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LECTURE-11
Introduction to X-Ray Diffraction
With
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Hello, in our previous lecture, so you have already discuss about the crystal structures inter atomic bonding, about the metals, there are make polymers and some kind of advance materials, today actually, we are going to discuss about the introduction to X-Ray diffraction, so before going to start about the X-Ray diffraction let us know, what are X-Rays? So X-Rays are the,

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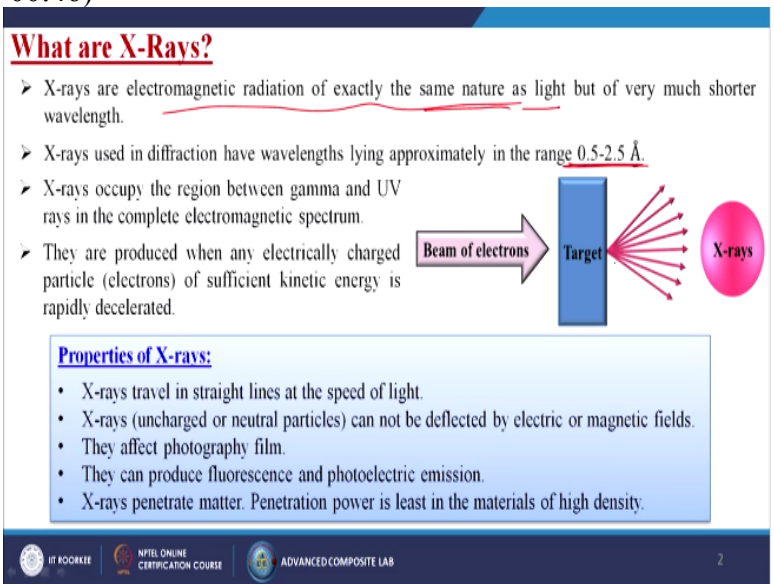

Electromagnetic radiation of exactly the same nature, as like, but of very much short or wave length, X Rays used in diffraction have wave lengths lying approximately in the range of 0.5-2.5 angstrom, X-Rays occupy by the region between γ and UV rays in the complete electromagnetic spectrum, that I will show you later.

They are produced in any electrically charged particles like, electrons of sufficient kinetic energy is rapidly de-accelerated so, here the beam of electrons is coming, hitting the target and then, from that X-Ray is generating so, what are the properties of X-Ray? First X-ray is travel in straight lines at the speed of light.

X-Rays uncharged or neutral particles cannot be deflected by any electric or maybe the magnetic fields, they affect the photography film, then they can produce fluorescence and the photoelectric emission and also they can penetrate the matter, penetration power is least in the materials of higher density. So, this known as the electromagnetic spectrum, so left hand side top is known as the,

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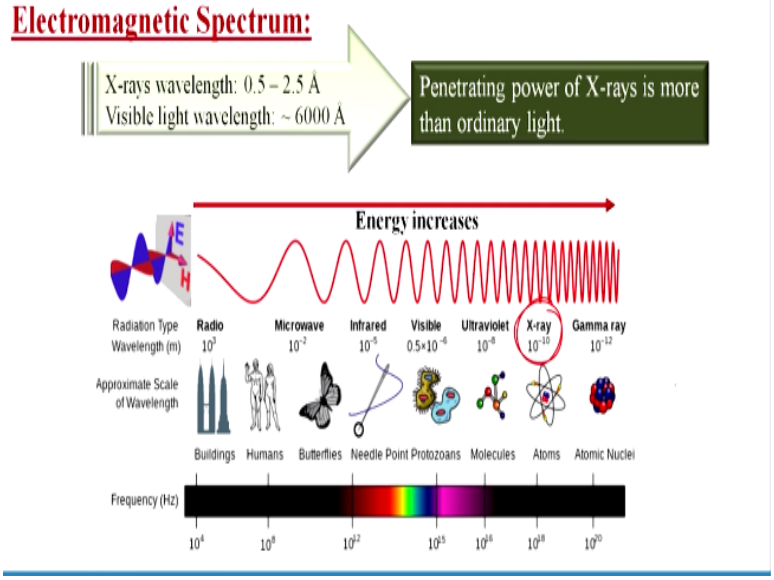

X-Ray wave length, which is nothing but 0.5- to 2.5 angstrom, which I have already told generally, the visible light wavelength is 6000 angstrom, so penetrating power of X-Ray is more than the ordinary light, and if we see carefully the electromagnetic spectrum, so we can find the X-Ray is lying over here, so X-Ray the radiation type wavelength is 10^{-10} meter and these way the energy is increasing. So, after the X-Ray, it is already founded by γ ray.

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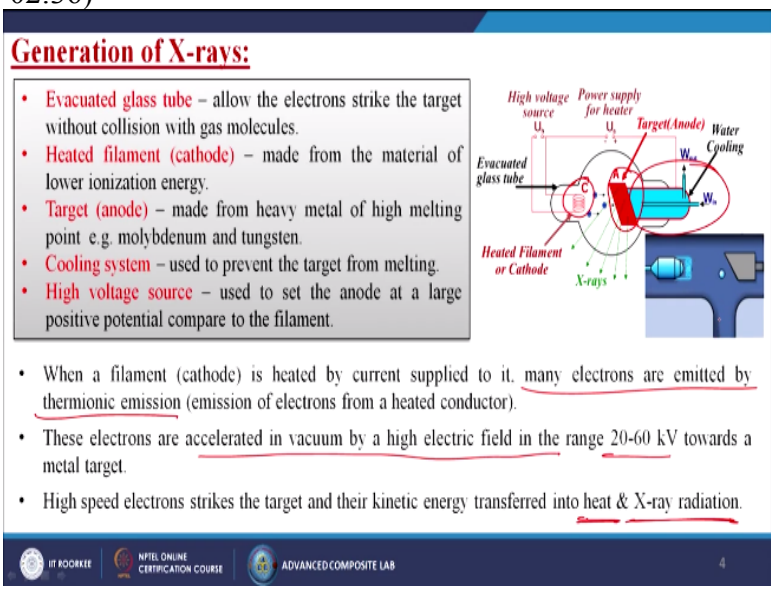

So, now we are going to discuss about the generations of X-Ray, so X-Ray is generally, it is why we are doing it in to some paste chambers, so first we are talking about the evacuated glass tube, which allow the electrons strike, the target without the collision with the gas molecules. So right hand side picture, you can see, so this is known as the black in color is known as the evacuated glass tube.

Inside that glass tube you can see that heated filament over there, the target is there, and some water cooling systems are there, so, how it is actually occurring, so first heated filament which is nothing but known as the cathode, made from the material of lower ionization energy, so this is

known as the cathode over there, so now we are having that target or, it is sometimes it is called the anode also, anode material makes from heavy metal of high melting point like, molygeram or maybe the tungsten, So this is our anode materials.

Then, we are having some cooling systems, cooling systems because when, the X-Rays generating the heat as well as the X-Ray ray, both are generating, so that anode materials should not be melt, so that is why? We are using some kind of cooling arrangement over there, so that anode materials should not melt. Now we are having another high voltage source, which is nothing but used to set that anode at a large positive potential compare to the filament.

So that, all the electrons should be attracted towards the anode, so when a filament, it is heated by current supply to heat many electrons are emitted by thermionic emissions, which means emission of electrons from a heated conductor. These electrons are accelerated in vacuum by a high electric field in the range of 20-60 kilo volt, towards the metal target, high speed electrons strike the target and their kinetic energy transferred in to heat energy and the X-Ray radiation. So, this is the simple image, that through that the electron, it is generating, then it is hitting your target materials and through that the X-Ray formation is taking place.

