INDIAN INSTITUTE OF TECHNOLOGY ROORKEE
NPTEL
NPTEL ONLINE CERTIFICATION COURSE
Structural Analysis of nanomaterial's
Lecture – 12
Diffraction Methods and Directions of XRD
With
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In our previous lecture, we have discussed about the, basics of XRD or may be the X-ray diffractions and the equipment, so today we are going to discussed about diffractions methods and the directions of XRD, so first we will discussed about, what are the different diffractions methods.

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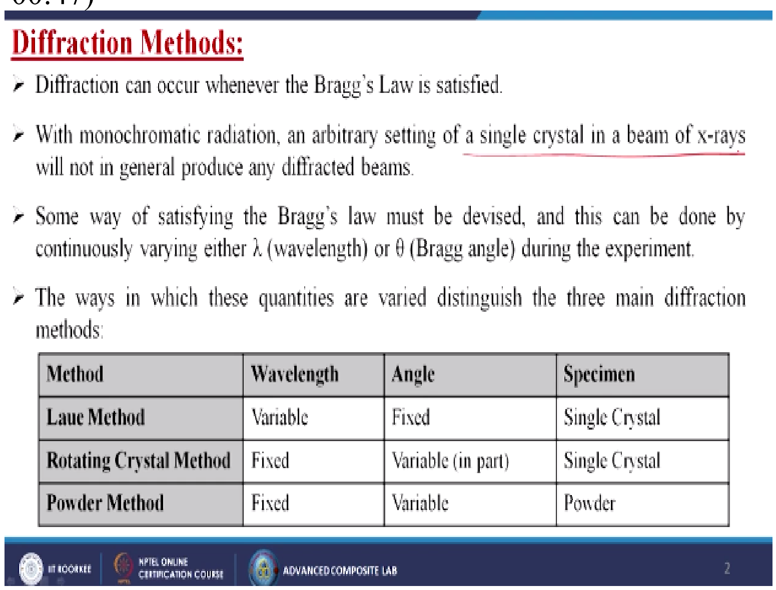

So diffractions can occur whenever the Bragg's law is satisfied, this we have already gone through, in our pervious lecture, with monochromatic radiations, an arbitrary setting of a single crystal, in a beam of X-rays, will not in general produce any diffracted beams, some way of satisfying the Bragg's law must be devised and this can be done by continuously, varying either, the wavelength λ, or may the Braggs angle, which is nothing but the θ, during the experiment, the ways in which these quantities are varied distinguish, the three main diffractions methods, so first is known as the Laue method where you, we are wearing the, variables is the wavelength angle is fixed and generally, we are doing it, for the single crystal, if it talk about the second one that is Rotating crystals methods, where the wavelength is also is fixed, angle variables in some part and that is also, we are doing for the single crystals and when we are talking about the powder XRD or may be the powder method, so the generally the wavelength is fixed here the Bragg's angle is the variable one and generally, we are doing it for the powder sample, so first we are going to discussed about the Laue method.

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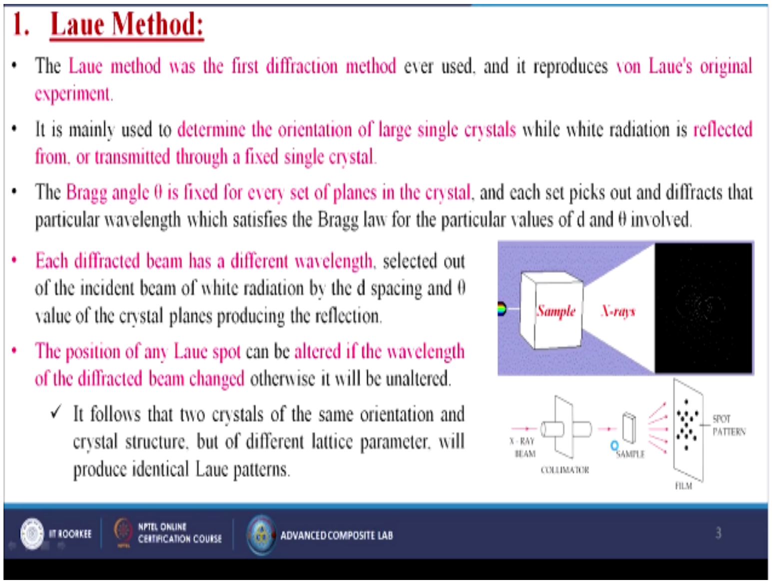

