So, from this continuous spectrum on top of the continuous spectrum, there will be a characteristic spectrum and characteristic spectrum lies in the way, these incoming electrons are interacting with the target material the atoms of the target material itself. So, that is the source that is the origin of generation of characteristic spectrum, what we got. So, one is the continuous spectrum generated by rapid deceleration of electrons and that process is sort of stochastic in nature, as a large broad range of wavelength and range of energies.

Whereas, characteristic spectrum is a special phenomenon, in which again it is also deceleration of the electron, which is also electrons interacting with the target metal atom and giving rise to another one kind of an X-ray signal, which is very much specific to that particular anode material used. So, now, if we consider then an atom consisting of in central nucleus, positively charged central nucleus and surrounded by electrons in various shells.

And these shells we know already K, L, M etcetera corresponding to the principal quantum number 1, 2, 3 etcetera. It is one of these electrons highly accelerated electrons under several

1000s of volts kilo electron volts, generated from the cathode and hitting this target metal. So, if this electron has sufficient kinetic energy it will knock an electron from this inner shell; that means, most problems will consider or it can happen for higher shells as well;

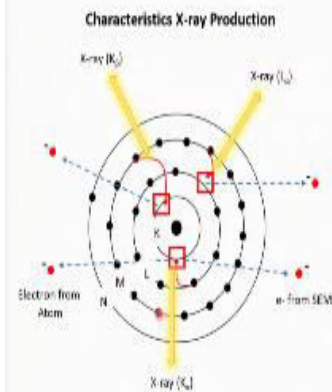
But let us consider that that electron has sufficient energy and it could knock out a K electron, which is closest to the positively charged nucleus. So, it knocks out an electron from this k shell, the incoming electron. So, definitely we have already seen this is kind of inertial excitation, it is an process of inelastic scattering in electron interaction and what happens is, the electron that atom goes into higher energy state;

And in the process of coming back to the ground state, what happens is, an electron from the higher from the outer shells from higher orbital, an electron jumps back to this K electron cell. And in the process, there is an amount certain amount of energy because all of these K, L, all the electron shells they have a very sharply defined energy. So, this process in this electronic transition process, a certain amount of energy is released and that energy usually is released in the form of an X-ray photon.

So, this X-ray photon that is released in the process is basically the characteristic X-ray photon. Because its wavelength, its energy is very much well defined and it is related to the atomic structure of this target metal atom. So, that is why this characteristic to this target metal atom. So, this is how basically characteristic spectrum or characteristic X-ray radiation happens and since it happens from the K shell electrons the initial process involves a K shell electron knocked out and then from higher outer electron shell and electron jumping to K shell. So, this is a characteristic K X-ray radiation.

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- K -shell vacancy may be filled by electron from any of the outer shells, thus giving rise to a series of K lines.
- K_{α} and K_{β} lines result from the filling of a K -shell vacancy by an electron from the L or M shells, respectively.
- $K_{\alpha 1}$ and $K_{\alpha 2}$ forms due to electronic transition from s or p -orbitals in the L shell towards the vacant K -shell.
- It is possible to fill a K -shell vacancy either from the L or M shell, so that one atom of the target may be emitting K_{α} radiation while its neighbor is emitting K_{β} .



- It is more probable that a K -shell vacancy will be filled by an L electron than by an M electron, and the result is that the K_{α} line is stronger than the K_{β} line.



Now, the K -shell vacancy that is created first, that can be filled by electron from any of the outer shells. There is no restriction as such. But, as I just now said that, there is a certain amount of energy is involved and that energy is related to the energy of the K -shell, and the energy of this outer shell. Basically the difference between the two and both of them all of them this electron shells between which this electronic transition happens, they are all have very sharply defined energy.

So now, if we imagine that this electronic transition can happen from any of the outer shell, we should get a series of K lines depending on where exactly between which two shells this electronic transition happened. For example, now, if we take some specific example, so we may have two different K radiations, depending on the outer shell or the electrons coming from the outer shell being, either the L shell or M shell.

