

# Biomedical Ultrasound: Fundamentals of Imaging and Micromachined Transducers

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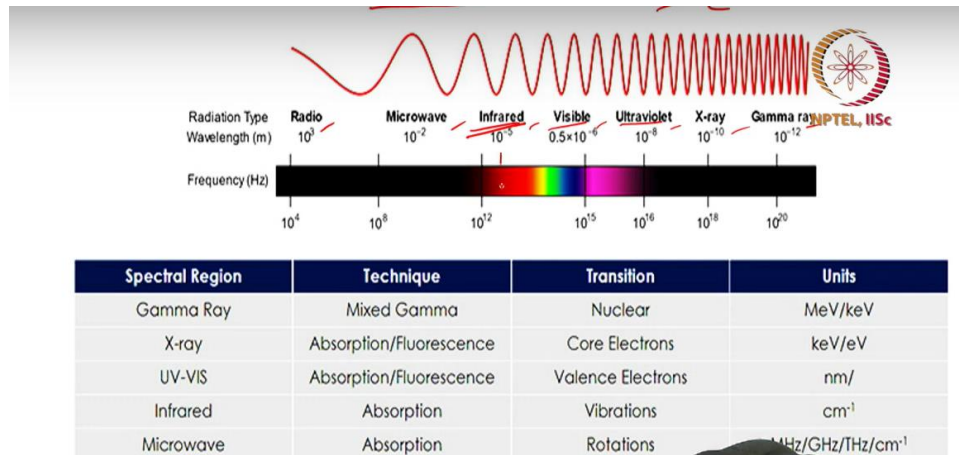
IIT Gandhinagar, IISc Bangalore

Lecture: 62

## Characterisation of materials III

So, hi everyone I welcome you all to another lecture of characterization of materials. So, we are studying it under the biomedical ultrasound course. I am Dr. Simranjeet Singh a national postdoc fellow at IISc Bangalore. So, let us begin. So, coming to the very basics.

So, first we will start with a quick recap. In the first lecture we talked about the electromagnetic radiation. Now electromagnetic radiation can be of several types based on the wavelength and the frequency. So, here is the spectra of the electromagnetic spectrum.

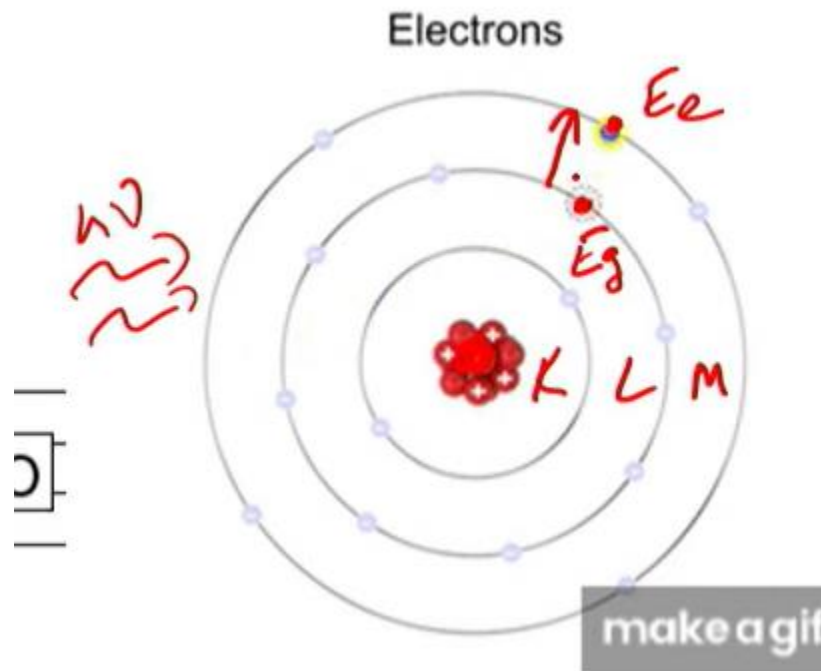


So, we go from the radio waves to the gamma rays, the energy increases. We talked about various classification from radio waves, microwaves, infrared, visible, ultraviolet, X-rays and gamma rays and currently we have talked about the infrared radiation and the visible and the UV radiation in our previous lectures. So, our visible radiation is only this part of the electromagnetic spectrum and only this region we can visually see and here this part is called the ultraviolet which is derived from the violet colour. it is just beyond the violet color. So, it is called the ultraviolet and then we have the infrared which is derived from the red color.

So, it is called the infrared it is just beyond the red color. So, we started from this. And then we talked about how we can use all these ways for the practical application and how we can utilize them for our characterization of the materials. So, in the first lecture we talked about the infrared radiation. So, in the infrared radiation just to give you a brief of this.

So, we have what is called the natural frequency in the molecules. So, if we consider the analogy that our atoms are a ping pong balls and the bond. that we have is a kind of spring. So, they have the natural frequency and they have the ability to absorb some part of the IR and according to that we get an IR spectrum typical IR spectrum to in detail if you want to learn about this you can refer to a lecture in the previous lecture regarding the basics of the EM waves and the basics of the characterizations and the infrared spectroscopy. So, in the last lecture we also talked about the UV and the visible spectroscopy.