So the Laue method was the first diffraction methods, because it has been developed fast, ever used till today and it reproduces von Laue's original experiment, it is mainly used to determine the orientations of large single crystals, so generally this is for the large single crystals samples, while white radiations is reflected from, or transmitted through a fixed single crystal, the Bragg's angle that is fixed in this particular case, for every set of planes, in the crystal, in the crystal and each set picks out and diffracts that particular wavelength which satisfies the Bragg's law for the particular values of D which is nothing but the inter-planer distances and θ involved, so from this particular image, you can see that we are having the sample which is nothing but the single crystals, so the X-rays are coming from these way that incident beam and then through this, the X-ray are maybe the diffracted beam has been generated, in this particular region and then it is directly falling on to the particular film and we are getting some kinds of white spots over there, so that is nothing but the different diffraction picks, or maybe the spots, each diffracted beam has a different wavelength selected out of the incident beam of white radiations by the D spacing and θ value of the crystal planes producing the reflections, the positions of any Laue spot can be alter early, if the wavelength of the diffracted beam changed otherwise it is not, it follows that two crystal of the same orientations, suppose we are having the two crystals having the same orientations and same crystal structure, but of different lattice parameter, will produced the same identical Laue patterns.

So whatever I have discussed into the upper image the same thing is replicated over here, we are having that incident beam then we are having collimators, then it is going through the samples then rays has been diffracted and then on the film it is giving some kind of spot pattern.

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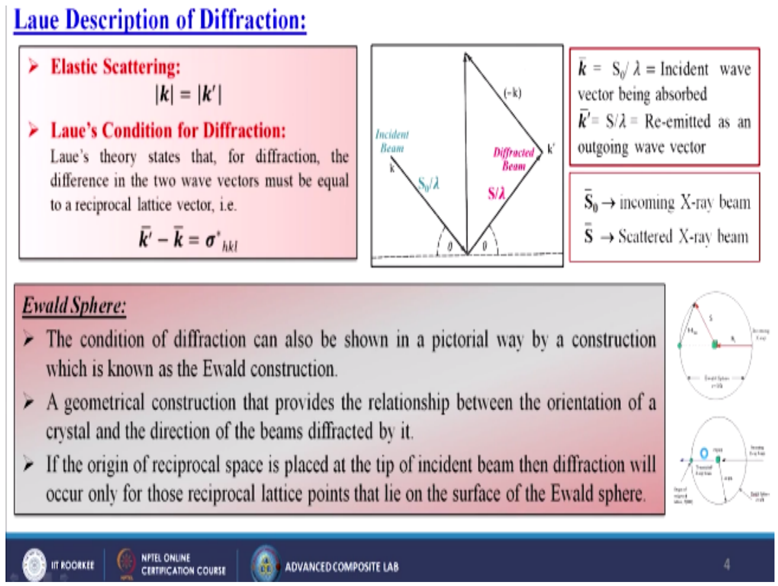

Now what is the Laue descriptions of diffractions, so now we are going to discuss from this particular graphs, so in this particular case, first we have to know what is the Elastic Scattering, it is nothing, but the total K, I means magnitude, magnitude of the incident beam and the magnitude of the diffracted beam which is nothing but the K and the K prime, now is the Laue's on this on spot diffractions, so Laue's theory states that for diffractions the differences in the two wave vectors must be equal to a reciprocal lattices vector, what is that, that is K prime vector minus K vector is equal to sigma prime HKL, so what is K prime, so K prime is nothing but the S not by the λ which is nothing but the incident wave vector break it being absorbed, so here the D is the S not by λ and K prime vector, which is nothing but the A by λ Re-emitted as an outgoing vector, which is nothing but the S λ, so what is S's not prime incoming X-ray beam and S prime is the scattered X-ray beam.

Now from this particular we are going to discuss about the Ewald sphere, so Ewald sphere generally, it is a one kind of conditions of diffractions can also be shown in pictorial way by a constructions which is known as the Ewald constructions, so this is known as the Ewald constructions, so here the incoming beam is coming is nothing but the S0, then through single crystal it has be diffracted and it is going to the S and it has falling on to the film in this particular point.

So generally the Ewald sphere, if we make the thing that a virtual, or maybe the imaginary sphere that sphere is known as the Ewald sphere, whose radius is one by λ, I means the one by the wavelength, now this actually if is put the perpendicular, this is known as the one by DHK, so geometrical constructions that provides the relationship between the orientations of a crystal and the directions of the beams diffracted by it, if the origin of reciprocal space is placed at the tip of incident beam then diffractions will occur only for those reciprocal lattice points that lie on the surface of the Ewald sphere.

So in this particular case also we can see that the incoming X-ray beam is coming through is this one and then one this is for the, if it is passed diffracted and one is through transmitted, so if it is transmitted it is also coming into this particular point and here also the same thing Ewald sphere the radius is one by λ, now actually we are going to make some relations or may be co-relations in between the Laue descriptions and Ewald sphere with the Bragg's law.