So, if this transition let us say this is the K shell whether the vacancy has happened, this is the vacancy and this electron from the K shell, from L shell immediately next to the K shell. This electron jumps to this vacancy. Then the X -ray that will be produced is called the K alpha, K alpha radiation. And remember all of these electronic transitions are very much energetically, well defined specified energy. So K alpha electron will have correspondingly an energy and wavelength, which is depends on the energy difference between the K -shell and L -shell.

Fine, and then what will happen I have again next one is M-shell. Now, the same vacancy in K-shell vacancy; if it gets filled up by an electron from the M-shell. Then I will again have an X-ray photon coming out of it, and that X-ray photon will carry energy and wavelength correspondingly its wavelength will be decided by the energy difference between the M-shell and K-shell. So, that X-ray photon will be called as K beta X-ray photon.

So, this is the origin of K alpha and K beta lines, or K beta characteristic spectrum, characteristic radius. Now out in with in between this K alpha K beta within themselves, there can be even far more divisions. Because we know, these outer shells have orbitals, note the principal quantum number is the secondary quantum numbers. So, depending on that they can have s, p, d, f and so on and so forth.

So, let us imagine that we are using an electron, or we are using a target metal which has in the L-shell, we have two different orbitals s and p. So, if the electrons are now coming from in their L-shell, both of all of them are K-alpha. Now, if it is coming from s orbital of L-shell to the K-shell and K-shell only has s orbital, it does not have p and all. So, if it happens between the s orbital of L-shell and the K orbital, it will be called K alpha 1.

Similarly, if it happens between the P orbital of L-shell and the K orbital, then this electronic transition will give you X-ray photon known as K alpha 2 and they have as such as I said, all of these s, p all these orbitals also has a very specified energy. So, the X-ray photon which is generated will also have a very specific energy and correspondingly wavelength. Whatever minute difference, but they will have a different. K alpha 1, K alpha 2 will have a difference.

K alpha, K beta will have a different, fine. What also can happen is that, so this is what I am saying about one single atom. Let us say imagine, I have a same atom similar kind of atom next to it. So, once one atom once I generate K-shell vacancy for one atom, this vacancy is getting filled by say L-shell. So, I am getting a L alpha, K alpha radiation. The next atom it may so happen that this vacancy again an K-shell vacancy is created by the bombarding electrons.

Now, this vacancy is filled up by an M-shell electron. So, that will be emitting a K beta. So, there is no restriction from the target metal I can have all of this radiation possible, with very much random. Finite probability will exist of having even within the K alpha I can have L K alpha 1, one atom is emitting K alpha 1, another one is emitting at any given point of time any given instant. One of these atoms is giving rise to a K alpha 1, one of them is giving to K alpha 2.

Then one next one neighbouring atom is giving K beta, so on and so forth. That means the entire characteristic radiations they will have, it will be a combination of various energy and wavelength. Very close to each other of course, but still it will have a finite with all of this, if we consider these peaks K alpha 1, K alpha 2, K beta and all of this, they will give a peak in the wavelength versus energy diagram, we will see that.

So, that is why instead of characteristic X-ray radiation or line, it is a spectrum. The peak will have certain width, because of this random and stochastic nature of it. So, out of all of this K alpha, K beta and all these things, we can have K shell vacancy, the most probable cases it will be filled up by L electrons, then by M electrons. Because, the energy difference between L and K is lower than between that between L and M.

So, that is why the electronic transition is more probable between L-shell and K-shell. That means, we should get a stronger K alpha radiation compared to the K beta radiation. Even any given point of time, even if it is the process is completely random the probability of getting a K alpha line, is much higher than the probability of getting a K beta line. Because the K alpha line contains lower or needs lower amount of energy, it is lower energetically or rather energetically favourable process.

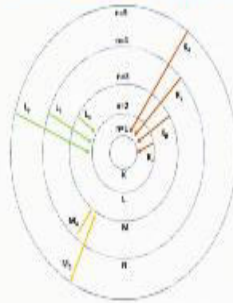
Because the energy difference between K and L shell is much lower than that between K and M shell, what other things?