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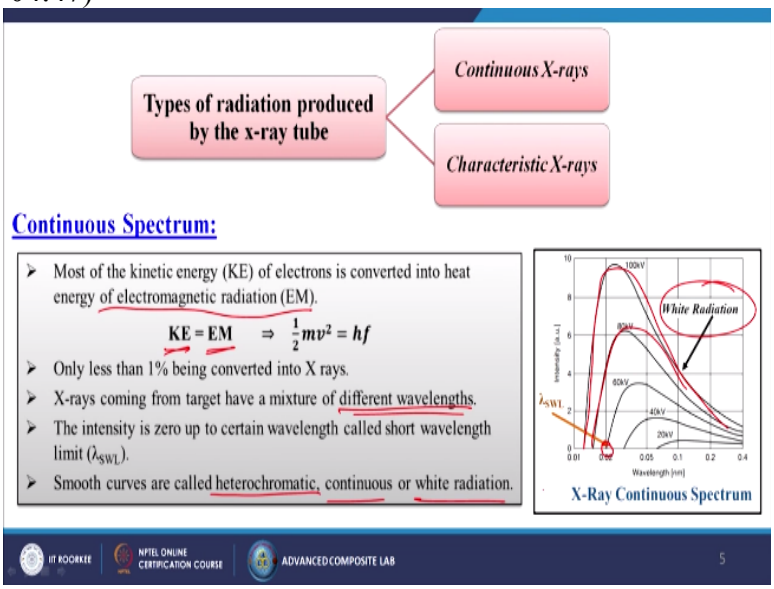

So, now there are actually two types of radiations by the X-Ray tube, one is called the continuous X-ray and another one is called the characteristic X-Rays, so what is known as the continuous spectrum? Generally the continue spectrum taken care by the heat energy, so most of the kinetic energy of electrons is combining in to the heat energy or electromagnetic radiation, which is nothing but the kinetic energy is equivalent to electromagnetic radiations, which is nothing but the 1/2 mv^2= hf so, here only less than 1% being converted in to the X-Rays, that means X-Ray accept 1%, this 99% is converting in to the heat energy.

So, X-Ray is coming from the target having mixture of different wavelengths, the intensity is zero up to certain wavelengths called the short wavelength limit, which is denoted by generally the (λswl), smooth curves are also called the heterochromatic, continuous, or maybe the white radiations, so from this particular figure, you can see that we are getting some smooth curve over

there, so which is nothing but known as the white radiations.And these points actually it is known as the short wavelength, so the intensity in these particular points is zero.

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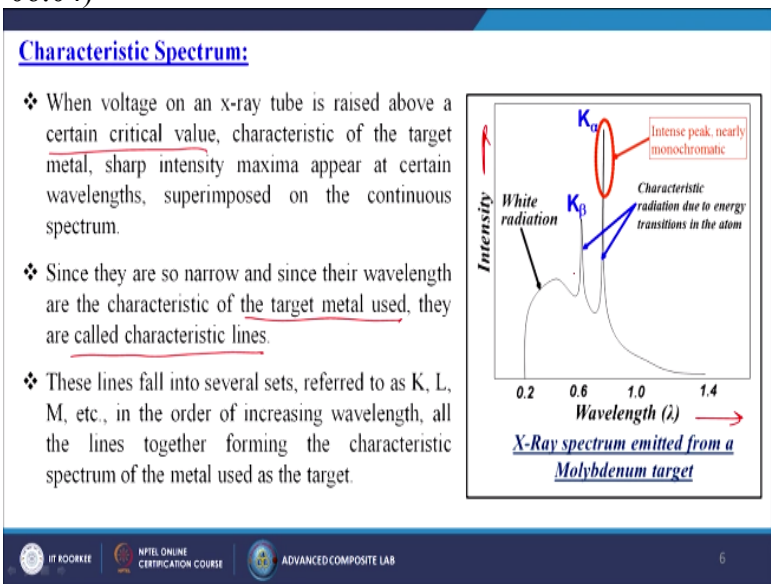

Now, will discuss about the characteristics spectrum, so what do you mean by characteristic spectrum? So, when voltage on an X-Ray tube is raised above a certain critical value, characteristics of the target metal, sharp intensity maxima appear at certain wavelengths, super imposed on the continuous spectrum, since they are so narrow, and since their wavelength are the characteristics of the target metal used, they are also called as the characteristic lines.

These lines fall in to several sets, referred to as K.L.M, this actually are all the shells in the order to of increasing the wavelength, all the lines together forming the characteristic spectrum of the metal used as the target. So, actually after doing that one, whatever the characteristic peaks are coming, maybe the characteristic peaks forming their actually super imposing in to our white radiations or maybe that normal peak. So, in this particular case if you see this particular graph you can find out that X axis is denoting the wavelength, and Y axis is giving in the intensity and these shown in the continuous line is known as the white radiations.

And then, after that you are getting some kind of characteristic radiations like, K β, K α, so here you can find some peaks are brought in, some peaks are so sharp, so these are all known as the characteristic peak.

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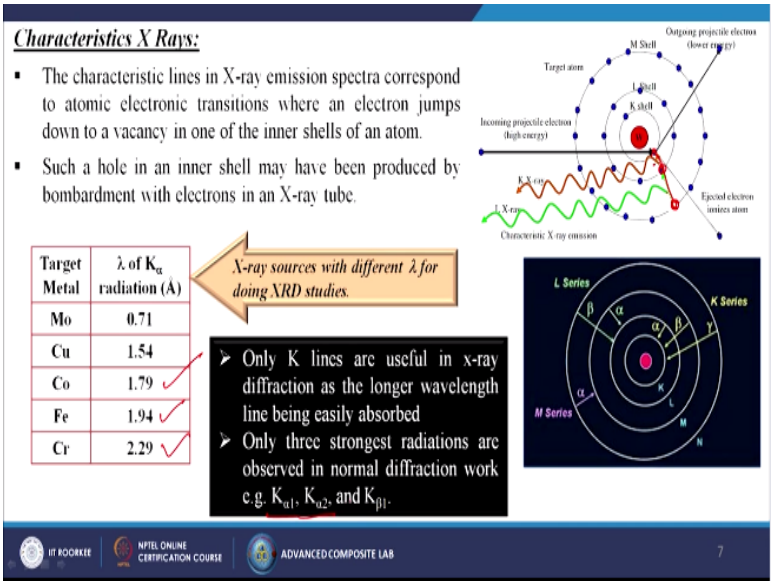

Now, characteristics X-Rays, the characteristic line in X-Rays emission spectra correspond to atomic electronic transitions, where an electron jumps down to a vacancy in one of the inner shells of an atom, so from this particular case you can see initially it has been started with the M shell, then it is jumping to the L shell, and then it is jumping to the K shell itself, so the electron is jumping from one shell to another, such a whole in a inner shell, may have been produce by bombardment to the electrons in an X-Ray tube, so when the electrons is jumping from one shell to another shell so, generally that emission of the X-Ray is taking place, but generally we pay for that electron is jumping in the inner shell so, that intensity will be more, so now X-Ray source also with different λ, doing for doing XRD studies, so here depends upon the different target metals. If we talk about the molygeram, so λ for K α radiations 0.71, for copper it is 1.54, for cobalt is 1.79, for Irion it is 1.94, and for chromium, it is 2.29

So, only as I told you already only K lines are useful in extra diffraction as the longer wavelength lying being easily absorbed, so generally we pay for electron should jump from K shell to the L shell only three strongest radiations are observed in normal diffraction like, K^1 α, K^2 α , and K^1 β.