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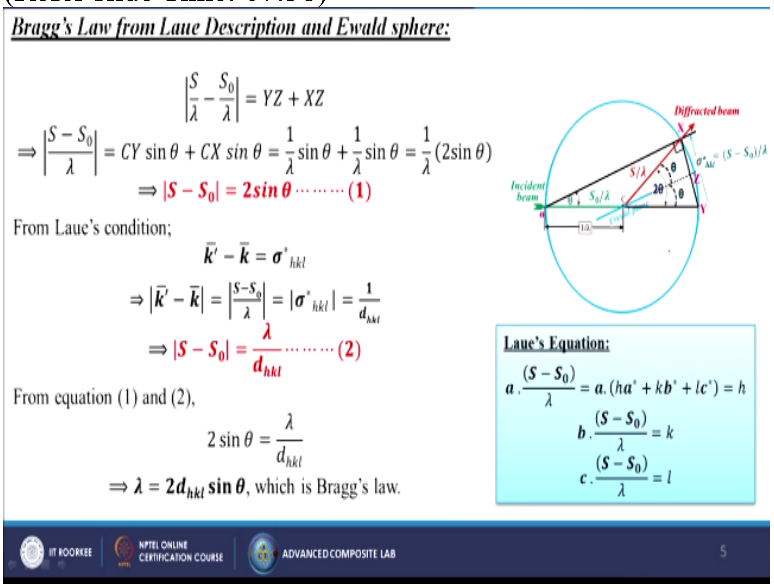

So in this particular image may that figure you can understand that the incident beam is coming and then you are having your samples are maybe the single crystals over here and it has been diffracted beam and it falling on to the film, in this particular point which is nothing but known as the X, so now if this angle is two θ it is nothing but the diffracted angle, so this is the total two θ value over there so here in this particular cases so your C is going if we make a lines so this angle is θ and this angle is θ, so whole parts is known as the two θ value.

So now this value is 0 by λ which we have already gone through and now this one diffracted beam is also by the S λ so now we are going to make a perpendicular on to this point on X and we are going to make a perpendicular from O to X also, so what is total XY, XY is nothing but the XZ+ZY, which is nothing but the sigma prime HL, which is nothing but the S-S0 by λ, so now come to here and here this is the radius here R is equal; to one by λ so now if we write like this that S by λ- S0 by λ which is nothing but the YX+ XZ, so here S0 λ and S λ so S λ – S0 λ is equal to YZ, YZ + XZ which is nothing but the S –S0 by λ is equal to CY, CY mean this C and Y, so CY sin θ + CX cos θ so this one is cos θ + CY sin θ which is nothing but the one by λ sin θ +one by λ sin θ that means if we it common one by λ so one by λ two sin θ so that means S- S0 is equal to two sin θ this is the number one equations.

Now from Laue conditions already we have gone through in our last slides that K prime vector – K vector is equal to sigma prime HKL where the magnitude of K prime vector- K prime is equal to S-S0 by λ which is nothing but the sigma prime HL is equal to one by DHKL so from this particular point we are getting this value so that means S-S0 s equal to λ by DHKL now from equations if we combining the both so now two sin θ here two sin θ and in teams of S- S0 we are replacing it by the λ by DHKL so two sin θ is equal to λ by DHKL that means λ is equal to two DHKL sin θ which is nothing but the Bragg's law so what is the Laue conditions so Laue condition is that A multiplications with S-S0 by λ is equal to A into HA prime + KB prime +LC

prime is nothing but the H so B prime into S-S0 by λ is equal to K and C prime S- S0 by λ is equal L so this is the Laue equations so from this particular case we have make some co-relations with the Bragg's law from the Laue descriptions and the Ewald sphere.
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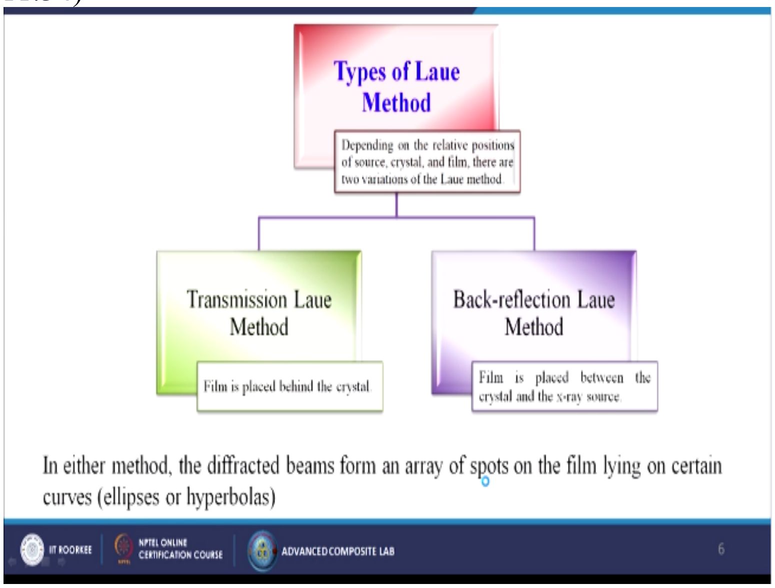

Now we are going to discuss about the different types of Laue methods so already what is Laue method so it is depending on the relative positions of the sources crystal and film there are at two variations of the Laue method one is called the Transmission Laue method another one is called the Back-reflections Laue method what is transmission Laue method that is film is placed behind the crystal so that means you are having that source of X-ray you are having are having that crystals and you are putting film the that means so that means the ray is transmitting through crystal and falling on to you are film and what is back reflections Laue method the film is placed between the crystal and the x-ray source that means you are having that source you are having the film and you're having that crystal.