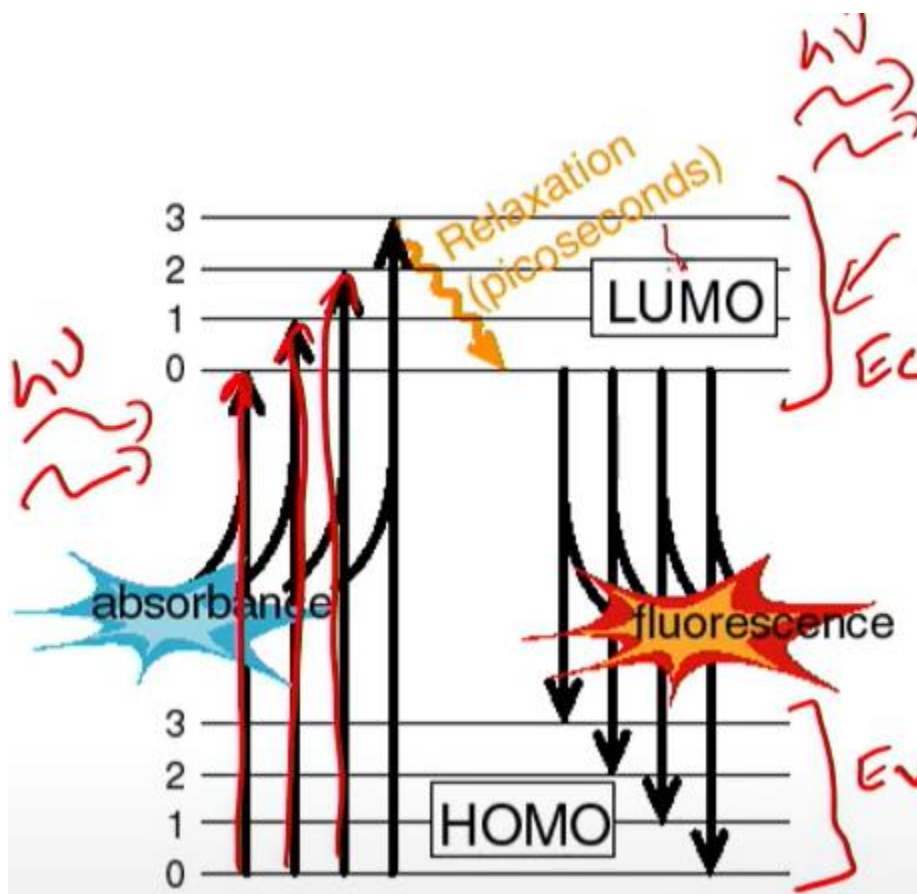
So, let us talk about this in brief. So, what we have is here is an atomic model you could see we have a nucleus, we have electrons which are revolving around the nucleus. they are revolving in form of inside the shells. So, let us talk about the basic terminology we call it KLMN. So, our electron revolves and rotates in these shells.



So, we talked about a phenomenon which was called absorbance. So, whenever the photon so, in form of energy in form of UV and the visible radiation is incident on our

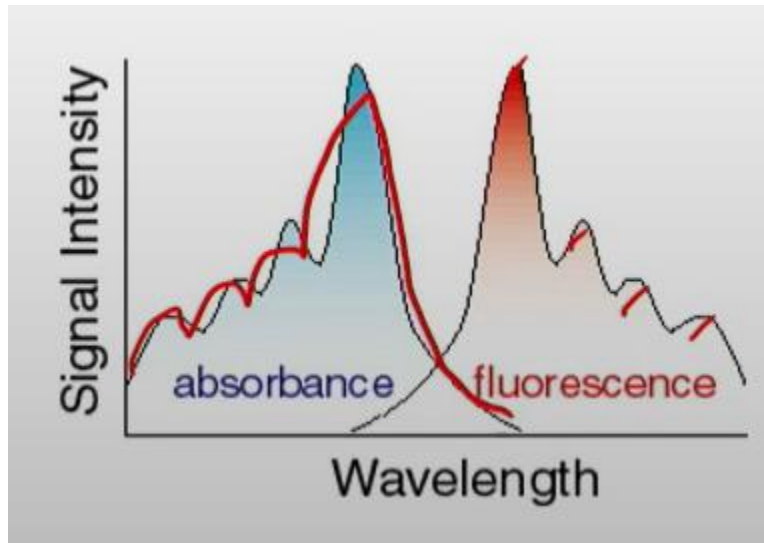
material. So, our electrons from the ground state. So, the initial state we will call the ground state which we are representing at  $E_g$ . and the excited state will be called  $E_e$ .

So, whenever the energy is incident on our sample our electrons from the ground state get excited and absorbs the energy and gets excited to the excited state. So, we are represented the ground state as the  $E_g$  and the excited one as the  $E_e$  and the phenomena is called absorbance. So, this was in very detailed explained to you in the previous lecture. So, if you want to know it in detail and the basics of the UV visible you can refer to our previous lecture. So, the initial phenomena are called the absorbance, but ultimately the energy is lost by the electron and will come back to its original ground state and the phenomena is called fluorescence.



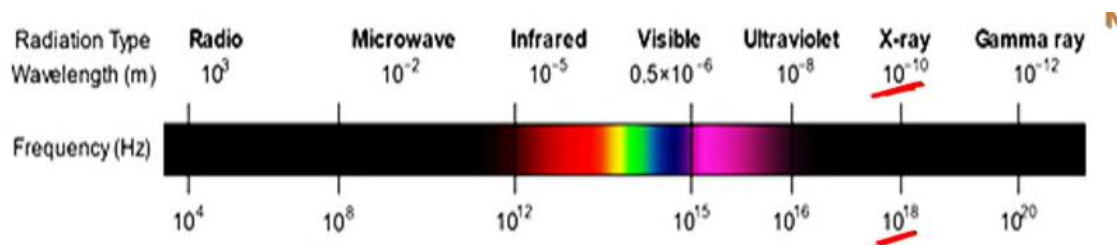
To better understand this, we will represent this in the form of molecular orbital theory where we have the valence band. So, we have the valence band, and we have the conduction band we are representing as  $E_v$  and  $E_c$  and the initial phenomena where light is incident in the form of UV visible is called the absorbance. So, based on the allowed transitions we have the typical absorbance spectra in case in our UV visible spectroscopy and after the certain thermal relaxations we get the electron comes back to its ground state or the valence band you can say in this case and the phenomena is called

fluorescence. So, we get a peak in the form of fluorescence. So, this we learnt it in the last lecture and today we finished our infrared, we finished our UV and the visible spectroscopy.