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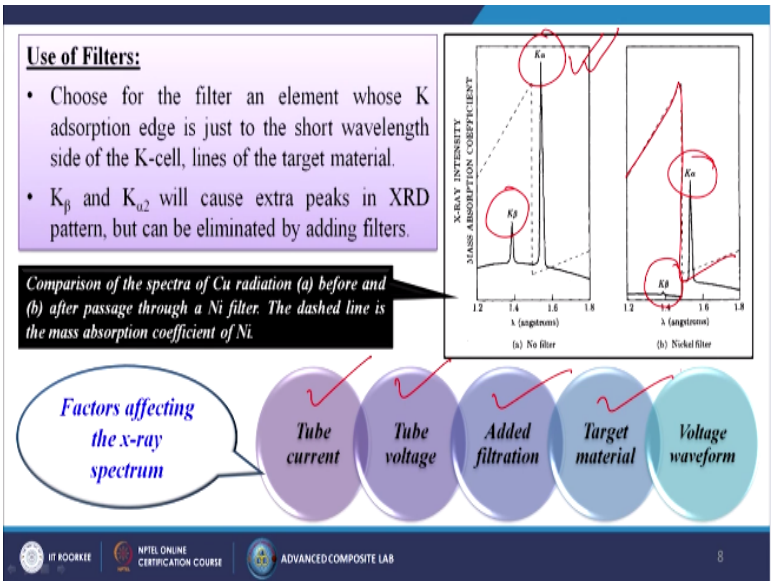

Use of filters, so now we will discuss about the filters so in this particular case actually what happen? We are getting so many peaks over there, but some peaks are due to that temperature, some peaks is due to the material characteristic, some peaks is due to the different radiations. So, now actually we are using certain kind of filters, to get that actual crystal structures or maybe the actual crystaling formulations of that particular materials.

So choose for the filter and element, whose K adsorption edge is just to the short wavelength side of the K-cell lines of the target materials, so that it can eliminate the other sources, generally K β and α^2, will cause extra peaks in XRD pattern, but can be eliminated by adding filters. So that is why? We are adding filters, just to restrict the other spectra over there, so in this particular figure you can see that why? We are using No filter, so we are getting K β as well as the K α, but in this particular case the K α is required for us.

So, that is why? We are using certain kind of nickel filter, due to that K β radiation as been surprised and we are getting only the K α radiations, so comparison of this spectra of copper radiations, before and after passage through, a nickel filter, the dashed line is the mass absorption coefficient of the nickels, so this is the mass absorption coefficient of the nickel filter.

So, which are the factors, which affects the X-Rays spectrum, first one is called tube current then, tube voltage, added filtration, target material and the voltage waveform. Now, let us come to the actual point that why? X-Rays are used in crystallography, so for the electro magnetic

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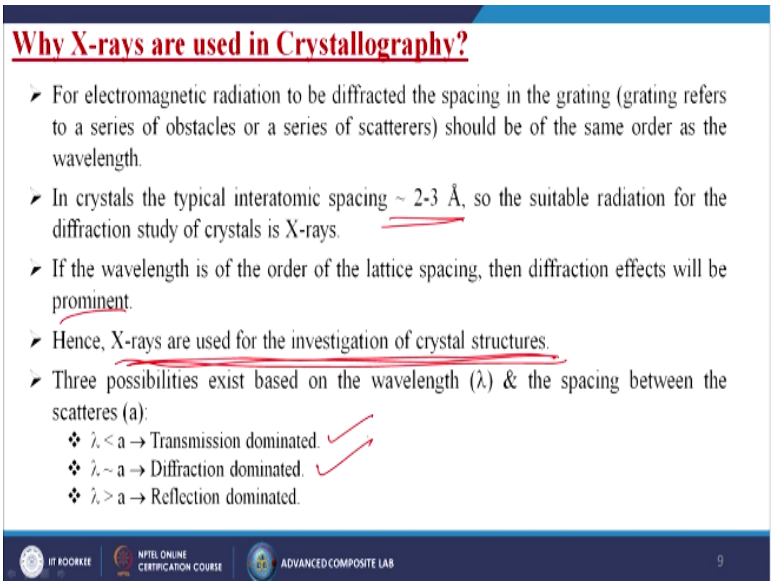

Radiation to be diffracted the spacing in the grating refers to a series of obstacles or a series of scatters, should be of the same order as the wavelength, so that X-Ray can easily pass through that gap, in crystals the typical interatomic spacing, generally it is 2-3 Armstrong. So, the suitable radiation for the diffraction study of crystals is X-Rays. If the wavelength is the order of the lattice spacing, then the diffraction effects will be prominent, hence X-Rays are used for the investigation of the crystal structures.

So, nowadays for any characterization of about that crystal structures for any known materials or maybe the unknown samples or maybe the unknown materials, the X-Ray diffraction study is the vital one, three possibilities exist best on the wavelength λ, and the spacing between the scatters (a), like if λ, is the (a), the transmission dominated, if λ is equivalent to (a), the diffraction is dominated, and if λ is greater than (a), then reflection dominated.

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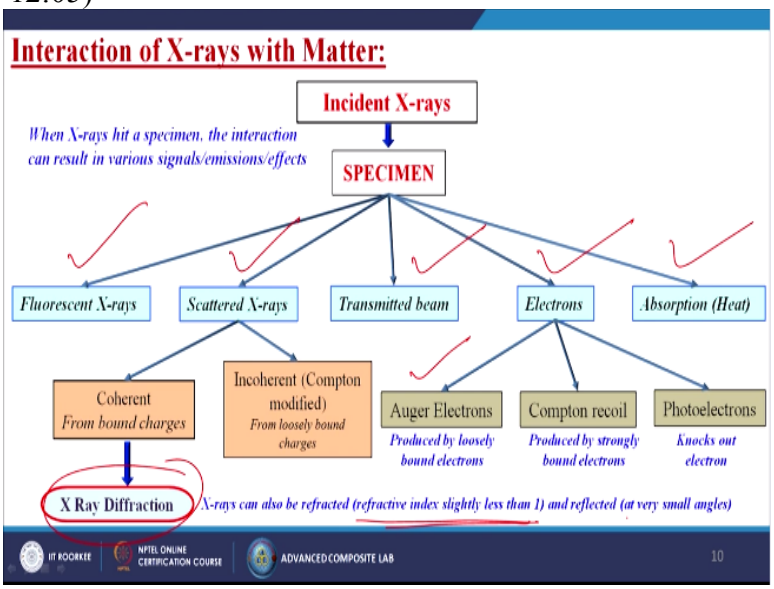

Now, we are going to discuss about the interaction of X-rays with the matter, means the materials, so first I already have told you that incident rays it is coming through the specimens

and then, generally it is divided in to five parts, one is called the fluorescent X-rays, then scattered X-rays, transmitted beam, electrons, and absorption or maybe the heat, So scattered X-rays are also is divided in to two parts one is called the coherent from bound charges, so which is nothing but known as the XRD study or maybe the X-ray diffraction and another one is called the incoherent or maybe the Compton modified from loosely bound charges.

And if we talk about the electrons, the electrons are also divided in to three parts, one is called the auger electrons, produced by loosely bound electrons, another one is called Compton recoil produced by strongly bound electrons, another one is called the photoelectrons, which is nothing but the knocks out electrons, so generally X-ray can also be refracted index slightly less than 1 and reflected at very small angles.

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X-ray sources with different λ for doing XRD studies:

Elements	(KV)	λ Of $K_{\alpha 1}$ radiation (Å)	λ Of $K_{\alpha 2}$ radiation (Å)	λ Of K_{β} radiation (Å)	K_{β} Filter (mm)
Ag	25.52	0.55941	0.5638	0.49707	Pd 0.0461
Mo	20	0.7093	0.71359	0.63229	Zr 0.0678
Cu	8.98	1.540598	1.54439	1.39222	Ni 0.017
Ni	8.33	1.65791	1.66175	1.50014	Co 0.0158
Co	7.71	1.78897	1.79285	1.62079	Fe 0.0166
Fe	7.11	1.93604	1.93998	1.75661	Mn 0.0168
Cr	5.99	2.2897	2.29361	2.08487	V 0.169

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Now, we are going to discuss about the different X-rays sources with the different λ, for doing the certain studies, so generally we are using the copper as it is, because it is widely used, so sometimes we are using the silver or molygeram or maybe the cobalt also, so here you can see that what is the energy value? And what is the λ value? For K^1 α , K^2 α, and K and K β, and K β filter, means after using the filter that work the value with generally we are subtracting from it.