So direct incident beam is going on into the crystals then it is back scattered, or maybe back reflected and then it is falling on to the film itself in either method the diffracted beam from an array of spots on the film lying on certain curves maybe whatever the points it is falling on to the film of you see it carefully we can find it either the shape of that point is maybe the elliptical, or some kind of hyperbolas, okay

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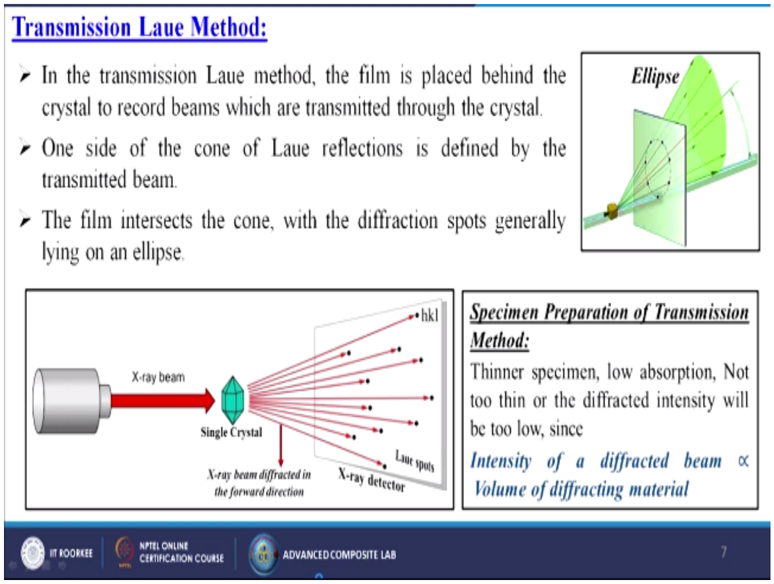

Now we are going to disused about the Transmission Laue method, so in the transmission Laue method the film is placed behind the crystal to record the beam which are transmitted through the crystal itself, so in this case this is our crystal films so one side of the cone of Laue reflections is define by the transmitted beam, so this side is the transmitted beam, so the film intersect the cone with that diffractions spots generally lying on the ellipse, so you can this in this particular image.

Now specimen preparations how we are going to prepare the specimen for transmission maker, thinner specimen, low absorption not too thin or the diffracted intensity will be too low, since intensity of the diffracted beam is directly proportional to the volume of diffracting of diffracting materials, so in this beam you can see the X-ray beam is coming from this particular side then we are having that single crystal and through that the beam is transmitted and it is falling on the X-ray detectors, these spots are known as the Laue spots and which we can determine by the HKL value.

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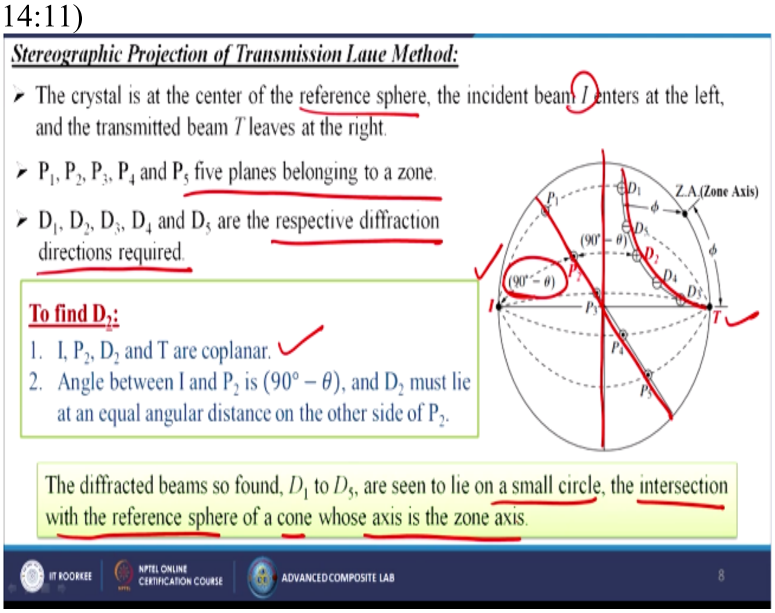

