Techniques of Materials Characterization
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Lecture - 44
Characteristics X-ray Radiation (Continued) and X-ray Absorption

Welcome everyone to the NPTEL online certification course on techniques of materials characterization. We are in module nine and we were discussing about x-ray diffraction. So, until now, we have discussed about the history of x-ray and then x-rays and electromagnetic radiation and also, we have discussed about the continuous x-ray spectrum, how it is generated and what are the characteristics of it.

And then in the last class we just started with characteristic x-ray radiation which we will continue, in this one. And also, we will try to cover a little bit or we will at least try to start x-ray absorption.

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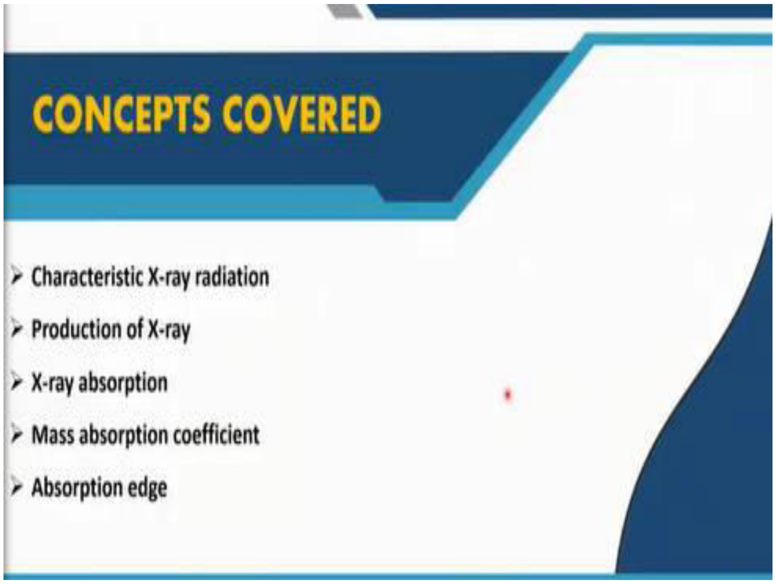

CONCEPTS COVERED

- Characteristic X-ray radiation
- Production of X-ray
- X-ray absorption
- Mass absorption coefficient
- Absorption edge

So, concepts of course, we will discuss about characteristic x-ray radiation first, then a little bit about the production of x-ray, and then x-ray absorption. And in x-ray absorption we will be discussing about mass absorption coefficient. And also, absorption edge we will discuss if time permits.

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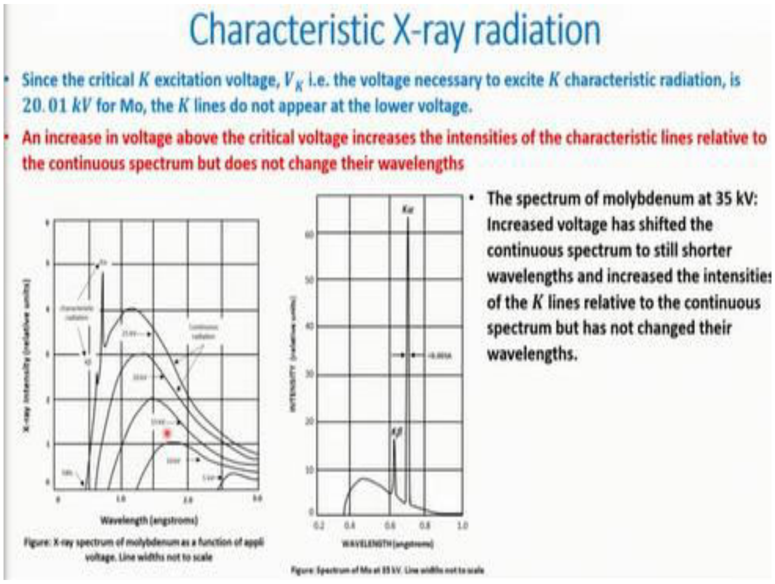

Characteristic X-ray radiation

- Since the critical K excitation voltage, V_K i.e. the voltage necessary to excite K characteristic radiation, is $20.01\ kV$ for Mo, the K lines do not appear at the lower voltage.
- An increase in voltage above the critical voltage increases the intensities of the characteristic lines relative to the continuous spectrum but does not change their wavelengths

- The spectrum of molybdenum at 35 kV: Increased voltage has shifted the continuous spectrum to still shorter wavelengths and increased the intensitie of the K lines relative to the continuous spectrum but has not changed their wavelengths.