So if we talk about the copper because am giving the example copper because it is widely used.

So, generally the λ, K^1 α, radiation is 1.540598, and λ K^2 α radiation is 1.54439 and λ K β radiation is 1.39222, so generally here we are using the nickel filter, whose value is 0.017, so, generally that 0.017 value has been subtracted from the λ value and all we are getting the copper K α value and which is, we are using for our X-ray diffraction studies.

Now, let us discuss about that diffraction, what is diffraction? So, diffraction refers to,

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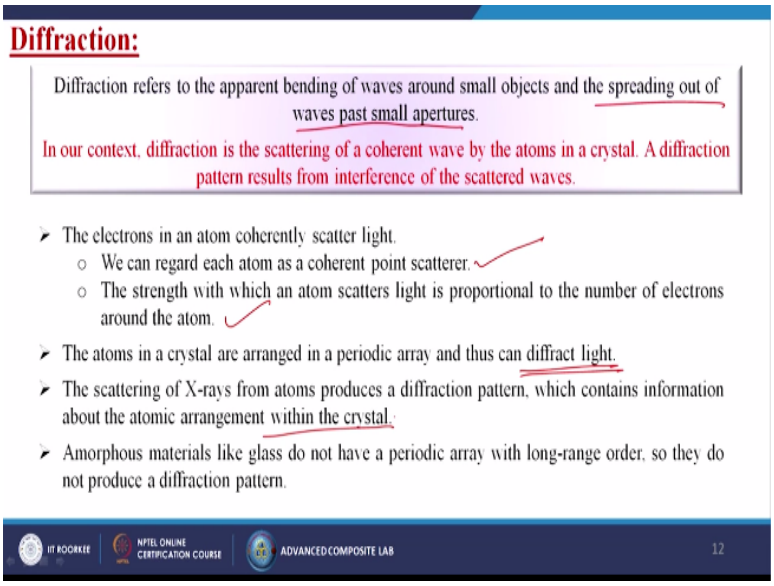

The apparent bending of waves around small objects and the spreading out of waves past small apertures, in our context diffraction is the scattering of a coherent wave by the atoms in a crystal, a diffraction pattern results from interference of the scattered waves. So actually the thing is that when something it is hitting on to the materials, the materials actually it is giving some coherent wave, from by the atoms, when there is in a particular crystal structure, so the electrons in an atom coherently scatter light, when we can regard each atom as a coherent point scatter, the strength with which an atom scatters light is proportional to the number of electrons around the atom.

Another point is that the atom in a crystal are arranged in a periodic array, and thus can defect the light, the scattering of the X-rays from atoms produces diffraction pattern which contains the information about the atomic arrangement within the crystal, amorphous materials like, glass do not have a periodic array with long-range order, so they do not produce a diffraction pattern. Now, what is the basic of the X-ray diffraction? So, X-ray diffractions

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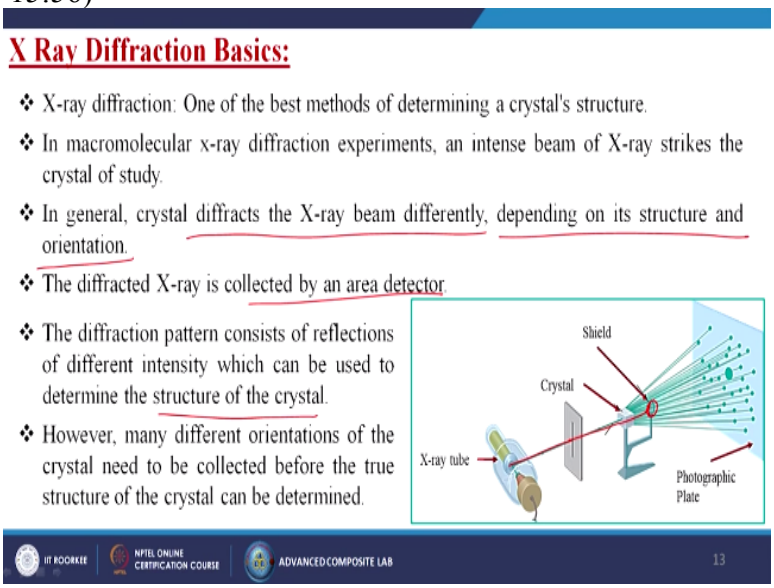

First one of the best methods of determining a crystal structure which I have already told you, in macromolecular X-ray diffraction experiments, an intense beam of X-ray strikes the crystal of study, in general crystal diffracts the X-ray beam differently depending on its structure and orientation, yes off course, because there is from material to material and about their atoms. The diffracted X-ray is collected by an area detector; the diffraction pattern consists of reflections of different intensity, which can be used to determine the structure of the crystal.

However, many different orientations of the crystal need to be collected before the true structure of the crystal can be determined, because in a material maybe if it is composite or maybe some other materials or maybe some impurities can be present, so there are four many types of elements can be present and all the elements is having different diffraction pattern. So from right hand side image you can see from the X-ray due to, the X-ray has been generated and then, it has directly fallen on to the crystal or maybe the material.

And then, after the diffraction it is having some shielding arrangements through that shielding directly is going on to the photographic plate and it is giving in to some dot dot structure by which, we can get the crystal structure of that particular materials. Now we are going to discuss about the diffraction phenomenal, so generally diffraction is consequence of,

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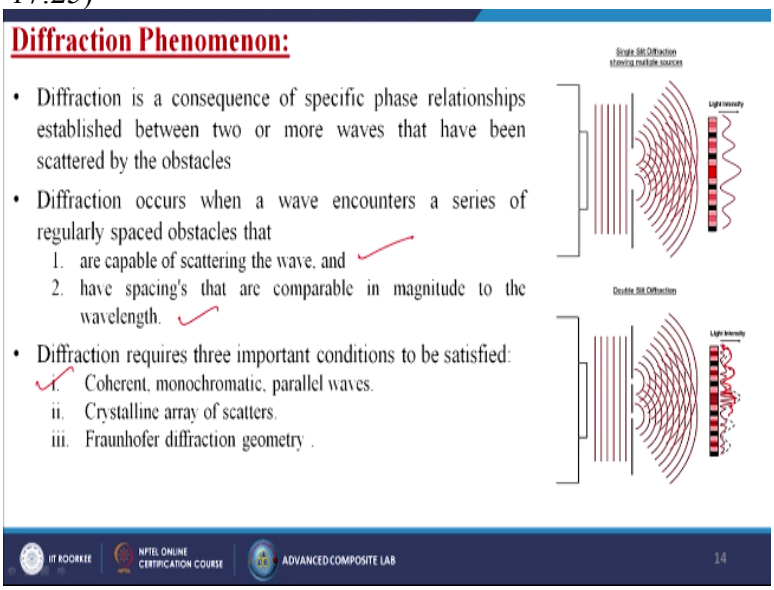

Specific phase relationships established between two or more waves, that have been scattered by the obstacles, maybe sometimes they can be together or sometimes they can be over late so, diffraction occurs when a wave encounters a series of regularly spaced obstacles that, are capable of scattering the wave and have spacing‘s that are comparable in magnitude to the wavelength. Diffraction requires three important conditions to be satisfied, first one is known as the coherent monochromatic and the parallel waves, second is that, crystalline array of scatters and third one is called the fraunhofer diffraction geometry, so in right side also you can see that why? We are talking about the single slit diffraction.