So in the next slides we are going to discuss about the stereographic projections of transmission Laue methods, so the crystal is at the center of the references sphere the incident beam is I, so this is the I enters at the left, so this is the left side and the transmitted beam T leaves at the right side, now this P1, P2, P3, P4, P5 this five planes belonging to a Zone, so this is known as the zone D1,D2,D3,D4,and D5 are the respective diffractions directions required.
So this one is the diffractions directions, now suppose we are going to find the point D2, so what we have to do we have to know that I P2,D2 and T are the coplanar, so this I, this P2, then D2 and the T are coplanar, now angle P2 and I and P2 is 90- θ and D2 must lye at an equal angular distances on the other side of P2, so if you take this one as a mirror so just D2 the is the mirror image of P2 now the diffracted beams so found D1 to D5 are seen to lye on this small circle the intersections with the references sphere of a cone whose axis is the zone axis.
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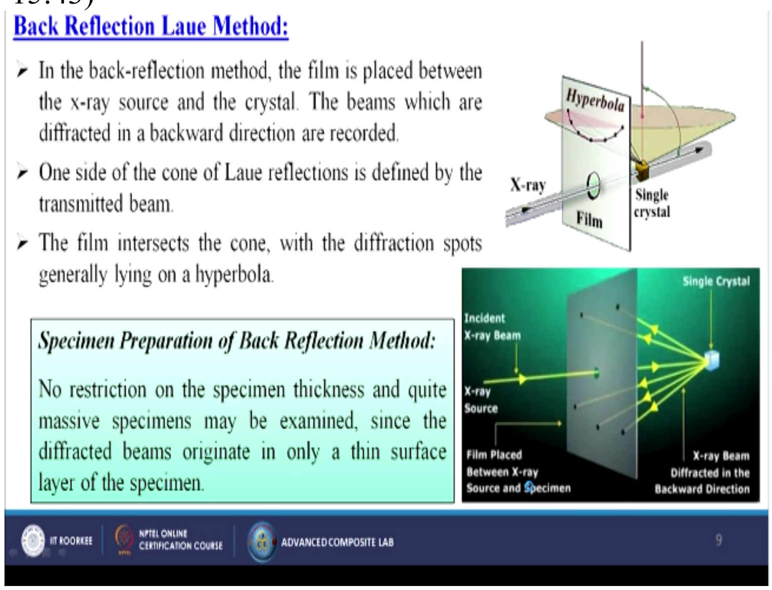

Now we are going to discussed about the back reflections Laue method so in this back reflections method the film is placed between the X-rays sources and the crystal the beams which are diffracted in a backward directions are recorded see so this is our film and this is our single crystal, so in this case the incident beam is going through this particular hole and directly it is falling on to the single crystals and then the back reflections is talking place and then those diffrected beam is falling on this particular film and they are making the shape in a hyperbola manner.
So the beams which are diffracted in the backward directions are recorded on this particular film one side of the cone of the Laue diffractions, is define by the transmitted beam the film intersects the cone with the diffractions spots generally lying on a hyperbola, so how we are going to prepare the specimen for this back reflections methods, no restrictions on the specimen thickness and quite massive specimen maybe examining, since the diffrected beam originate in only a thin surface layer of the specimen.
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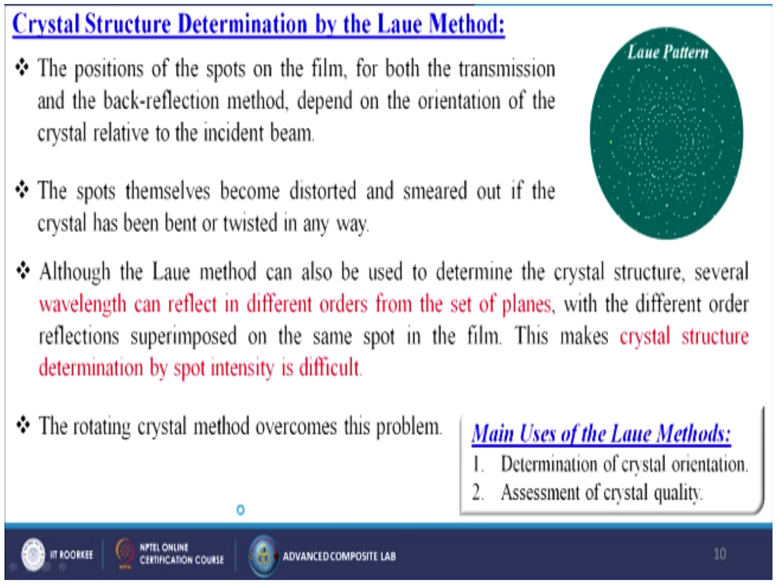

Now we are going discuses about the Crystal Structure Determinations by the Laue method, the positions on the spot on the film for both the transmission and the back-reflections method depend on the orientations of the crystal relative to the incident beam, of course from this particular image you can see that we are having the Laue pattern and through that you are getting some spots which is nothing but the Laue spots and it is giving you the picks are may be the information's about the crystals, the spot themselves they became distorted and smeared out.