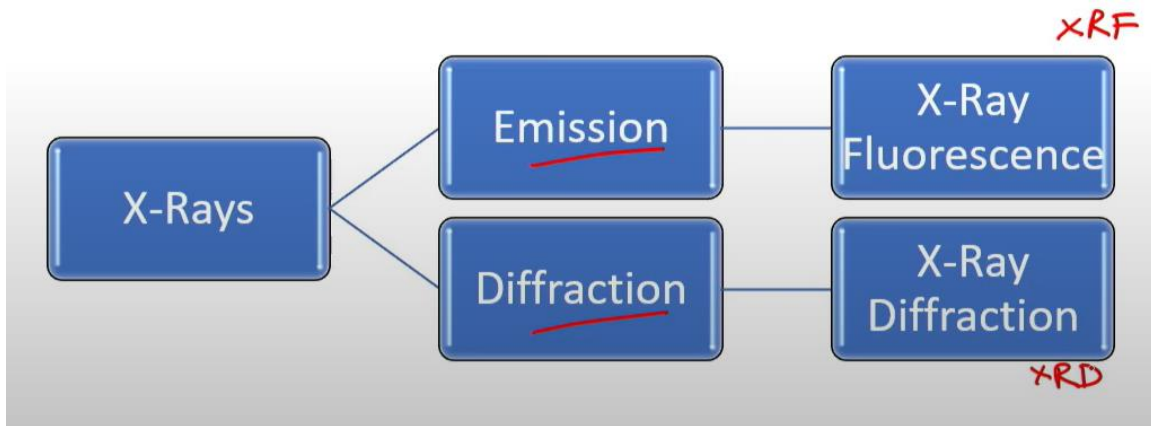
Now, we are jumping to the X-rays. So, as I said earlier, these three rays are very primitive and are very useful for the basic characterization of our materials. So, these are very extensively used. In this lecture, we will be talking about X-rays and how they can be used for the characterization of materials. Now, coming to the X-rays.

So, first I will talk about its wavelength. So, if you see this part of the electromagnetic spectrum is called the X-rays and the wavelength is the order of  $10^{-10}$ . So, as you can see the energy is increasing, so we call the X-rays very high energy rays. Wavelength is of the order of  $10^{-10}$  meters or if you want to see it in nanometers, it is of the order 1 to 10 nanometers, or we call it of the order of 1 Armstrong. So, Armstrong is a unit which is usually used for atomic spacing.



So, we will be using this term in the upcoming lecture also. Coming to the X-rays, the frequency is of the order of  $10^{18}$ . So, the frequency is directly proportional to energy. So, if energy is increasing our frequency will also increase, but the wavelength will decrease. So, this was the basic concept that we learned earlier also.

So, coming to X-rays, X-rays are really high in energy as I talked about and based on the X-rays, we have two phenomena that can take place, and we can utilize this for our characterization of materials. So, first one is called the emission, and the second one is called the diffraction and the emission phenomena we could use in an instrument which is called X-ray fluorescence in short, we call it XRF and the diffraction phenomena we could use in the XRD, or we call it the X-ray diffraction spectroscopy. So, we could use X-rays in XRF and XRD spectroscopy. So, what are these phenomena? Since x-rays are really high in energy, they have the ability to knock off the inertial electron and because it knocks off the inertial electron, we have a phenomenon which is called emission. As I talked in the initial one also, I will talk about the emission in detail in the later slides.

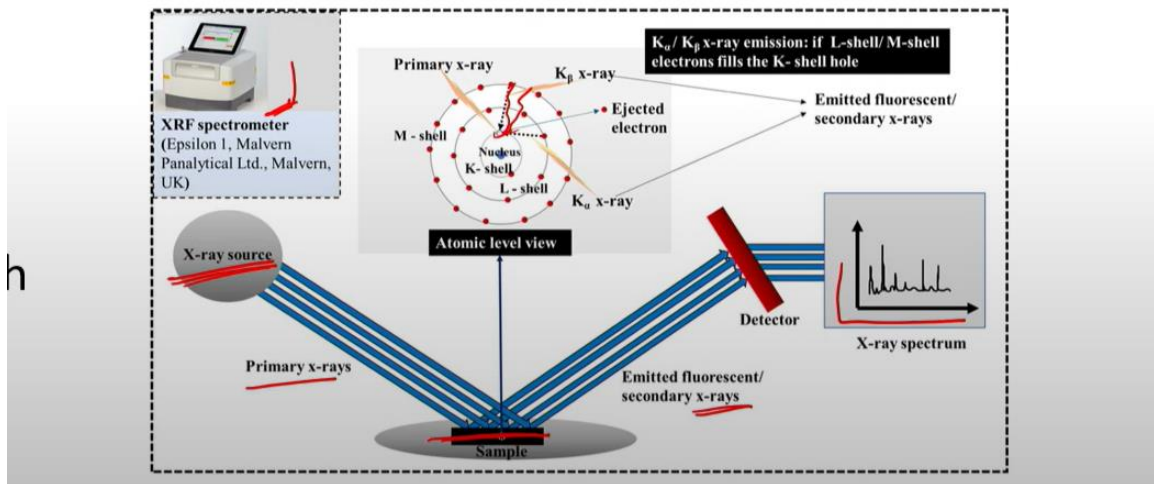
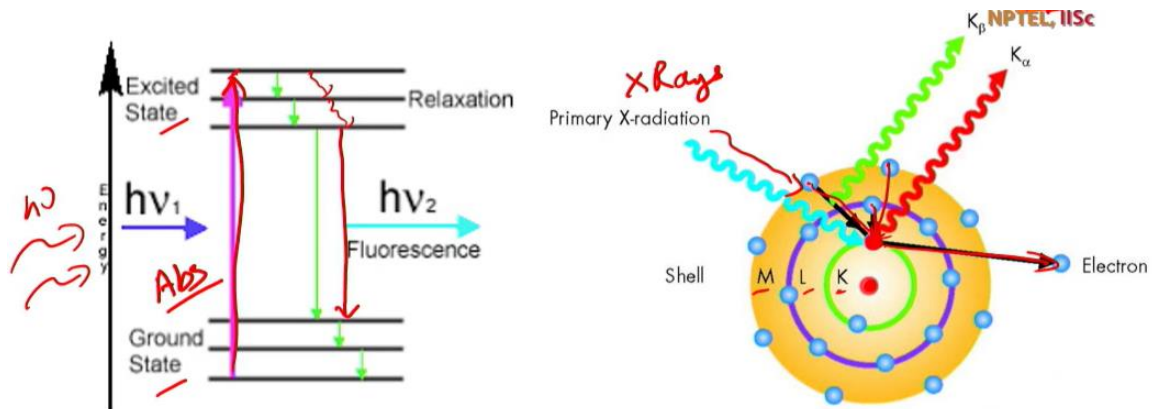