So, here it is showing the multiple sources but it is going in to in a single wave, but when you are talking about that, double slit diffractions. So, you can see that the light wave when it is falling

on to the materials and then, after that all the signals it is actually they are mixing all together but for the single case you can get only the single signal over there.
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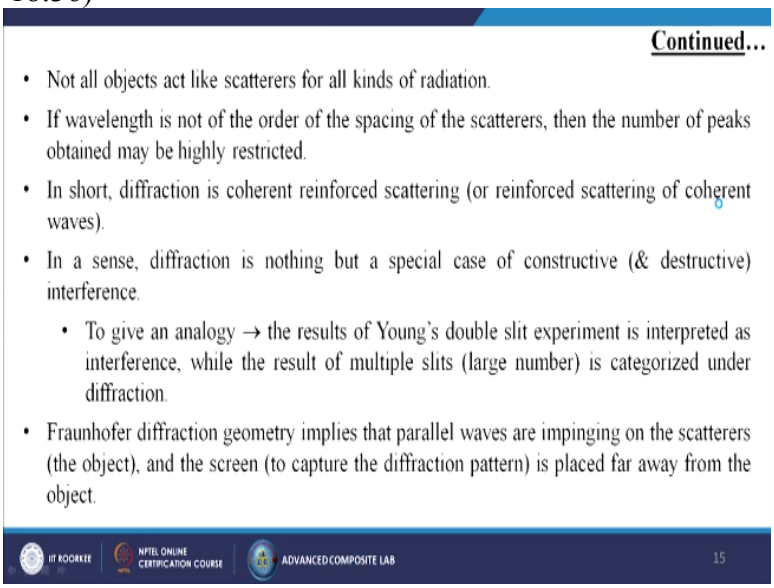

Not all objects like, scatters for all kinds of radiations, if wavelength is not of the order of the spacing of the scatters, then the number of peaks obtained maybe highly restricted, in short diffraction is coherent reinforced scattering or reinforced scattering of coherent waves. In a sense diffraction is nothing but a special case of constructive or maybe destructive interference, to give an analogy the results of young‘s double slit experiment is interpreted as interference, while the result of multiples slits, like large number is categorized under diffraction.
Fraunhofer diffraction what is telling about? fraunhofer diffraction geometry implies that parallel waves are impinging on the scatters means, the object and the screen to capture the diffraction pattern is placed far away from the object, so the distance is more in this particular case .Now, we are talking about the effect of the part difference, so first we will discuss about the optical interference, so difference in the length
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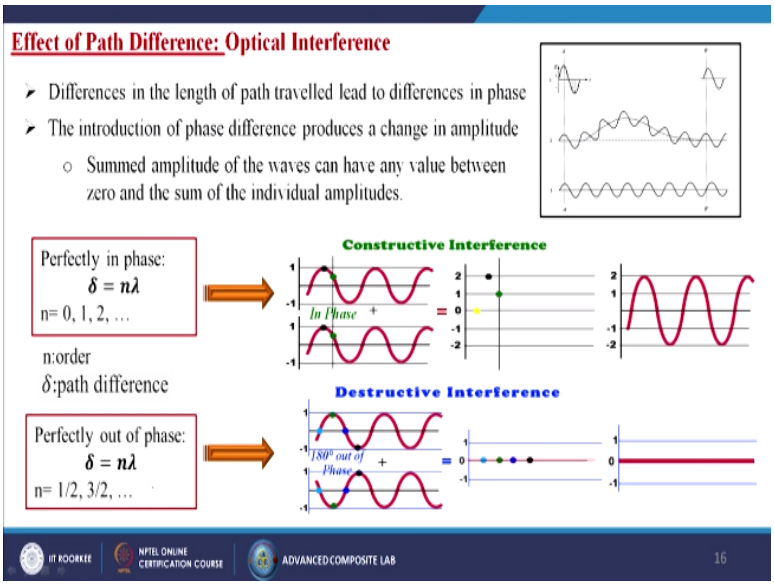

Of path travelled lead to differences in phase, the introduction of phase difference produce a change in amplitude, summed amplitude of the waves can have any value between zero and the sum of the individual amplitudes, so when we are talking about the constructive interference so, in this particular case, you can see that in this particular phase, generally the aim value is coming around 0 1 or 2, that means is, nothing but the order and delta is the path difference, so in this particular case you can see that, we are getting certain value in o difference phases.

But, why we are talking about the destructive interference, you can see the value is allow is 0, over there, so perfectly out of phase it is out of the phase so, that is why it is known as the destructive interference, in value is,1/2 or 3/2 or maybe so on.

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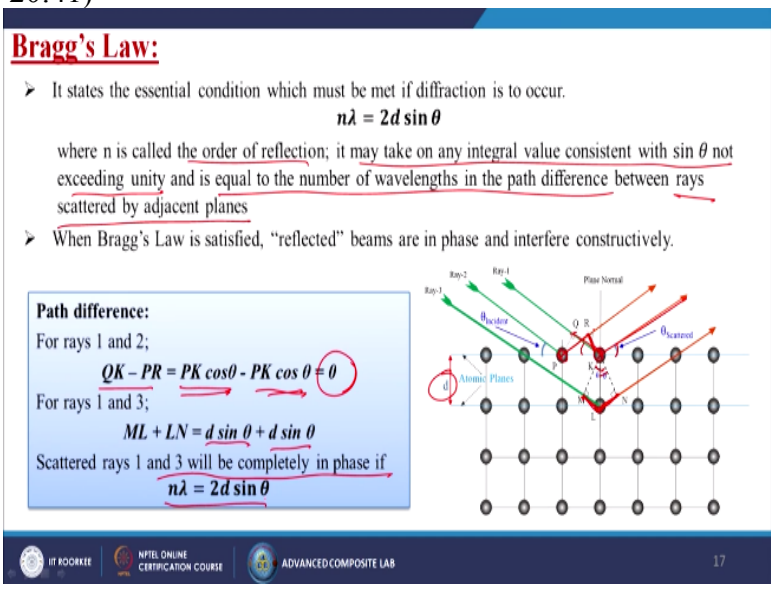

Now, we are going to discuss about the Bragg's law, it is the law has been discovered by the famous scientist and this law is very very essential to major the interact atom distance of any materials or maybe any other unknown samples, so their famous law is known as the Bragg's law it states the essential conditions, which is must may if diffraction is through occur. So what is

that law? Which is nothing but the M λ,=2d sin θ, so where in its called the order of reflection it may take on any integral value consistence with sin θ, not exceeding the unity and is equal to the number of wavelengths in the path difference between the rays and scattered by the adjacent planes.

So, when Bragg's law is satisfied, reflected beams are in phase and interfere constructively, so that that means we can get the results, so right hand side image you can see that when the rays are coming because, rays is not a single one, there are so many rays are coming together so, when we are talking about the ray1 it is falling in this particular point and then it is reflecting in this directions. So, when it is falling it is creating some angle θ over there, and with same angle also it is going back, after scattering, then when we are talking about ray 2, it is falling over here, but you can see, when you are talking about the ray 3, ray 1 and ray 3, plane is same but when, we are talking about the ray 1 and ray 2, the planes are different.

So now, how to calculate the path difference over there, so, when you are try to calculate the path difference of ray 1, and ray 2, from this you can see, the ray 1 is coming at a θ angle and it is going back also in to the θ angle, so if we put a perpendicular line from this P point, so this is known as the Q point over there, so, we are doing the Q, now when we are talking about the ray 2, from this K point.

We are trying making a perpendicular R, so what will be the path difference over there, for ray 1 and 2, the path difference will be QK, Q and K over here, and –PR, so what is the QK? QK is nothing but the PK cost eta, and what is PR, PR also nothing but the PK cost eta, because this is the P and this is the K, so angle is θ so, what is the value over there, it is 0, but when we are talking about the rays 1and 3, because they are in to the same plane.

So, how we are getting, when the rays are coming over here, same thing we are doing, we are making the perpendicular from the K point, on to your rays, so here ML+LN, so here the distance in between the K and L is small d, which is nothing but the interatomic distance. So in this particular case how we are trying to calculate, these angle also θ and these angle also θ, so, now we have to calculate the ML+LN, so ML is what? Which is nothing but the sin θ, and what is LN, which is also nothing but the d sin θ.