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- There is a critical excitation voltage for characteristic X-ray radiation related to the sharply defined shells of target atoms being bombarded.
- *K* radiation, for example, cannot be excited unless the tube voltage is such that the bombarding electrons have enough kinetic energy to knock an electron out of the *K* shell of a target atom.
- If W_K is the work required to remove a *K* electron, the necessary kinetic energy of the incident electrons:

$$W_K = \frac{1}{2}mv^2$$

- It requires less energy to remove an *L* electron than a *K* electron, since the former is farther from the nucleus; the *L* excitation voltage (W_L) is less than that for the *K* radiation, W_K .



- It is impossible to excite one *K* radiation without exciting all the others.
- *K* characteristic radiation cannot be produced without *L, M* etc. radiation accompanying it.



Now, this process the entire process basically starts from the electrons, knocking out one K shell electron of the target metal. So, that means the incoming or incident electron that must have some amount of energy, critical energy that means, it should be accelerated to a certain under certain critical excitation voltage. If it is not getting that much of energy it will not be able to knock out an electron from K shell.

Because the electron; that electron in the K shell is bounded by the positively charged nucleus, with sudden work function. So, we have seen that, binding energy and so on. So, the incoming electron must have or must exceed that binding energy, then only it can knock out the K shell electrons. So, there is a critical excitation voltage, that is there, that exists. And usually in the electronic structure, the K shell electrons are having highest binding energy, that means they are strongest or their binding with the positive nucleus is strongest, compare to L, M and other shells.

So, that means the K radiation cannot be excited and in this process. So, one more thing. This process, I just now I took the example of K shell. Now, this can happen with L, M and other shells as well. So, the incoming electron may knock out instead of a K electron it will may knock out an N shell electron and L shell electron or an M shell electron. It can knock out. And those are obviously energetically, the energy required will be lesser than the K shell because of the binding energy difference.

To knock out an L-shell electron will be much easier for the incident electron, than the K-shell electron. So, that means, the K radiation and when I have a vacancy in L shell, same thing will happen. So, from higher energy state I will be getting or high outer shell, I will be getting an electron jumping to the K-shell vacancy. For example, it is shown here. So, when I have let us say imagine I have K shell vacancy.

Incident electron has sufficient energy and it knocks out the electron from K shell. Then what will I have, is that from M shell, I will have let's say an electron jumping to L shell. That will give now L alpha radiation. And if this happens, for example, let us say from N shell, an electron jumps to L shell, I will be having an L beta radiation. So, I can have L alpha, L beta, M alpha, M beta so on and so forth.

I can have all possible kinds of characteristic X-ray and all of them are very well defined, energy is very well defined, its wavelength is completely well defined because it is related to the atomic structure of the target metal. And similar to K alpha, K beta and all those things, I can have, like L alpha, L beta and all. And similar to K alpha₁, K alpha₂ like this, we will also have L alpha₁, L alpha₂ and even higher L alpha₃ and so on.

Because it depends from which if the outer shell electrons like M and N they have for example, if they have more higher order orbitals s,p,d and all, then I will be started getting L alpha₁, L alpha₂, L alpha₃ and so on and so forth. The point is, the critical excitation voltage on the energy of the incident electron, the energy needed to remove a K-shell electron will be much higher, than the energy needed to remove an L-shell or M-shell electron.