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

Figure: Spectrum of Mo at 35 kV. Line widths not to scale

So, characteristic x-ray radiation we will continue where we ended in the last class. So, we discussed how the characteristic x-ray radiation basically occurs over and above the continuous spectrum. When the, when we apply the tube voltages above certain critical value and how we are getting K alpha radiation, K beta radiation etcetera, at a fixed wavelength. So, this critical excitation voltage VK which is basically the voltage necessary to excite K characteristic radiation.

And it is depends on, again this voltage strongly depends on the atomic structure or protons strongly depends on the element itself that is used as a target. But whatever if we imagine that that target metal is molybdenum and we are working at an excitation voltage above that or at an excitation voltage for molybdenum which is around 20.01 kilo electron volt. Then if we are excitation voltage that is the tube voltage.

If that tube voltage is less than this value then we will not get any of this characteristics radiation that is exactly what is happening here. So, if you look at this until up to around 20 kV, we did not get any of this characteristic x-ray radiation. Only when the tube excitation voltage is increased beyond these value 25 kV then we are getting that does not mean that critical excitation voltage is 25 kV, no critical excitation voltage is slightly above 20 kV, so, 20.01 kV.

So, if I can give a critical excitation voltage of around 20.05 kV then also, I should get K alpha and K beta radiations. And it is just that they experiment with the experimental limitation the voltage could not be increased or the voltage rather is only possible to increase at a multiple of 5 kilo electron volts. We could not do it for even less than, for this particular experiment at least. The point is then the critical excitation voltage in order to generate the characteristic x-ray radiation you have to go beyond this critical excitation voltage.

Then only we will be seeing in the spectrum x-ray intensity versus wavelength spectrum, we will be able to see the characteristic x-ray lines K alpha, K beta and all. So, if we increase it further the critical excitation voltage if we increase the tube voltage more than the critical excitation voltage. So, the acceleration voltages for the electrons are now increased beyond critical excitation voltage. Then what will happen is?

The intensity of the characteristic lines that means, the K alpha intensity K beta intensity all of them will increase. And that increase of course, we have also discussed this that if you increase the tube voltage, the intensity of the continuous spectrum will also increase and it will be shifting to shorter and shorter the short wavelength limit will go towards lower and lower wavelength. So, that will continue to happen with the continuous spectrum.

So, continuous spectrum will definitely start if you increase the voltage continuously beyond let us say 25 kV I increase, here there is one another example is shown for the same molybdenum target up to 35 kilo electron volt. And here you can see the short wavelength is now come down to around less than 0.4, whereas here it is still about point four until up to 25 kV. So, this will definitely happen the continue short wavelength will start coming in the continuous spectrum it will be continuously coming at lower and lower wavelength.

But the characteristic x-ray will form exactly at the same wavelength, whether you increase that tube voltage it will not change. So, if you increase the tube voltage from 25 kV to 35 kV the K alpha line will form exactly at that 0.7 around 0.7 Angstrom. And K beta will form exactly around 0.6 whatever we discussed in the last class within that value. That is not going to change between this tube voltage.

What will change is? The intensity of this K alpha line with respect to the continuous spectrum. So, continuous spectrum intensity will also increase continuous spectrum minima the maximum and continuous spectrum will also shift to slow smaller and smaller wavelength all of these things will happen. On top of that, the characteristic x-ray intensity will also increase if we increase the acceleration voltage.

And that is what is shown here you can see that both the K alpha, K beta both of them now become much more intense when I increase the acceleration voltage to 35 kV. And that we increase it further the same thing will happen this K alpha, K beta that intensity will keep on increasing but their position will not increase. So, unlike this is another this is a major difference between the continuous spectrum and characteristic spectrum.