So, ML+LN, is nothing but the 2d, sinθ, so scattered rays 1 and 3, will be completely in phase, if n λ=2d sin θ.

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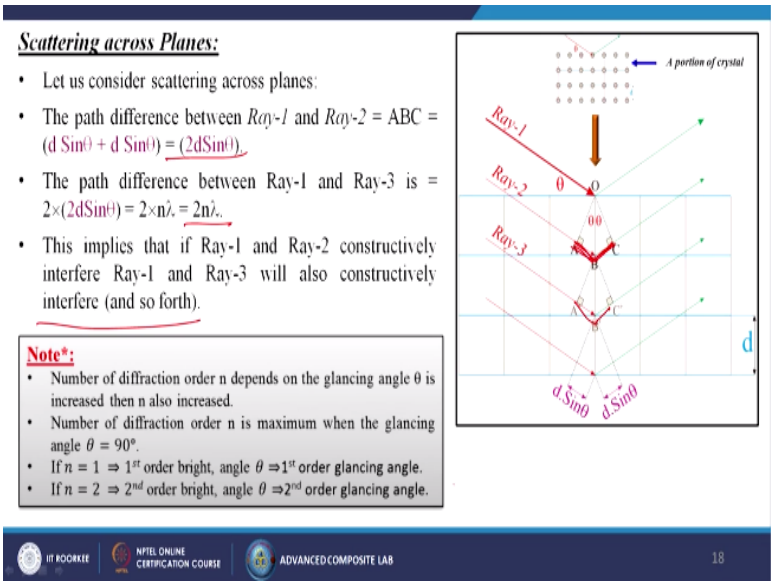

So, now we are going to discuss about the scattering across the planes, so here first let us consider as the scattering across the planes, in this particular case you can see that we, are having different planes over there, so the path difference is between the ray 1, and ray 2, which is nothing but the ABC, means AB+BC, which we have already discuss that, AB is d sin θ and BC also sin θ, so it is called the 2d sin θ.

So, the path difference between ray 1, and ray 3, so it will be this 1+1N, this 1, that means, 2d sin θ in multiplication by twice, that is nothing but the 2nλ, or maybe 2nλ, so these in plus, that if ray 1, and ray 2, constructively interfere rays 1 and rays 3, will also constructively interfere that has been proved, so what are the notes over here, the number of diffraction order in depends on the glancing angle θ is increased, and then N also increased.

Number of diffraction order N is maximum, when the glancing angle θ=90°, if N=1, that means, first order is bright, that means, angle θ is also means the first order of the glancing angle, if N=2, that means the second order is bright, or maybe angle θ is also meaning the second order of glancing angle.

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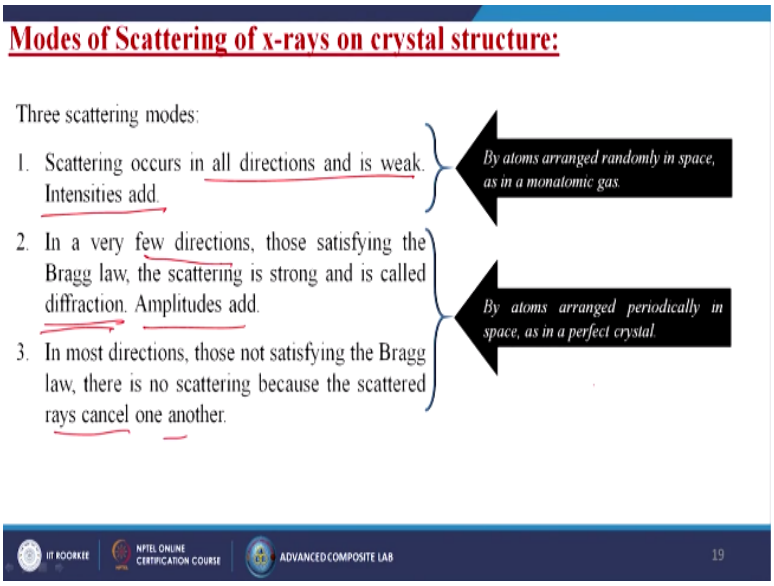

Now, we are going to discuss about the modes of scattering of X rays on the crystal structure, so there are three scattering modes, so first one is called scattering occurs in all directions and is weak intensities add, which is nothing but by atoms arranged randomly in space, as in a monatomic gas, then we are talking about the second and the third point, what the second point says, it says in a very few directions, those satisfying the Bragg's law the scattering is strong and is called the diffraction amplitudes add.

And third in most directions, those not satisfying the Bragg's law, there is no scattering because of the scattered rays cancel one another, so by atoms arranged periodically in space, as in a perfect crystal, now we are going to discuss about the difference between the diffraction and the reflection, so diffraction means,

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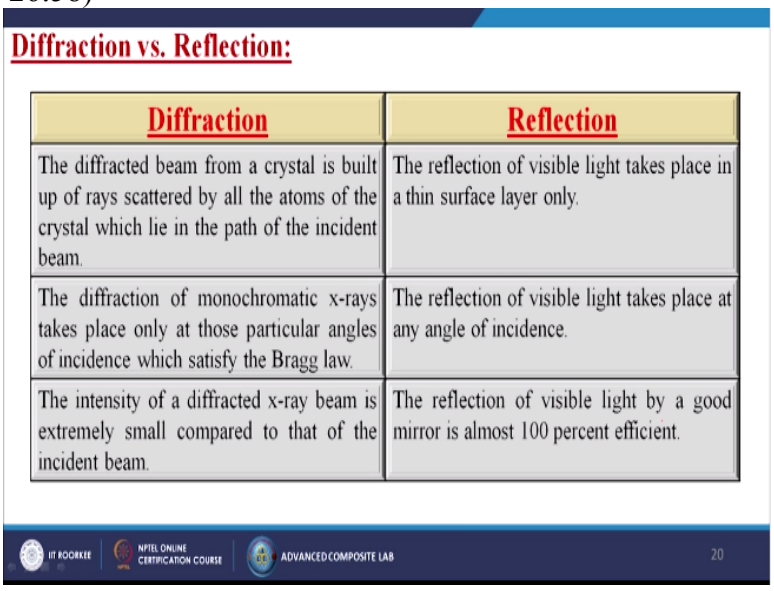

The diffracted beam from a crystal is built up of rays scattered by all the atoms of the crystal which lie in the path of the incident beam, where as the reflection of visible light takes place in a thin surface layer only. The diffraction of monochromatic X-rays takes place only at those

particular angles of incidence, which satisfy the Bragg's law, the reflection of visible light takes place at any angle of incidence, the intensity of a diffracted X-ray beam is extremely small compared to that of the incident beam, where as the reflection of visible light by a good mirror is almost 100 percent efficient.