And also, since the wavelength of the radiation is of the order of Armstrong's. So, Armstrong's is usually the atomic spacing also is usually in the Armstrong range. So, since the radiation matches with the atomic spacing. So, we also have a phenomenon which is called the bending of light which is called diffraction, and we can utilize it for the XRD spectroscopy. So, we will be learning about XRF and XRD in the later slides.

So, coming to the X-ray fluorescence spectroscopy or the XRF. So, let us talk about the very basics of fluorescence. So, as I initially talked also, so if an energy is incident on our material. So, we have a phenomenon which is called absorbance, we utilize this in UV visible absorbance spectroscopy. So, we have something which is called absorbance, the energy is absorbed by the material, it gets excited from the ground to the excited state by absorbing that radiation. So, once it absorbs the radiation it is an excited state. So, what electron does is that it loses some of the energy in the form of thermal relaxation which we call and then a fluorescence is emitted then the energy is emitted in the form of fluorescence. So, we could utilize this phenomenon in our X-ray fluorescence spectroscopy. So, what happens is when we have this atomic model, we have this nucleus, we have the shells K, L, M, N.

So, what happens is we have x-ray radiation. So, we will have the primary x-rays. So, the x-rays fall onto our sample. So, what it does is that it knocks off the inner shell electrons. So, the electrons that are in K shell for example, are knocked out.

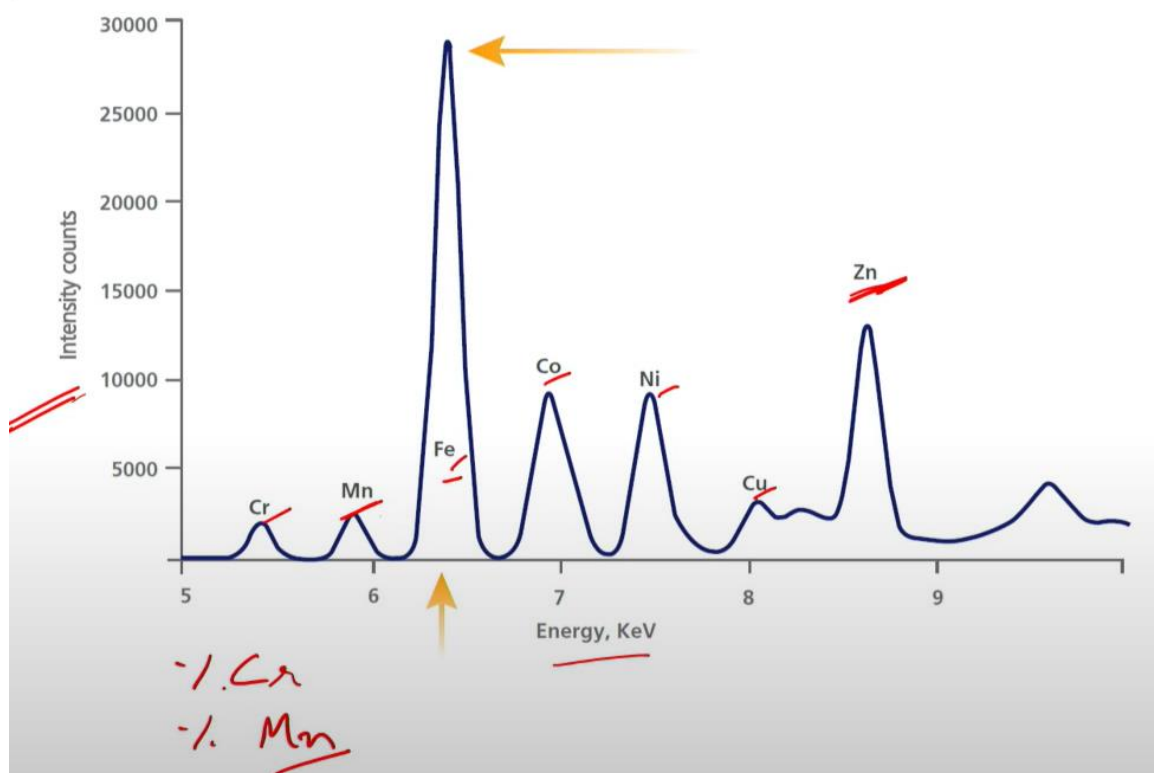
What happens is a vacancy is created there. Now, what will happen? The electrons from the outer shell like from the M shell or the L shell will tend to take their place or will tend to fill that void. So, during that a secondary emission which is in the form of fluorescence is generated and we record which is called secondary X-rays. So, secondary X-rays are emitted, and we detect that. So, what happens is that the secondary emission that is emitted is very primitive and it is very distinctive to that particular material.



So, what we can do is it helps us to identify what all elements are there in our sample. So, let us talk about the basic working of our XRF. So, the first thing that we have is the X-ray source, it falls on to our sample. So, in the form of primary X-rays is folded on to our sample, then the vacancy is created the inner electrons are knocked out and then the secondary the electrons from the outer shell tend to fill the inner void and the secondary fluorescence are emitted. So, secondary X-rays are emitted which are then recorded in our X-ray fluorescence spectroscopy or the X-ray fluorescence spectra that we get.