Continuous spectrum I can change or I can shift the maximum, I can change the short wavelength limit by just by changing the tube voltage. But for characteristic x-ray spectrum, I cannot change the position of this characteristic lines. I only can change the intensity of them. So, this is one major change and that is another reason why these are characteristic because the way they are generated is basically related to only the atomic structure of the target metal.

Atomic structure atomic element atomic number only on that it does not depend on any other thing it does not depend the wavelength does not depend on anything else. It does not depend on the tube voltage it only depends on the atomic number or the atomic structure electronic structure of that particular atom.

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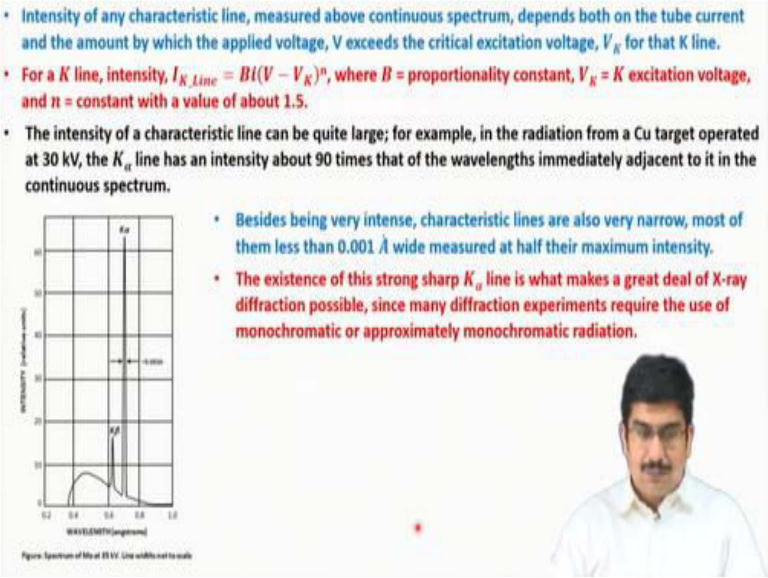

- Intensity of any characteristic line, measured above continuous spectrum, depends both on the tube current and the amount by which the applied voltage, V exceeds the critical excitation voltage, V_K for that K line.
- For a K line, intensity, $I_{K\_Line} = Bi(V - V_K)^n$, where B = proportionality constant, V_K = K excitation voltage, and n = constant with a value of about 1.5.
- The intensity of a characteristic line can be quite large; for example, in the radiation from a Cu target operated at 30 kV, the K_α line has an intensity about 90 times that of the wavelengths immediately adjacent to it in the continuous spectrum.
- Besides being very intense, characteristic lines are also very narrow, most of them less than 0.001 Å wide measured at half their maximum intensity.
- The existence of this strong sharp K_α line is what makes a great deal of X-ray diffraction possible, since many diffraction experiments require the use of monochromatic or approximately monochromatic radiation.

Figure: Spectrum of Mo at 35 kV. Line widths not to scale

Now, the intensity of any characteristic line which is measured as I said, it is measured over and if you look at another important point, if you look at this continuous spectrum here, now, the short wavelength limit has come to smaller wavelength compared to others. And the maxima; also has shifted to even smaller wavelengths and that is even smaller than the characteristic line. So; this continuous x-ray spectrum earlier up to 25 kilovolt.

This continuous spectrum the maximum in the continuous spectrum. So, that was at a higher wavelength limit than the characteristic x-ray. But now at the 35 kV that maxima has gone in the smaller wavelength side of compared to this K alpha. So, the intensity of this continuous spectrum and the intensity maximum intensity of continuous spectrum and the intensity of the characteristic spectrum these two are also pretty much related.

Because, if you yourself can understand that if the maximum of the continuous spectrum is falling exactly in here, then the intensity of the complete characteristic spectrum will also increase. That is why when we are considering the intensity of the characteristic spectrum always make this assumption or we always make this point that the intensity above the continuous spectrum. So, intensity of the characteristic spectrum above the continuous spectrum.

Or rather intensity of characteristic spectrum is the total intensity minus the intensity of the continuous spectrum for that particular wavelength. So, this is an important point to remember.

So, this intensity of characteristic line that is why it is written measured above the continuous spectrum, this depends both on the tube current. Tube current means number of electrons that is hitting.