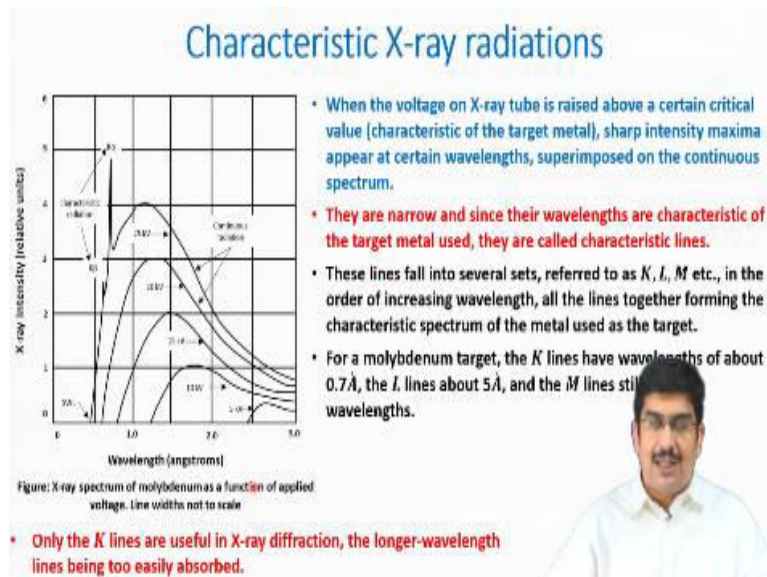
That means, what it means is that, in the process of generating a K characteristic radiation, I will be also generating L, M all possible characteristic radiation. It is invariably it will happen. So, that means, without exciting all other characteristic radiation L, M, N etcetera. I cannot generate or K radiation K characteristic radiation will not be produced until and unless there are L, M etcetera radiation are generated.

So, all together they make this characteristic spectrum, right. But let us just consider that, I am having a K characteristic radiation and this work required to remove this key electron and that is related to obviously the kinetic energy of the incident electron in this way. So, ultimately what will happen is that, I will be getting from the same specimen, I will be getting now K alpha, within first I will be getting a K characteristic radiation, M characteristic radiation, L characteristic radiation.

All of them I will be getting. Then within K characteristic radiation, I will be getting K alpha and K beta. Within K alpha I will be getting something like K alpha 1, K alpha 2 and so on. And same thing for L alpha and L beta; M alpha and M beta and all those things. So all of this together, will give the characteristic spectrum of and which is very much characteristic why it is characteristic, because it is completely related to this atomic structure of the target metal.

Even this WK the excitation voltage, that also is decided by the target metal itself. So, what excitation voltage I need to cause this characteristic emission, that also depends basically what is the atomic number or what is the electronic structure of this target metal.

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Now, this clear right; how the characteristic X-ray radiation are generated. Just know what I said that, I need to have a critical excitation voltage. So, let us now see this X-ray intensity versus wavelength curve again and this is again for molybdenum, this we have already seen in terms of

continuous spectrum. Couple of things we have clearly understood for continuous spectrum at least that, when I increase the continuous spectrum, when I increase this voltage to continuous spectrum has this short wavelength limit.

When I increase the voltage the short wavelength limit is coming in the left side, that means it is coming to the shorter wavelength side. That is first thing. Second thing, the highest the maximum in the continuous spectrum intensity, that also is coming to a lower and lower wavelength side meaning that, now the X-ray which is generated in the continuous spectrum, it is having higher energy.

Because of this increasing the tube energy means, the X-ray that is generated out of this continuous process, out of this rapid deceleration process. So, that also will be now having a higher energy. What if, we increase this tube voltage, higher than the certain this critical excitation voltage. Then what will happen? Now that is the question. So, what we can see is that up to let us say around 20 kilo electron volt, 20 KV for a molybdenum no change.

So, I have the continuous spectrum only thing I am keeping increasing in from 5 KV, 10 KV, 15, 20 etcetera. Short wavelength limit is shifting towards lower and lower wavelength, fine. And maximum the intensity is increasing and intensity at all wavelengths are increasing and the maximum in the continuous spectrum curve is also shifting to lower wavelength, three things is happening.

The moment I increase it to something like; 25 KV. Now, I get see that, in the continuous spectrum for some very specific wavelength, I am getting a sharp peak. On and above the continuous spectrum and the sharp wavelength the intensity of the sharp wavelength or the sharp peak for a specific wavelength, is way higher than that continuous spectrum. So, I am getting multiple such peaks as I was saying, so I am getting one peak here and then another peak here.

So, this shorter peak is basically K beta and the longer one the peak here is K alpha. K beta is forming at a smaller wave length because, it has higher energy, because it is happening between M and K. So, it has an higher energy that, X-ray which is generated due to electronic transition

between M-shell and K-shell, is having a higher energy and correspondingly higher energy, because higher energy difference. And correspondingly lower wavelength.