So, based on this we can identify what all elements are there in our sample. So, here you can see is the instrument of the X-ray fluorescence. So, it is the model of Malvin Pan analytical. So, it is the company that makes the X-ray fluorescence instrument. So, this is the basic phenomenon and based on this we can use it in several kinds of applications.

So, we will talk about some of the applications. So, the very first application is the identification of unknown constituents in the sample. So, as I talked about so, supposingly let us take an example. So, you are an archaeologist, and you get a sample from so, you have a soil sample and you want to identify what all elements are there in that sample. So, what you will do is run it through the XRF spectroscopy and the XRF will help us to identify what all elements are present in that particular sample.



So, basically what is the advantage of XRF. So, let us talk about the advantages of XRF. So, the advantage of XRF is it is a really tiny instrument, and it is a real cost effective and efficient technique and moreover it is portable as well. So, you can take it to a particular site. archaeology site and you can run the XRF there itself.

So, the advantages it is easy portable, and it is non-destructive as well. So, you can utilize it for powder samples, for liquid samples and for any kind of samples XRF is really easy to use and it is a non-destructive technique. So, you can utilize your sample again and again for different kinds of characterization. So, talking about the advantages it gives us the identification of the unknown samples.

So, let us talk about the example here. So, here is the typical spectra of our XRF where we have the intensity versus the energy peaks. So, supposingly we have this unknown material, and we run it through the XRF, and we get the typical XRF peaks, the typical secondary fluorescence of all these materials. So, it gives us confirmation whether all the materials are present or not. and not only quantitative XRF also has the ability to give the quantitative analysis as well. So, we could actually find out how much percent of chromium or how much percent of manganese is or how much percent of iron is available in our particular sample.

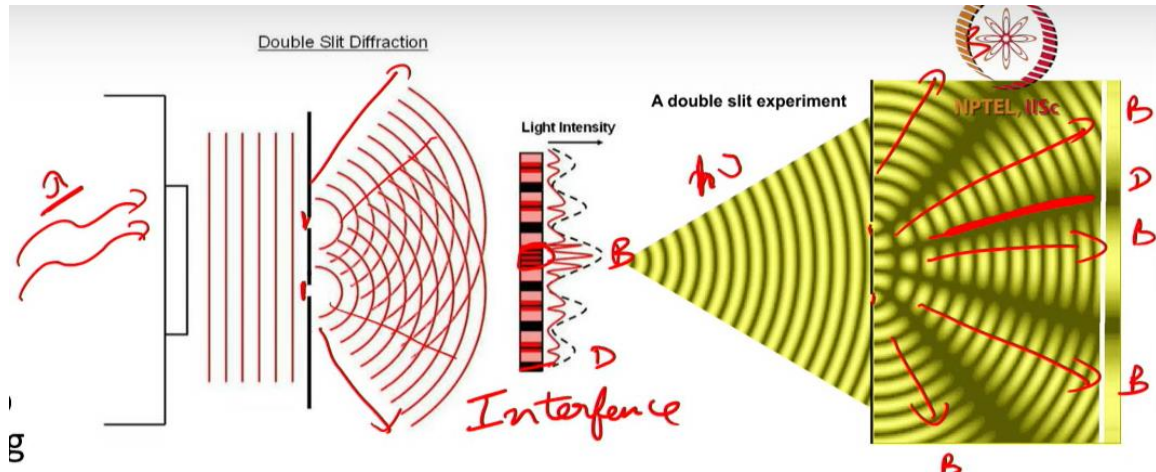
So, this is the advantage of using XRF moreover if you are a material scientist. you are synthesizing some kind of nanomaterials or any kind of materials. For example, I have talked about the zinc oxide earlier also, supposingly we synthesize zinc oxide material and then we have used some dopants like we have some dopants like manganese, or we have doped it with supposingly silver or manganese, supposingly we have doped it around 10, 10 or around 10 or supposingly 20 percent. So, 20 percent manganese, sorry 10 percent manganese and 20 percent of silver we have doped it and we want to see whether our material has been successfully synthesized or not and we also want to quantify whether our dopants that we have doped is of the correct order or not. So, what we will do we will run it through the XRF, we will get a typical Zn peak, we will get a typical Zn peak in our sample.

And we will also get the MN peak and we will also get the silver peak in our sample and it will give us a quantification chart like how much percentage or which sample is available or which element is available and based on that we could actually confirm that our material has been properly synthesized and it has the proper amount of dopant that we have used during the synthesis. So, that is the second application that we can do. The third application is for the key chemistry scientist also they synthesize different kind of material also and then they functionalize those material. So, the functionalization of the material. So, they can detect it using the XRF and also the electronics people whenever they synthesize. They deposit thin films onto a substrate, and they want to confirm whether the thin film has been successfully deposited or not and how much is the quality of the thin film that can be detected using the XRF spectroscopy.

So, coming to the, taking, looking at the advantages of all the possible XRF spectroscopy and talking about some of the applications of the XRF, we will jump to another application of the X-rays. There is a phenomenon which is called diffraction. Now, what is diffraction? Diffraction is basically the bending of radiation. So, this phenomenon you might have read in your plus 1 or plus 2. So, we will understand diffraction, we will talk about the very basic example.



So, this experiment is Young's double slit experiment. This you might have read in your plus 1 or plus 2 in very detail. So, in Young's double slit experiment we have two slits, and the slit width is very less. So, we have some radiation incident radiation and what happens is that in this experiment the wavelength that we have of the radiation is of the order of slit width here. So, bending of light happens or a phenomenon of diffraction happens.