So, basically the how many electrons are produced from the cathode it depends on that and the amount by which the applied voltage acceleration voltage exceeds the critical excitation voltage. So, these two things decide the intensity of the characteristic line which is measured above the continuous spectrum. So, how it is done? So, the number of electrons, if the number of electrons increases, then their intensity that will there will be more and more electron available to hit.

This target metal and in the process, there will be more possibility for generation of K shell vacancy and that will give rise to K characteristics radiation so on and so forth. More the probability of having K shell we can see will increase. Proportionally the probability of having or the intensity of K alpha lines will also go become higher and higher. So, that is how it depends on the number of electrons impinging on the target metal.

And in other words, it will depend on the tube current as well, again, it also will depend on the acceleration voltage. So, minimum acceleration voltage; is needed that is the critical excitation voltage. If I give anything extra to it, that means, the electrons will be much higher or having a much more energy carrying with them. So, they can produce a much more stronger impact on the target metal and can produce more K shell vacancy.

And correspondingly can increase the intensity of the characteristic line. So, this intensity of the characteristic line basically depends on these two things tube current and the acceleration voltage in excess of critical acceleration critical excitation voltage. So, if we consider this for the K line, then the intensity of K line again measured above the continuous spectrum, this is expressed as Bi into (V - VK) raised to the power n, B is the proportionality constant and n is another constant.

Usually, the constant value of n is 1.5. VK is K excitation voltage the minimum voltage required to produce this K characteristic lines that is already we discussed it is around 20.01 kilo electron volt for molybdenum. And the V is the applied voltage. Now, if we as we were discussing about here that we have applied 35 kilo electron volts which is around 15 electron volt kilo electron volt higher than the critical excitation voltage of molybdenum.

So, then what we can see is the intensity of this characteristic and intensity you can see it here the continuous spectrum is below 10 around 7 or 8 whereas, the characteristic spectrum is almost above 60. So, counts per second if we take the intensity as counts per second. So, this is around of that so, it is a relative unit usually it is expressed at counts per second x-ray photons counts per second.

So, this is almost six times or even more than if not 6 even 12 or 15 times then the maximum in the continuous spectrum. So, that is what one point if we suitably choose this acceleration voltage we can produce and characteristic K alpha characteristic line which is way more intense than the white radiation maximum and maximum of white intense and we can make it in fact at an example, we can take that if we use something instead of molybdenum if you use a copper target.

And the acceleration voltage is around 30 kV then the K line, K line characteristic line can be 90 times stronger than the wavelength which is immediately adjacent to it in the continuous spectrum. That means, the wavelength if you consider this is just for Moly, but if you consider the similar intensity versus wavelength diagram, it will draw it or if you can consider it for copper, then the characteristic K alpha radiation intensity of the characteristic K alpha radiation versus the intensity of any wavelength which is just adjacent to the characteristic radiation.

The difference is around 90 times. So, it is a very, very strong and very intense characteristic very intense signal not only it has a very specified wavelength but also it has a very it is very, very intense or you can make it so intense just by choosing the excitation voltage properly. Another important point is already we have discussed that besides being very intense the characteristic radiations are also very narrow.

That means the wavelength is very, very well defined. And there is very little deviation from that character from that particular wavelength because it is energetically. So, whatever be the randomness in the energy is there only that much randomness will be reflected in the width of this characteristic x-ray radiation which is often 0.001 Angstrom wide. This is when it is measured at half maximum, full width at half maximum that is almost of that range.

So, because of these two properties that is it is very, very intense, this characteristic x-ray radiation and it has a very particular, its wavelength range is also very, very narrow. That is why this x-ray; this characteristic x-ray radiations are best suited for x-ray diffraction experiments we have discussed about that. That x-ray diffraction experiments means n lambda = 2d sin theta. Now d is constant for any material that you are doing.

You have the sin theta which you are measuring in the detector and you need a monochromatic source for diffraction. So, that monochromatic if you have a very monochromatic source then you can have this entire diffraction condition satisfied for your sample, for a particular given sine theta. If it is not a monochromatic source, then d is fixed, you will have a range of sine theta for which the diffraction condition is satisfied.