If the crystal has been bent or twisted in any way, otherwise it will be a form spot on it, although the Laue method also can be used to determine the crystal structure, several wavelengths can reflect in different order from the set of planes is of course with the different order reflections superimposed on the same spot in the film this makes crystal structure determinations by spot intensity is very, very difficult because there is a chance of overlapping of different spots each other, the rotating crystal methods overcome this problems, what is the main use of the Laue methods determinations of the crystal orientations and the assessment of then crystal quality.

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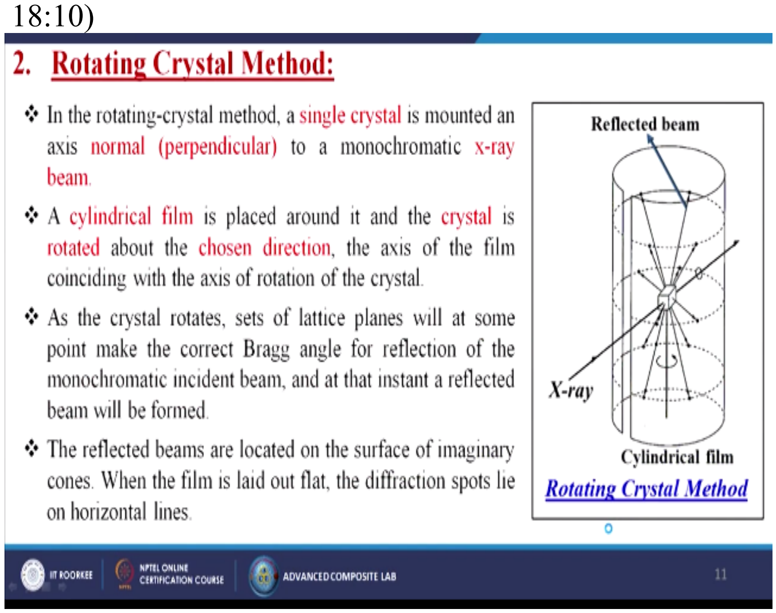

Now we are going to discussed about the second types which is nothing but the known as the Rotating crystal method, so in the rotating crystal methods a single crystal is mounted on an axis normal to a monochromatic X-ray beam, so in this particular case we are putting the crystal in this particular positions a cylindrical film is placed around it and the crystal is rotated about the chosen directions, the axis of the film coinciding with the axis of rotations of the crystal also.

So in this particular case what we are doing that we are putting a single crystals and then single crystal is also rotating in a particular directions and that crystal is surrounded by some films and that film is also rotating into the same directions with the crystal itself so as the crystal rotates the set of lattice planes will at some point make the current Braggs angle for the reflections of the monochromatic incident beam and at that instant a reflected beam will be formed, the reflected beam are located on the surfaces of imaginary cones, when the film is laid out flat, the diffractions spots lie on the horizontal lines, so you can see that do you are getting that horizontal spots on to that from the diffractions beam.

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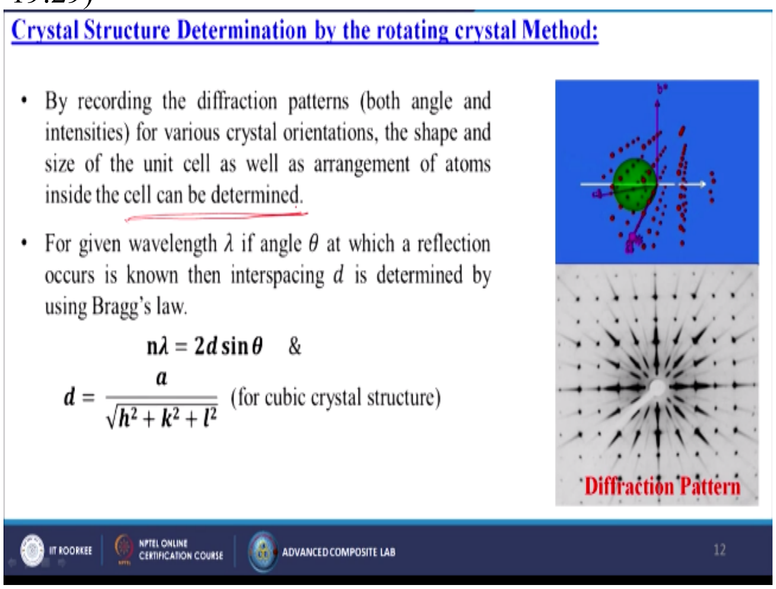

So how to get the Crystal Structure Determinations by the Rotating Crystal Method, so by recording the diffractions patterns both the angle and the intensities, intensities here you can that how we are getting the different points on to the film itself, so for various crystal orientations the shape and the size of the unit cell as well as the arrangement of atoms inside the cell can be determined for given wavelength λ, if Bragg angle θ at which a reflections occurs is known then interspacing D is determined by using the Bragg's law which is nothing but the L λ is equal to 2D sin θ and D is equal to A by root over $H^2+k^2=l^2$ generally it is for the cubic crystal structure.