So, the light will bend like this from both the slits. Since we have two slits, we have two diffraction patterns and these two wave like diffraction pattern will interfere with each other and what we will have is interference pattern. we have an interference pattern between these two wave like structure and because of which we have some bright bands we will represent as B and we will have some dark bands we will represent as D. To better understand this, we have this.

So, we have a wave. So, we have the incident radiation it is in the form of  $h\nu$ . So, this wave will actually fall on these two slits and a diffraction pattern will occur and based on the diffraction pattern, these diffraction pattern will interfere with each other, and we will have some bright bands in which these waves interfere constructively, and we will have some dark bands in which they will interfere destructively. So, we have destructive and constructive interference based on which we will get the bright and the dark bands. So, this is the very basic and I probably feel that you must have read this in very detail in your previous classes.

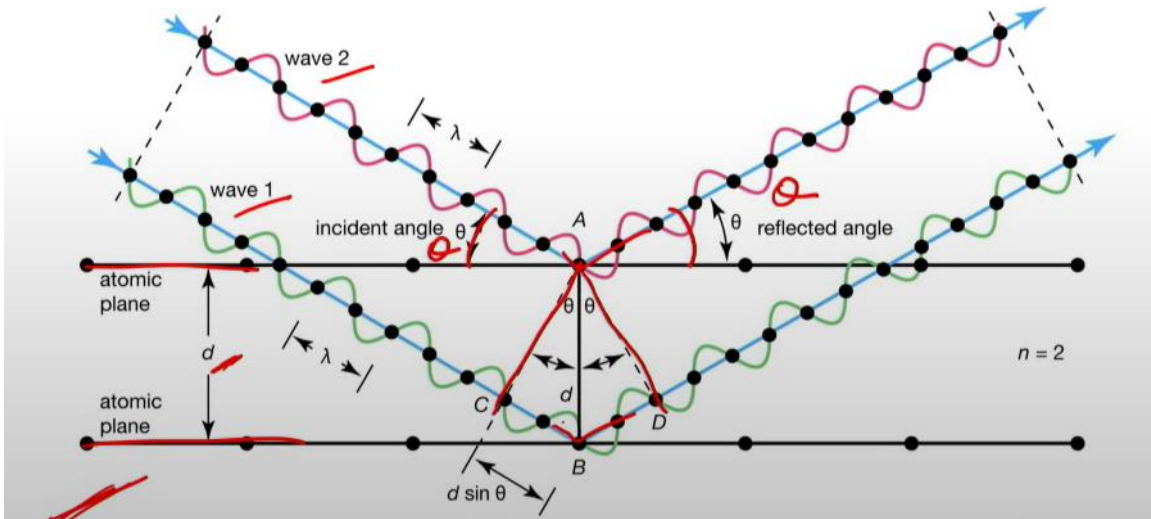
So, this was a quick recap of this. So, how we can utilize this diffraction for our spectroscopy or for our characterization of material. So, what happens is the X-rays that we are using. I discussed this earlier also the wavelength of X-rays is of the order of atomic spacing. Now, what is atomic spacing? So, to understand diffraction and to use it for practical application what happens is we have the primary X-rays, and the wavelength of X-rays is of the order of the atomic spacing. Now, what is atomic spacing? So,

whenever we have the material, they are arranged in a unique form in a unique crystal lattice. So, they have a regular arrangement in the crystal structure and the distance what we call is as planes.

So, we have the distance between these two planes, or all these planes, and we have the regular arrangement of atoms in us inside our material. So, whenever the incident radiation wavelength  $\lambda$  the x-rays is of the order of  $d$  which is the inter atomic the inter planar distance we have the phenomena of diffraction. So, we utilize this phenomenon for our in our X-ray diffraction instrument. So, since the order of the interplanar distance is of the order of the Armstrong's and are the wavelength of X-rays is also of the order of Armstrong's. So, we can utilize the phenomena of diffraction in our XRD spectroscopy.

Now, let us come to the very basics of principle like we want to see. So, if these are the inter atomic planes and these is the inter atomic distance between the two planes. and we have the x-rays which are falling onto our material. So, when it falls simultaneously on both the planes diffraction will occur and based on the diffraction it can interfere constructively or it can interfere destructively. So, as you can see when it falls on a particular angle, so if it is falling on this angle, you can see it is not meeting constructively, but when we have at a particular angle these waves interfere constructively.

So, whenever we have constructive interference, we will get a typical peak in our XRD and whenever there is a destructive interference we do not get a peak in the XRD spectroscopy. So, coming to that we will talk about the very basic law which is there which is called Bragg's law. Now to understand Bragg's law we must see this schematic here in the figure below in which we have the incident X-rays. To understand Bragg's law, we have this schematic here. So, these are us inter atomic planes and  $d$  is the distance between both the planes, and we have this incident x-rays.



So, wave 1, wave 2 are falling on to our sample. So, as you can see they will reflect and then they will have a particular diffraction pattern. to fulfill the diffraction pattern. So, let us suppose it is falling in an angle of theta and since the incident angle is theta this angle will also be theta right to fulfill. So, our diffraction will only occur. So, we can use diffraction only occur whenever they will meet constructively to meet that phenomena to meet that particular thing are Bragg's gave us a formula that our  $n\lambda$  should be equal to  $2d \sin \theta$ .