You will be getting and broadening we have discussed this when we were discussing about the reciprocal lattice and how we are getting a finite spot instead of getting one particular point we were getting a finite side for the spot remember that. So; similar argument here if you start with a very monochromatic beam. Monochromatic means the beam itself with very narrow wavelength then that will be the best for diffraction experiment.

So, that is why the characteristic radiations are used mostly as a diffraction when we are using a for x-ray diffraction machine x-ray diffraction system only the characteristic radiations are used. Now, that does not mean that we do not have anything to do with the continuous spectrum. Now continuous spectrum is used when we wanted to have an x-ray image radiography or x-ray absorption to x-ray micro CT.

When we wanted to have that kind of an imaging what we need is a continuous x-ray source, why we will discuss about all of this when we discuss about absorption and so on. But they are the white radiation is more important because they are we do not want the diffraction to happen and the amount of or the energy or this x-ray intensity to we do not want the x-ray intensity to go down because of the this diffraction phenomena.

Which is another which is an important part of X-ray absorption. So, we do not want diffraction to happen and x-ray intensity to reduce because of that effect. That is why we wanted to have as broad an x-ray wavelength as possible and we use this white radiation there. We will come to that when we discuss about x-ray absorption anyway.

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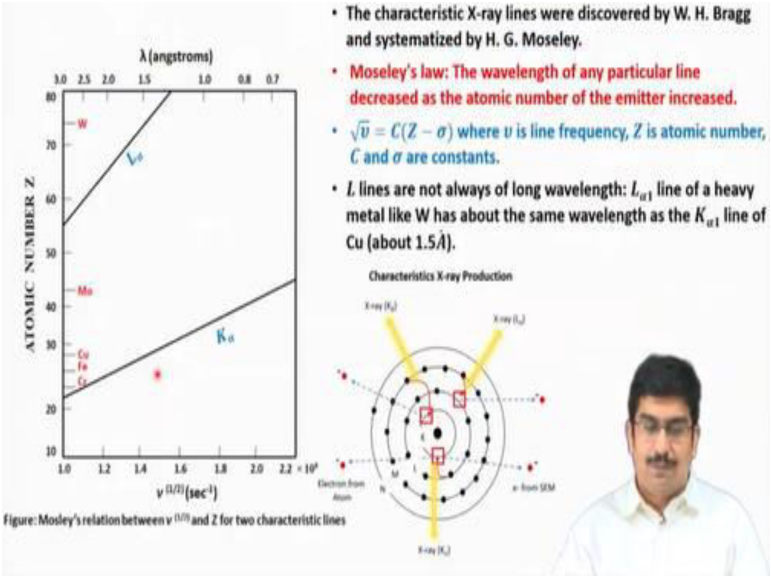

Figure: Mosley's relation between $v^{(1/2)}$ and Z for two characteristic lines

- The characteristic X-ray lines were discovered by W. H. Bragg and systematized by H. G. Moseley.
- Moseley's law: The wavelength of any particular line decreased as the atomic number of the emitter increased.
- $\sqrt{\upsilon} = C(Z - \sigma)$ where υ is line frequency, Z is atomic number, C and σ are constants.
- L lines are not always of long wavelength: $L_{\alpha 1}$ line of a heavy metal like W has about the same wavelength as the $K_{\alpha 1}$ line of Cu (about 1.5$\mathring{A}$).

Now, let us see on with what this characteristic radiation depends. As I said it depends only on the atomic number. So, basically the characteristic x-ray line was first proposed was discovered by W. H. Bragg. And after him this was systematized by another great scientist known as H.G. Moseley. What Mosely told is he has given basically a law and this law is connecting the wavelength of a particular characteristic radiation with the atomic number of the target metal which is used.

Basically, what it says is, the wavelength of any particular K radiation will decrease, as the atomic number of the target metal will increase. So, here it is shown in terms of line frequency you can you can imagine that this is inversely this is one by lambda basically root over 1 by lambda = C into (Z – alpha). So, that is the Moseley's law what he has given and this means that the weblength is inversely related to the atomic number.

So, if we see this is the Moseley's relation which is plotting here, we are plotting the frequency root over frequency root over frequency versus the atomic number and for different, different elements chromium iron copper moly tungsten and so on and two for two different characteristic radiation that is K alpha and L alpha radiation. Now, this gives us a very important clue and this is for L alpha.