Now, what are the conditions for the diffractions, so first as previously already we have discuss, diffraction

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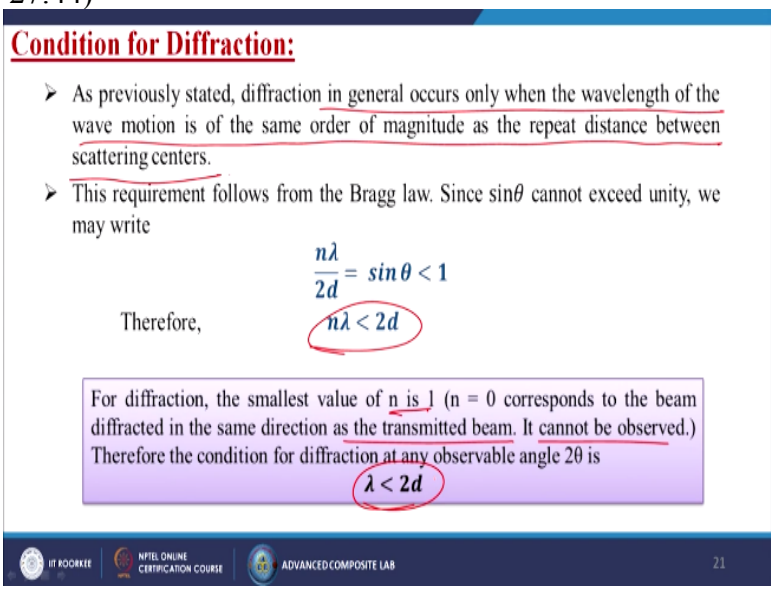

In general occurs only with wavelength of the wave motion is of the same order of magnitude as the repeat distance between the scattering centers, this is the first point, second is that, this requirement follows from the Bragg's law, since sin θ cannot exceed unity, we may write Nλ/2d is = sin θ, which is <1, they are for Nλ, should be allow <2d value, d is nothing but the inter atom with the distance

So, for diffraction the smallest value of N is 1, some times N=0, correspond to the beam diffracted in the same direction, as the transmitted beam, it cannot be observed, therefore the condition for diffraction at any observable angle 2 θ is λ, is allow is <2d, now what are the applications of the Bragg's law, experimentally.

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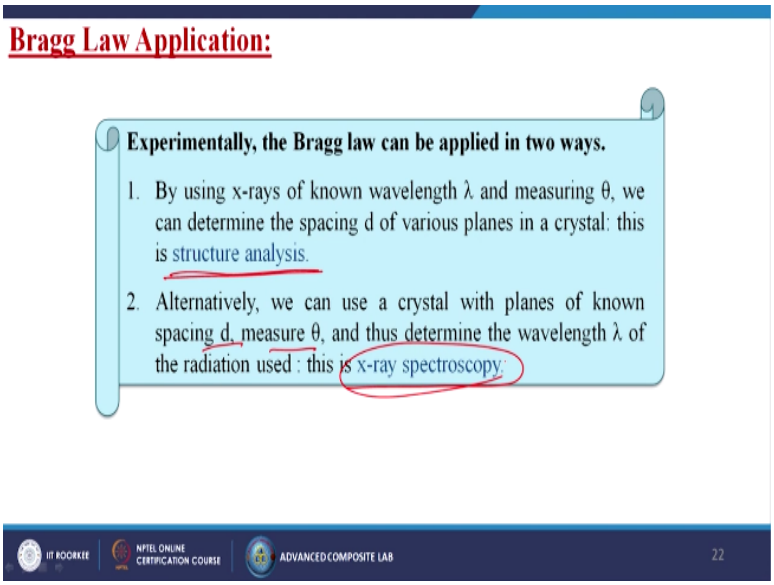

The Bragg's law can be applied in two ways, first is, by using the X-rays of known wavelength λ, and the measuring θ angle, we can determine the spacing d, of various planes in a crystal, this is called the structure analysis, so actually from this particular slides, we are actually starting the structural analysis of our materials. Alternatively we can use a crystal with planes of known spacing d, measure θ, thus determine the wave length λ of the radiation used, this is known as the X-ray spectroscopy.

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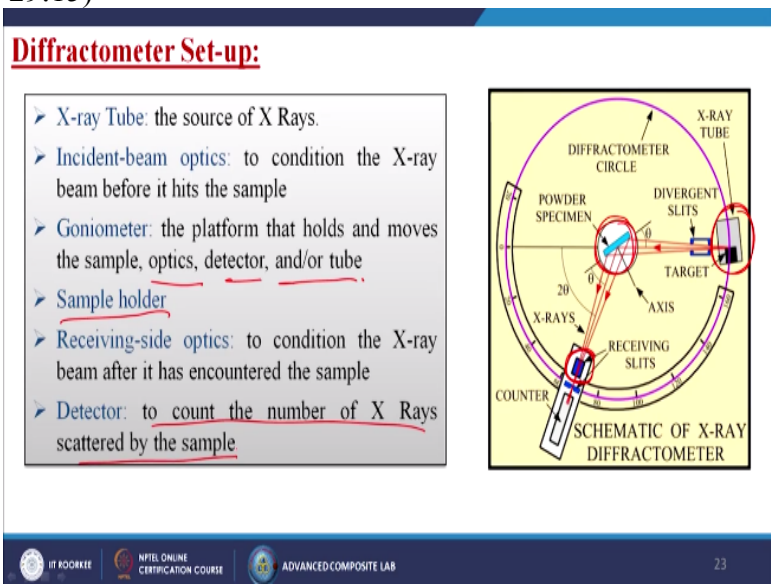

So, now we are talking to discuss about the diffractometer set-up, how it is work actually, so first it is having some X-ray tube, which is nothing but the source of X-rays, so in this particular case this is the X-ray tube, which is giving you the source of the X-ray, then incident beam optics, conditions the X-ray beam before it hits the sample, so we are having some kind of sleets, through that sleets, X-ray beams are coming and then it is falling on to the target itself, then we are having the goniometer, the platform that holds, so this is actually the platform, which is holding our sample, or maybe the materials

So, holds and moves are the sample of optics, detector, and or tube, then we are having the sample holder, which will actually hold the sample, then we are having the receiving side optics to conditions the X-ray beam after it has encounter the sample, so here we are having the receiving side which is just again managing all the diffracted beam over there, and then, we having the detector to count the number of X-rays scattered by the sample, so this is the whole set up of our X-ray machines.

Now, working up the X-ray diffracted meter, the θ is the angle

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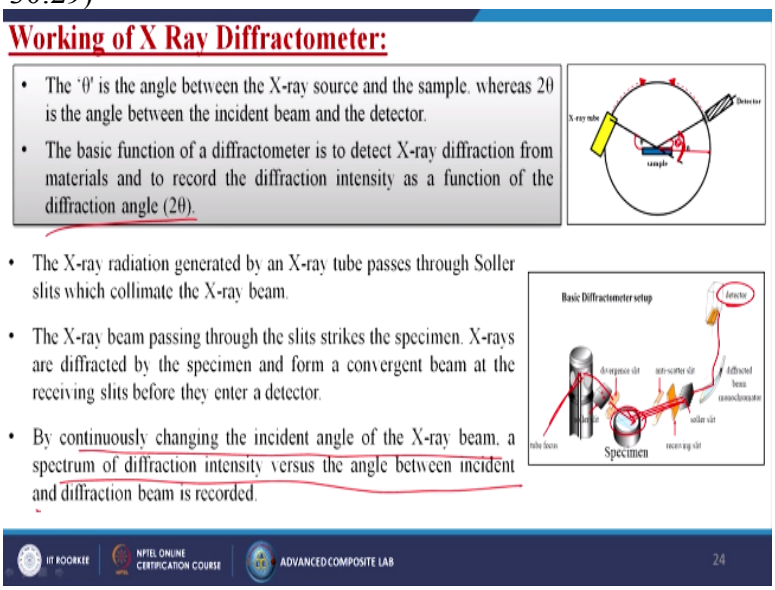

Between the X-ray source and the sample, where as 2 θ is the angle between incident beam and the detector, so this is the 2 θ, this is only the θ, and here it is also, this is also, only about the θ over there, so now, basic functions of diffractometer is to detect the x-ray diffraction from materials, and to record the diffraction intensity as a function of the diffraction angle, which is nothing but the 2 θ value.

The X-ray radiation generated by an X-ray tube passes through soller slits, which collimate the X-ray beam, so here focusing is going and then it is coming through this soller slits, and then it is falling on to your specimen here, we are keeping here, and then, we are having some antiscatters slits through that defected beam is going, and then we are having again soller slit through that it has going to some diffracted mono-chromater and then from that it is directly going through the detector itself.