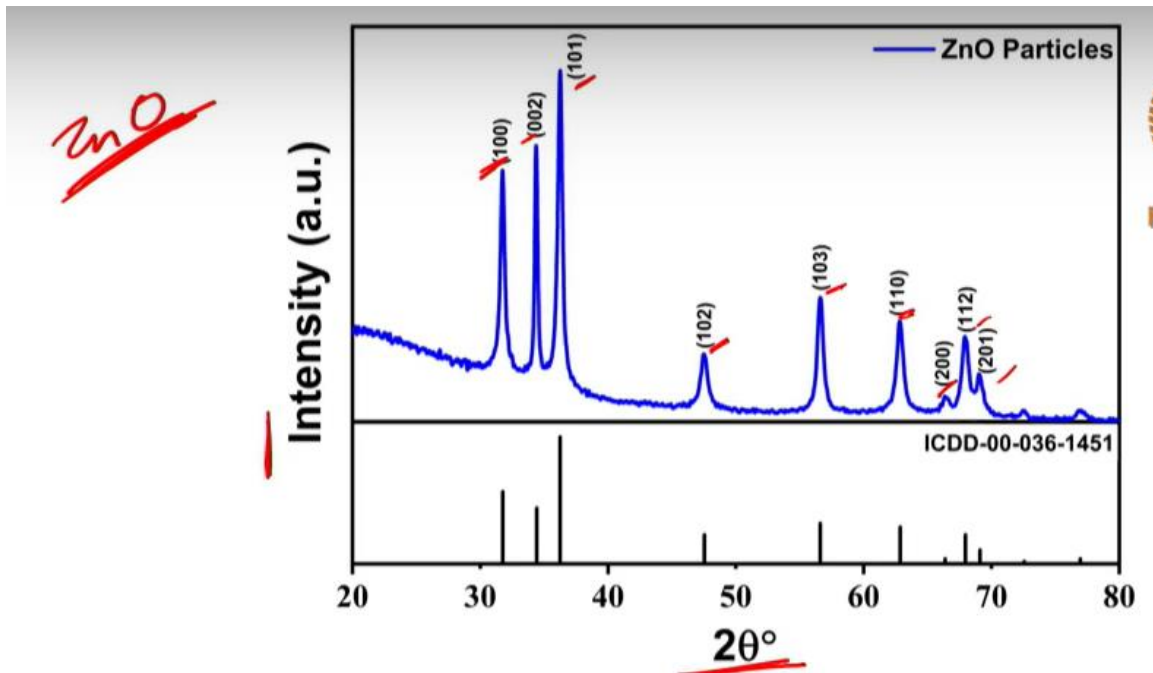
Now, since there are two waves wave 1 and wave 2 wave 1 has to travel this extra distance in order to meet constructively right. So, this is  $d \sin \theta$ , and this is also  $d \sin \theta$ . So, this condition needs to be met whenever the wave meets constructively. So, we have a formula which is given by Bragg which is  $n\lambda = 2d \sin \theta$   $n$  is the integer and  $\lambda$  is the incident wavelength and  $d$  is the inter atomic distance and this condition needs to be met to for the constructive interference to take place. So, talking about the basics of the X-rays for the XRF applications and the XRD applications, let us talk about some of the applications for the X-ray diffraction.

$$\text{Bragg's Law: } n\lambda = 2d \sin \theta$$

Now coming to the X-ray diffraction it can be used to identify whether your material has been successfully synthesized or not this you might have read I have told you in the previous lectures also all the characterization technique help us to identify whether our material has been synthesized or not right from the FTIR from the UV visible from the XRF to the XRD everyone tells us whether the material has been synthesized or not. But the advantages of using XRD is that it helps us to let us know the phase of the material. Now, what is the phase of the material? Supposing, we take the example of zinc oxide again. So, we synthesized. So, we being material scientists have synthesized the ZNO

particles and we want to see whether our material has been synthesized or not or what is the phase of the material.

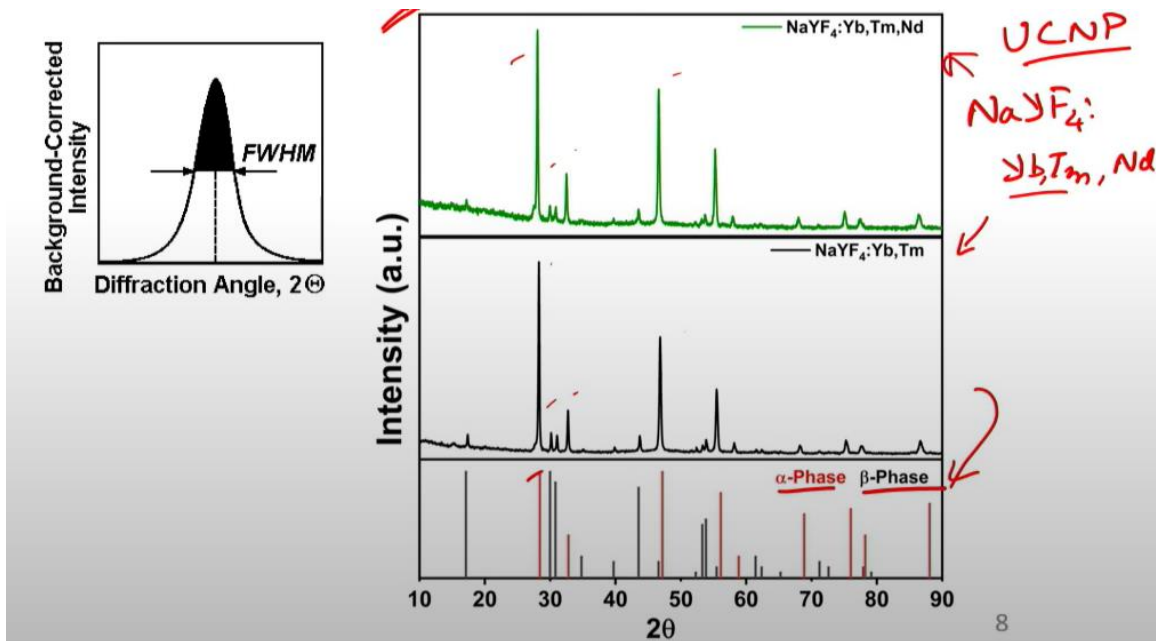
To identify this, we will run it through XRD. So, this is the typical spectra of XRD it is given in  $2\theta$  and the intensity the intensity of the X-rays. So, whenever they meet constructively, we will have these peaks here. So, based on the inter atomic planes. So, we have all these planes that are available like 1 1 0 0 0 2 1 0 1 1 0 2. So, all these planes are present inside our material, and we could find all these planes using our X-ray XRD spectroscopy.