So, tungsten K alpha is having a very low wavelength, so, that means very low wavelength, this one is higher frequency basically that means a very low wavelength of K alpha radiation for tungsten. So, by the time this one goes to here it is almost like it is not shown here, but, another important very important point so, K alpha radiation if you want to see for this transition metal copper iron chromium etcetera.

So, you will get the K alpha radiation within a reasonable wavelength the first thing and Molybdenum as well. Also, one more very important point to consider here, that the K L lines are not always up long wavelength that means, the L lines are not always of lower energy corresponding. We have not we already discussed this with K lines are usually of higher energy and lower wavelength compared to L lines but this strongly depends on the atomic number.

So, for example, if you consider something like tungsten, the L lines of tungsten has a much for, it is comparable that the wavelength of L lines of tungsten is comparable to that for K lines of copper. So, that is what it is saying. So, if you consider the K lines around copper K lines of copper is around 1.5 angstrom and the L lines of tungsten is also around of that wavelength. That is why the L lines of tungsten is almost as energetic or the energy of the L lines of tungsten is almost the same as copper K lines.

That means the energy ultimately the wavelength and the energy of this characteristic line strongly depends on the atomic number, if we use a heavy element then that element for that heavy element the K lines that will be characteristic lines that we will be getting will always be much more energetic. And correspondingly the wavelength will be much smaller than something like a lighter element compared to that. So, that is what Moseley's law and this can be explained from the Moseley's law.

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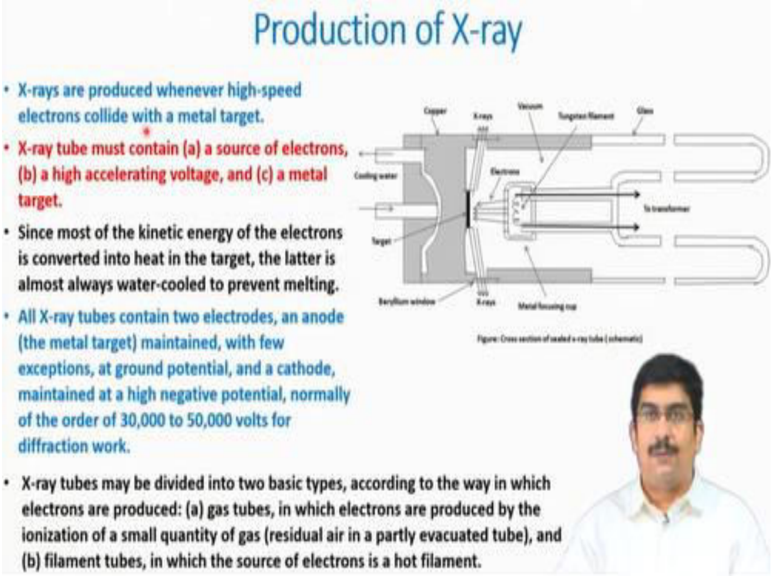

Production of X-ray

- X-rays are produced whenever high-speed electrons collide with a metal target.
- X-ray tube must contain (a) a source of electrons, (b) a high accelerating voltage, and (c) a metal target.
- Since most of the kinetic energy of the electrons is converted into heat in the target, the latter is almost always water-cooled to prevent melting.
- All X-ray tubes contain two electrodes, an anode (the metal target) maintained, with few exceptions, at ground potential, and a cathode, maintained at a high negative potential, normally of the order of 30,000 to 50,000 volts for diffraction work.
- X-ray tubes may be divided into two basic types, according to the way in which electrons are produced: (a) gas tubes, in which electrons are produced by the ionization of a small quantity of gas (residual air in a partly evacuated tube), and (b) filament tubes, in which the source of electrons is a hot filament.

Figure: Cross section of sealed x-ray tube (schematic)

So, here we will quickly discuss are from all of this discussion that we had till now about continuous spectrum and characteristic radiation. Now, we will be quickly see what is the implication of all of this, in the production of x-ray. So, production of x-ray as we already discussed is that two ways, we can do that. And both involve the whether we use continuous spectrum whether we use characteristic radiation whatever it involves one single thing.