So, the X-ray beam passing through the slits strikes the specimen X-rays are diffracted by the specimen and form a convergent beam at the receiving slits before they enter a detector, by continuously changing the incident angle of the X-ray beam, a spectrum of diffraction intensity verses the angle between incident and diffraction beam is recorded, so this is the working principle of that X-ray diffractometer

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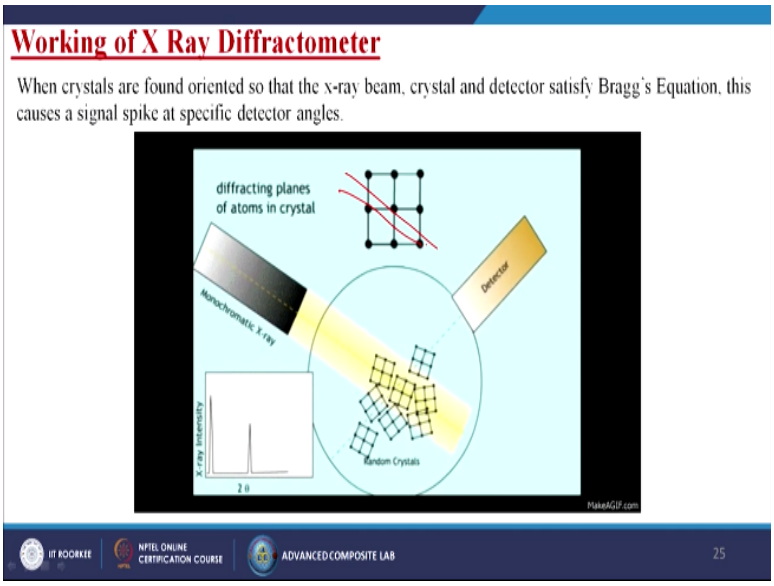

Which, I have already discuss from this particular just you can see that how you are getting the peaks, because it is hitting our materials in to different planes, so like, that D1, and then you can find out D3, so here, when it is hitting like, it is known as the D3, so different angle is hitting and then after that, we are getting the signals at the detector.

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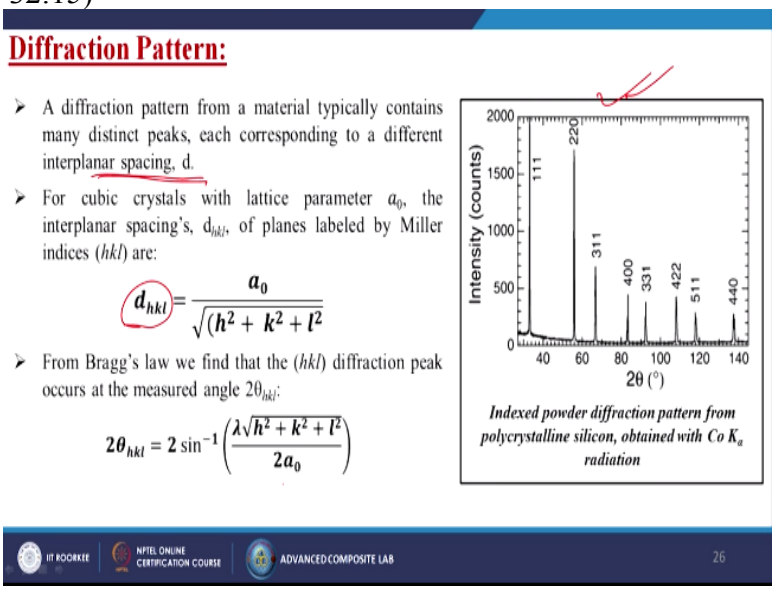

So, now we are going to discuss about the diffraction pattern, so generally after doing the certain study, whatever the results actually we are getting or maybe that, whatever graphs actually we are plotting, it looks like, this so here, diffraction pattern from a material typically contains many distinct peaks, each corresponding to a different inter-planar spacing or maybe the d, for cubic crystal with lattice parameter is 0, the interplanar spacing's d, generally HKL planes labeled by miller indices, miller indices is nothing but the HKL, these all are the planes are, d subscribe HKL=$a0/\sqrt{h^2+k^2+l^2}$, from Bragg's law, we find that HKL diffraction peak, occurs at measured angle 2θ, subscript HKL=2 $\sin^{-1}$,λ,$\sqrt{h^2+k^2+l^2}/2a_0$

So, now we are going to discuss about the almost the last part of this particular lecture is the study applications, so generally we are doing the phase identification is any materials, we can get the crystal structure determination, we can get the radial distribution functions, we can get the thin film quality, we can get the crystallographic texture of materials, percent crystalline, whether it is crystalline, or semi crystalline, or maybe the amorphous materials.
We can get the crystal size of the particular materials, we can get the residual stress or maybe the strain of that particular materials, we can get the defects, that is also another beauty of certain study, we can get the defects inside the crystal structure of any material, we can get the in situ analysis, phase transition, thermal expansion and coefficient etc.
and also the last but not the least is that super-lattice structure, so now we can conclude our lecture like, that in this particular lectures a overview has given to you about the X-ray generations and
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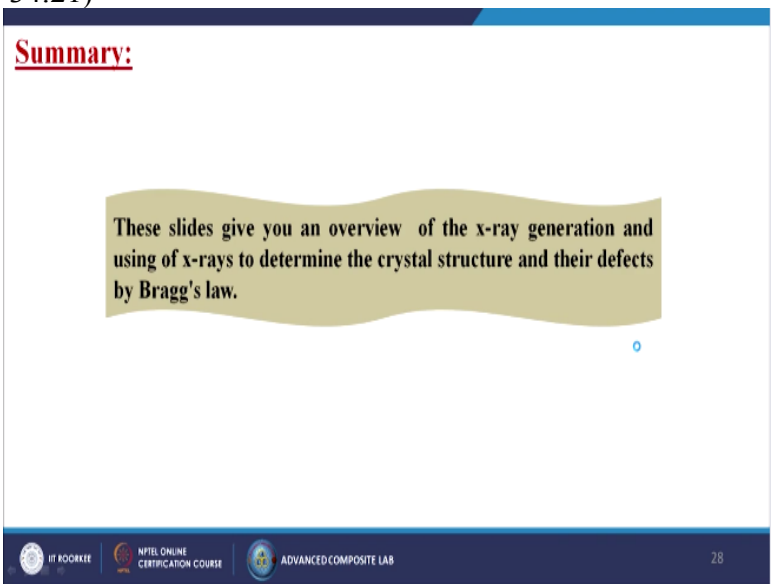

Using of the X-ray to determine the crystal structure and defects by the Bragg's law thank you.
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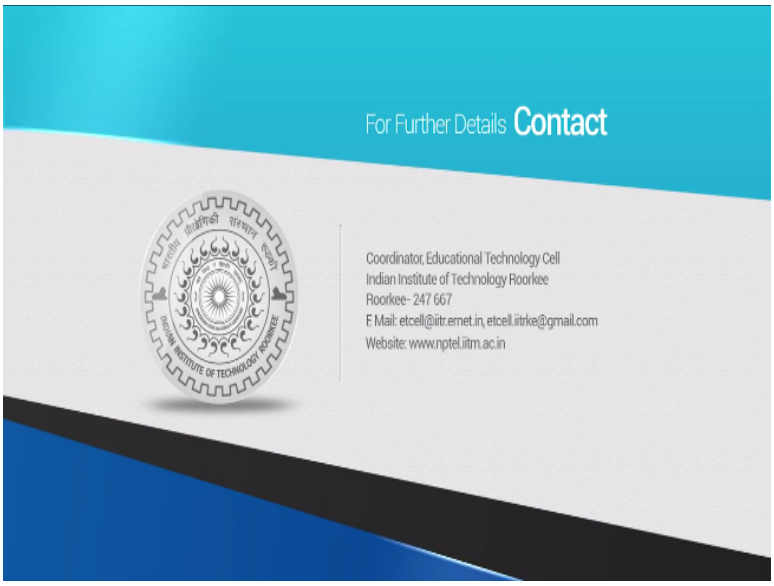

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