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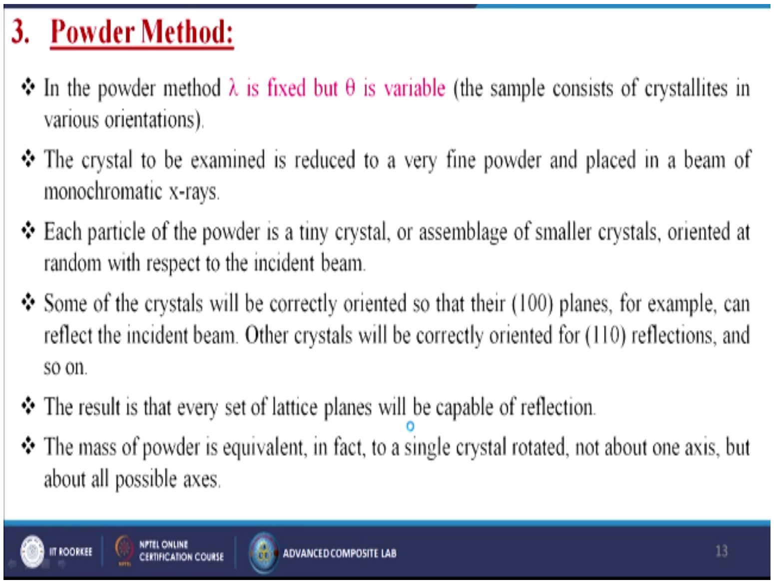

Now we are going to discussed about the Powder method, so in the powder method as I already told you initially that here the wavelength λ is fixed and the Braggs angle or maybe the θ is variable the sample consist of crystallites in various orientations, the crystal to be examined is reduced to a very fine powder, so first we are making the material in a powder in a powder form and then placed in a beam of monochromatic x-rays, each particle of the powder is a tiny crystal is a small, small crystals or assemble of smaller crystals, oriented at random with the respected to the incident beam, some of the crystal will be correctly oriented so that their 100 planes, for example, can reflect the incident beam, other crystal will be correctly oriented for 110 reflections and so on the result.

Is that every set of lattice planes will be capable of reflections, the mass of powder is equivalent, in fact to a single crystal rotated, not about one axis, but about all possible axes.

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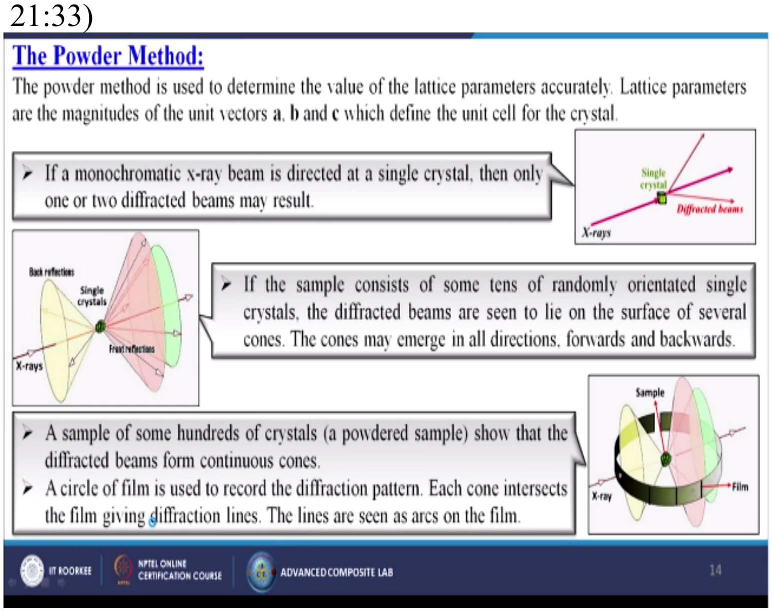

So how we are going to do the powder method, the powder is used to determine the value of the lattice parameters accurately, lattices parameters are the magnitude of the unit vectors A.B and C which define the unit cell for the crystal, so here simple in a monochromatic X-ray beam is directed at a single crystal then only one or two diffracted beams may result, so from this picture you can see that X-ray when it is coming, it is falling on to the single crystals may be one or two diffracted beam has been fall it, but when you are talking about the powder crystals.