That is we need to hit a target metal, with highly accelerated electron. So, in the process with rapid deacceleration of electron will produce the continuous spectrum whereas, this electronic transition K shell vacancy creation and so on, above a certain acceleration voltage above this critical acceleration voltage, we will be getting characteristic x-ray line. But whatever it is we put we better we need high speed electrons colliding with metal target.

So, that is what is the; basic working principle of this entire x-ray system that is called an x-ray tube. Usually, that is the name you will be seeing an extra tube or x-ray source. So, this x-ray tube or x-ray source usually contains a source of electron, that is the cathode a high accelerating voltage and then a metal target. And also then obviously, there are many more complexities involved in that already we discussed that this when these electrons hit the metal target.

Most of the energy of the incoming electrons goes in heat, goes or it converts to heat and hardly 1% of it converts to extra signal. That means the target metal or the entire system for that matter has to be cooled completely continuous cooling is needed and it is almost always water cooled this particularly this metal target is always metal always it is water cooled just so that it does not melt and that is why diffractive metals heavy elements like tungsten like Moly they are very good for as a metal target.

Because of their high melting point one advantage obviously is that they can give a highly energetic continuous x-ray spectrum the intensity of the continuous x-ray spectrum that these heavy elements they can get is much higher. We already discussed that even the characteristic x-ray spectra that they can give also is pretty high energy and intensity is very high. So, most often we see at least for continuous spectrum where the white radiation is needed, we generally tend to see the tungsten or tungsten molecule as a metal target.

If we need characteristic x-ray then generally, we use something like copper cobalt Moly and all those. Tungsten is rarely used for characteristic exploration because the acceleration voltage the critical excitation voltage for tungsten is very, very high. And it is very difficult to generate that much that amount because that is what is shown in the Moseleys law to generate K alpha radiation of tungsten it will be very much energetically high a very high energy is needed.

And electron should be accelerated to a very high voltage. So, that is why tungsten is rarely used for when we use the target source this source, we used it for x-ray diffraction experiments here we tend to use something like cobalt copper, iron, Moly all the materials all those sources. And wherever we need continuous spectrum not the characteristic spectrum we need continuous spectrum we tend to use tungsten target that is that is what generally is used.

But they are mostly always water cooled. Whatever the source you use whatever the target you almost always they are continuous water cool. Now, the x-ray source or x-ray tube contains ofcourse two electrodes, one is cathode and one is anode. The anode is maintained at ground state. It is a strongly it is grounded and it is almost at the ground potential the anode. Except for a couple of exceptions, we are not going into that.

But almost always this anode is kept at ground potential, whereas the cathode is maintained at a very high negative potential. And that is what gives this very high potential difference between the cathode and the anode, cathode produces the electron and under this very negative they are kept at a negative bias and under that this acceleration difference the potential difference between the cathode and anode.

And anode kept as grounded, electrons then gets accelerated and hits this anode material. And generally, the negative potential that is given depends on what is the target material use. Basically, what is the critical excitation voltage how or what above the critical excitation voltage what is the voltage you want to be depends on depending on all of these the critical the acceleration voltage is chosen.

Now, this x-ray tubes also can be divided in two types according to which the electrons are produced the how the cathode are basically producing the electrons. Number one it can be gas tube in which these electrons are basically produced by ionization of a small quantity of gas usually, residual air in a partly evacuated tube. So, this is called gas ionized or gas tubes where a gas is ionized and an electron flow is created on the free electrons are created.

Or otherwise, you can have a filament tube. In that case it is just like what we discussed for SEM just like the thermionic emission, you can generate it the filament basically works as a cathode and it generates a thermionic emission. And thermionic emission will give you this electron, as a electron beam which will then go and hit the anode. So, these are the this is basically the way the x- ray is generated.

The continuous and characteristic x-rays are generated and depending on the acceleration voltage and the target metal. You can either generate the same tube basically the way it is done is that if you want to generate a continuous spectrum you have to use a separate tube. If you want to generate a characteristic spectrum you have to use a separate tube. It is very much specific which wavelength which energy you are that x-ray sources giving it is very, very specific.