Whereas; K alpha is generated between L electronic transition between L-shell and K-shell. Of course, it has a lower energy and correspondingly, it will form at a higher wavelength in this spectrum. But as we just now discussed K alpha₁ is more probable than K beta₁, so the intensity of K alpha₁ will also be much higher than the K beta₁ finally ultimately, okay. So, this characteristic spectrum will form on and above the continuous spectrum at a very specific wavelength and they will form as a very intense X-ray signal.

In addition, they will be very narrow. The width of this characteristic spectrum will be very very narrow in the wavelength range and very sharply defined. So, I will have K beta at a particular wavelength and K alpha also at a very particular wavelength and they will not overlap in normal cases. They will not, they will be very much well defined in terms of wavelength and they will, their width the peak width will also be very, very narrow.

And so, these are the characteristic lines, they will fall as we just now discussed; they will be K alpha, K beta. Then similarly, I will have L alpha, L beta and so on and so forth. And that will be in the order of increasing wavelength of course, I will be having all K alpha here and then, I will be having K beta which is not shown here. The K beta lines will be forming an even higher wavelength, because they are much lower of energy.

They are, the electronic transition is happening L alpha, electronic transition is happening between M and L shell. Where the energy itself is much lower, binding energy is much lower. So, correspondingly the extra signal will also have much lower energy and much higher wavelength. Similarly for M- shell and all, now if we take couple of such example, let us say I have this molybdenum this one itself.

The K lines have wavelength of around 0.7. So, you can see the K alpha lines and K beta lines, they are all within this range around 0.7 a little bit different in there. The L lines will form somewhere over here, which is around 5 angstrom and M lines will form even at much higher

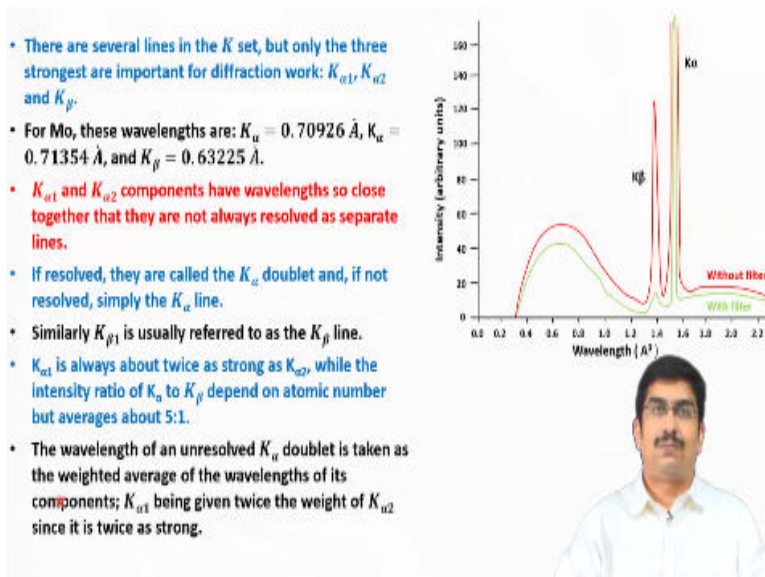
wavelength and generally, for diffraction experiment and remember one thing. We are talking about characteristic X-ray in terms of X-ray generation, as a source signal.

It is not we are not going in diffraction yet. When we go for diffraction, this characteristic radiation and all this continuous radiation diffraction or any kind of characterization with X ray, continuous radiation and characteristic X-ray signal all of them will be used as a source signal not a detection signal. So, right now we are just talking about what should be the best source signal.

And in that term, the K lines are useful for X-ray diffraction. We will see why? Just in a couple of minutes. So, K lines are very useful for X-ray diffraction the longer wavelength that is L, M etcetera. They are very easily absorbed; because their energy is very less and the wavelength is high energy is very very less. So they can be very easily absorbed, that X-ray absorption we will discuss, that time it will be more clear.