So, supposedly this is a typical spectrum of our ZNO particles. So, what we do is when we synthesize our material, we typically run it through XRD, and we get the XRD pattern of the material and then we have something which is called a reference card like we have a standard ICDD, or you can also say JCPDS. cards. So, these terms you might have heard it. So, we have this standard card for all the materials and based on that we could match it with these cards, and we could identify the peak positions depending on the planes we could match it with these lattice position and we could confirm whether our material has been synthesized or not or what is the phase of the material. So, for example, in this example of zinc oxide the material I have synthesized was pure. So, the phase was pure Wurzite.

So, this was pure, it is a single-phase material, and it was found to be a pure wurzite phase. Now, coming to the example below. So, here is an example of up conversion nano materials we call it in short form as UCNPs. So, we have NaYF<sub>4</sub> material, and we dope it with YbTm terbium, thulium and neodymium.

So, the spectra here is of YBTM doped and the spectra here is of YBTM ND. you could see that a lot of peaks you get a lot of peaks in both these samples while, but after doing the analysis we could say from all the characterization spectroscopy you could say your material has been successfully synthesized right, but when we match it with the phase We found out our material has been successfully synthesized that is fine, but the material is of two phases. So, our material has the alpha phase and the beta phase. So, we could say that our UCNP that we have synthesized are of two phases alpha and beta. So, it is a mixture of both the phases and the zinc oxide material that we synthesized was of pure Wurzite phase.



So, it is a single-phase material. So, this identification of the phase of the material can only be done by the XRD spectroscopy and it is mostly done using our XRD spectroscopy. So, this is the advantage of using our X-ray diffraction and another thing I want to discuss you probably might have noticed is that some peaks are sharp, and some peaks are broad. is peak broadening or what is determined using the sharpness of the peaks. So, what happens is that the sharpness of the peaks is usually determined by the amount of crystallinity in the sample. So, the more crystalline your sample is the sharper will be your peaks and the more amorphous your material is the broader will be your peaks.

So, this is what is called as the peak broadening or the sharpening of the peak. So, another example if I take. So, if you have a polymer. So, polymers are the mixture of amorphous and crystalline materials. So, mostly polymers are amorphous in nature.

So, in case of amorphous material you have something like this. So, the peak that you have in x-ray is broad in nature. So, that determines amount of crystallinity, or you could comment on the amount of amorphous present in the sample. So, our polymers are usually amorphous. So, you get a broad peak while from the nanomaterial that you synthesize you get some sharp peaks, but are still broad, but if you go towards the bulk sample these broad peaks are something like this.

So, it is in the form of line structure. So, the more towards the bulk side we will get a sharp peak and if we go towards the amorphosity side. and we get a something like this peak which is hump kind of structure. So, another thing that we could use the X-rays the XRD is for the determination of the atomic arrangements and calculation of the crystallite size. Now, what is crystallite size? As I already talked about there are some crystals that are found in the materials. So, based on those crystals we have the atomic arrangements, and we could calculate the crystallite size of the material using a formula that was given by Scherer.

So, Scherer gave this formula where D is equal to  $0.9 \lambda$  by  $\beta \cos \theta$ .

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

Now our  $\lambda$  is the wavelength of the x-rays, our  $\theta$  is the incident angle, and the  $\beta$  is the full width half maxima. Now what is the full width half maxima? of the maximum intense peak that we say. So, in this case we have the maximum intense peak as of the plane 101 and here in case of UCNP we have this peak at around 30 degrees. So, this peak we will take and. this while this is the width of that peak, and this is the intensity of that peak FWHM will be somewhere here which is the full width and half maxima of that peak that we will take it here and then we can calculate the d which is our crystallite size.

So, if you go through a lot of literature, you will find terms like crystallinity or the crystallite size or the phases determination. Everything I discussed here will be useful for you whenever you read a research paper in which a lot of characterization of that material has been done. So, these are useful whenever you go through your literature survey. Another thing that X-rays can be used is the detection of impurities. So, as I talked about earlier, we get a typical XRD spectra which is data which is represented for that particular material, and we have the JCPDS card which is literature card for that particular material and supposedly you have.

During the synthesis you used a bad spatula, or you used a bad magnetic bead and because of which you have some contaminants, and you have some impurities in your sample. And whenever you run it through XRD you get some additional peaks here or

some additional peaks here. extra peaks you could again refer to the JCPDS card and you could find out what these peaks represent and based on that you could say that impurities are present in your sample and what are those particular impurities. Supposing it is for copper or suppose magnesium or some other metal.

So, all the impurities can be determined using our XRD spectroscopy as well. So, talking about all these applications. So, we will talk about the summary, we will summarize what we learned today. So, what we learned today was the basics of X-rays. So, what are X-rays and how they can be used for two different phenomena which is fluorescence and diffraction. So, we have studied the very basics of XRF, the basic working mechanism and how they can be used for the quantification and quantification of unknown samples. And then we talked about the basic principles of diffraction, what diffraction is and what are interference and how we could utilize the diffraction phenomena for our characterization and how we could use it in our XRD.

Then we talked about the very basic principle and the mechanism and the working of the XRD as well. And we talked about some of the applications for XRD like identification of crystallite crystal phases calculation of crystallite size, then identification of the planes and then identification of the impurities that are there in planes. So, that summarizes our X-rays. So, how we could use our X-rays for the characterization of materials. So, for the after this. So, this summarizes everything that we learned so far how we can use the UV visible X-rays and the infrared spectroscopy for the characterization of materials.

So, this concludes our spectroscopy part. So, I hope this was really helpful for you and you learnt a lot about spectroscopy in this, how you could use infrared, UV visible and the X-rays for the characterization of material. So, in case you have any doubts you could reach us, and I hope this was helpful. Thank you very much and see you in the next lecture.