And even within characteristic x-ray if you want to generate only or want to use characteristic x-ray then the target metal you can change it you can go from copper to cobalt to Moly and each of them comes usually under a sealed tube and you can get you have to basically change the entire source. For changing for getting a different wavelength for the x-ray source you have to change the entire x-ray tube.

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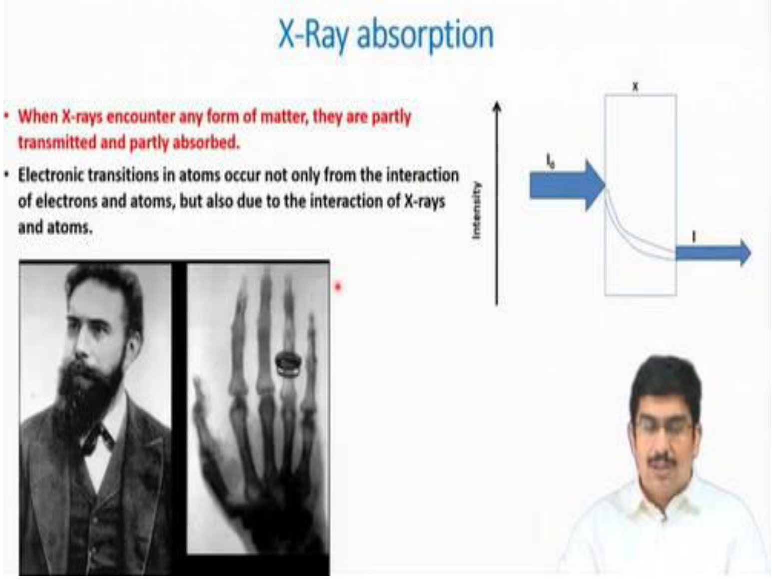

X-Ray absorption

- When X-rays encounter any form of matter, they are partly transmitted and partly absorbed.
- Electronic transitions in atoms occur not only from the interaction of electrons and atoms, but also due to the interaction of X-rays and atoms.

So, with this we close this discussion about x-ray characteristic x-ray and we will discuss about the x-ray absorption very we will start it and we will continue in the next class. So, when an x-ray encounters any form of matter part of them are transmitted and part of them are absorbed. Here extra encounters means I am talking now in the absorption part, I am talking about a continuous spectrum, I am talking about white radiation. I am not talking about characteristic x-ray.

So, I am talking just white radiation x-ray with all possible wavelengths. Just the intensity is important here not the wavelength of the x-ray signals. So, when an x-ray encounters any form of matter, they are partly transmitted and partly absorbed in the by the material. This is what basically is the first observations Rontgen made and that is why he could use it or people could use it for radiography.

Where we have seen that some material when it passes through the hand, then the flesh and the veins and other they were not able to absorb the x-ray they were basically transparent they were x-ray was able to penetrate they were transmitting x-ray. Whereas the bones which is much more denser, the bones was able to absorb the x-ray. So, on the other side, when you look at here the x-ray signal is entering with certain intensity and that intensity is somehow gets lost.

On the other hand, side, you are getting and modification of intensity and depending on the density of the material, if you consider that this extra beam is hitting different, different spots here. Then depending on the density, which density or the material it is encountering, I will have different intensity on the site. Now if I keep a film on this site, I will be able to generate this kind of an image that is what is basically the radiograph or that is what the x-ray imaging is all about.

The way this absorption happen this is basically related to electronic transition to some sense. The only thing to remember is that the electronic transition in atoms until up to now, we are only discussing the electronic transition because of the interaction of electrons and atoms. So, an electron is coming and knocking out some kind of K shell vacancy if we can see and then this electronic transition is happening, we are getting characteristic x-ray.

Sometimes we get Auger electrons as well depends on. But all of these cases we were just discussing are just considering the electrons as the source signal which is causing this vacancy and creating this interaction. So, now, in the x-ray absorption part, we have to consider the interaction between the x-ray and material atoms. So, that way it is a little different from the discussion previous discussion on characteristic x-ray generation.

Where it was electron material interaction which was giving the characteristic x-ray. Now, we what we have to consider or we have to discuss is basically the interaction between x-ray and atoms and how that is leading to x-ray absorption. So, this discussion will continue in the next class. Thank you.