But it is mostly for diffraction experiments, this wavelength range is what is most important K alpha rays and we will be just focusing on their L and L lines, M lines will not be counting that much will not be discussing about them in many good details.

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So just now we saw there are several lines in the K set, and we are just interested about three strongest of them K alpha 1, K alpha 2 and K beta. That is it. So molybdenum, if we look at the typical values, K alpha forms around 0.70926. So you should note down after zero, there are so many digits and this is an angstrom, so you can imagine how precisely this wavelength are measured.

This is an angstrom already in angstrom. So even after that, we have almost like five digits after point. So you can imagine finally, the precision of measuring this wavelength. So that is very important. And that is important because, we are finally going to use this in diffraction experiments. So the d value also is important to be calculated in lattice parameter. So, if we are knowing this lambda value in a great precision, we can calculate the lattice parameter also with that very high precision.

But forget about it. Now so, K alpha 1 is this K alpha, this is K alpha 2 is around 0.71354 angstrom and K beta line is 0.63225 angstrom. So that means, basically K beta is forming at a smaller wavelength, then this K alpha. This is not molybdenum and do not get confused. This is not from molybdenum, that is why K beta and K Alpha lines are forming at a different wavelength altogether.

This is, if you compare here then you will be able to see that K alpha, K beta all of them are coming within that range 0.6 to 0.7. So, K alpha 1 K alpha 2 components, sometimes if you notice they have very close wavelength. Almost the difference between two is hardly 0.01 or even less than, that much of different angstrom. So, very very small differences there between K alpha and K beta and most of the time, here it is clearly they were resolved, they were identified.

This is a real experimental one experimental X-ray intensity versus wavelength spectrum here it is done in a very ideal condition that is why K beta and K alpha lines could be identified separately. But most often, they cannot be identified in that way separately, when they are resolved, they are called K alpha doublet. K alpha1, K alpha 2 they are called K alpha doublet. If

they are not dissolved, they are simply called K alpha line that is it. Similarly, K beta 1 is called K beta. For K beta identified K beta 1, K beta 2 is even far more difficult, for K beta 3 also.

So, these are even far more difficult to be they are wavelength is so close, there the transition is happening between M s,p,d of M to K. So, their identifying those wavelengths are even far more difficult, very very difficult almost impossible, I would say. So there K alpha 1 which is the strongest of all of them highest energy and that is why the K beta is K beta 1 is basically called K beta.

Simply we take K beta when we refer to K beta, means we are referring basically K beta 1. And K alpha 1 generally, this is what happens for most of the target metals, that K alpha 1 is generally almost twice as strong as K beta, than stronger than K beta. That means the intensity of K alpha lines is almost double than the intensity of K beta line. That is the typical ratio between K alpha and K beta. Similarly, K alpha and K beta, so when we collectively take this K alpha collectively take alpha 1 and alpha 2 and collectively take them as K alpha, the ratio between K alpha and K beta, that averages out around 5 to 5:1.

So, K alpha line is almost five times stronger than the K beta line. The intensity of alpha is almost five times stronger than K alpha 1. But most of the metal although it depends on the atomic number, we will see that. So, how the; intensity depends on atomic number. So, this is the relationship. Now, if the K alpha doublet is unresolved, that means we do not, we could not separate out K alpha 1 and K alpha 2.

Then the wavelength of this K alpha doublet, that is calculated as a weighted average of the wavelength of its components, K alpha 1 and K alpha 2 and as we just now discussed K alpha 1 is twice as intense as then K alpha 2. So, finally, that is how we take the weighted average of K alpha 1 and K alpha 2 with K alpha 1, given a weight of twice or double the strength then K alpha 1.

And that is how, we finally calculate the intensity of K alpha doublet. So, if we take that basically this intensity of a K alpha line comes to be around 7.1, 7.11 or something like that,

sorry, 0.711 something angstrom. That is the K alpha doublet, when we take that average. So, we will stop here and we have some more discussion about the characteristic. So we will continue in the next class. Thank you.