Simple when the incident beam is coming on to that powder materials then there are, so many different diffracted beam has be generated and it is falling into the film itself, so if the sample consist of some tens of randomly oriented single crystal the diffracted beams are seen to lie on the surface of several cones, the cones, may emerge in all directions forwards and backwards.

So you can see that plane falling, there are so many single crystals over there that is some kind of powder materials, so tiny single crystals are there so when the X-ray beams are coming then after reflections it is creating so many cones for different crystal materials, so a sample of hundred of crystals a powder sample show that the diffracted beam from continuous cones, a circle of film is used to record the diffractions pattern each cone intersect the film is giving diffractions lines, the lines are seen as the arcs on the films , so this lines actually it is known as the arcs on to the film itself.

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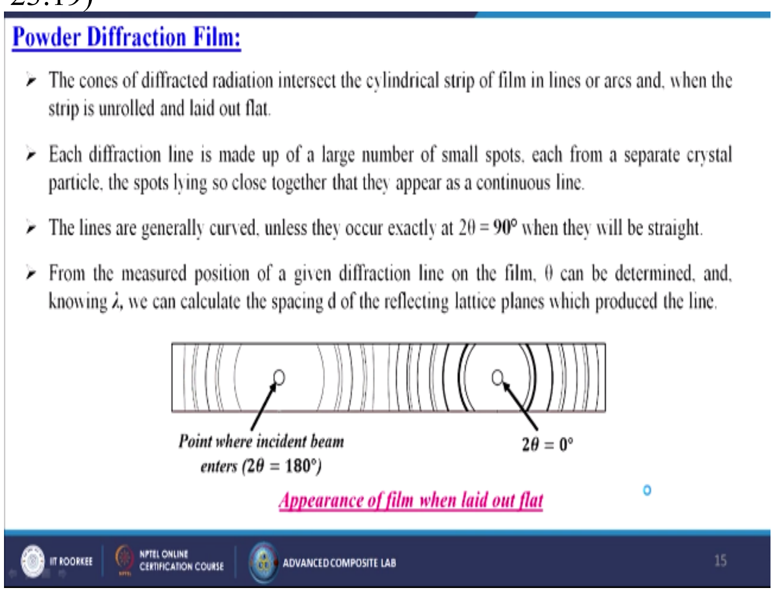

Now how it looks, Powder Diffractions Films the cones of diffracted radiations intersect the cylindrical strip of film in lines or arcs and when the strip is unrolled and laid out flat, so after that we are making the film in a flat and we are getting the lines in such a manner some lines are strip some lines are in a circular shape, or may be the round shapes, so each diffractions lines is made up of a large number of small spots each from a separate crystal particle the spots lying.

So close together that they appears as a continuous line, it looks like a bar code the lines are generally curved unless they occur exactly at 2 θ equal to 90 degree when they will be straight in this particular case you can see that we are getting the lines almost straight, so from the measured position of a given diffractions line on the film, θ can be determined and knowing the λ value we can calculate the spacing D of the reflecting lattice planes which produced the lines, so from this particular case we are getting the θ value is 0 and in this particular line here the two θ is 90 degree, and in this particular case the 2 θ is equal to 180 degree.

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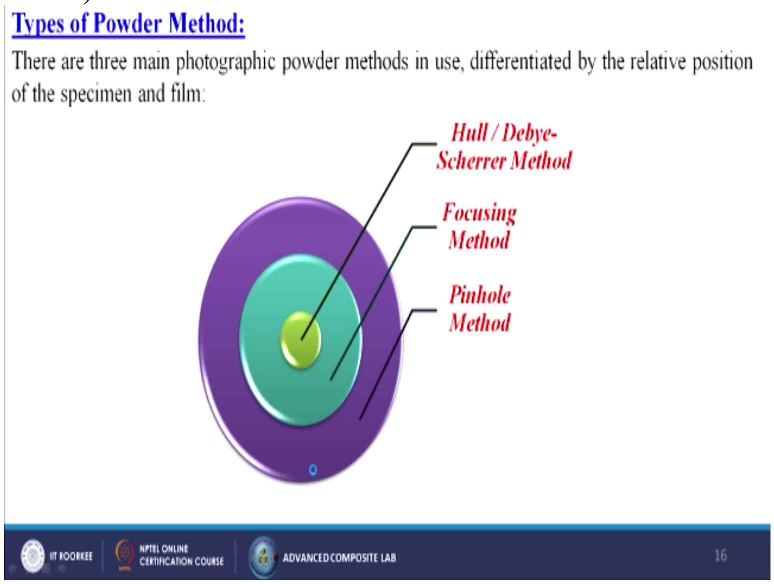

Now what are the types of the Powder methods so generally three types methods are available, so there are three main photographic powder methods in use, differentiated by the relative positions of the specimen and the films, so first one is known as the Hull Debye- Scherrer methods next one it is called the focusing methods and the third one is known as the Pine hole method.

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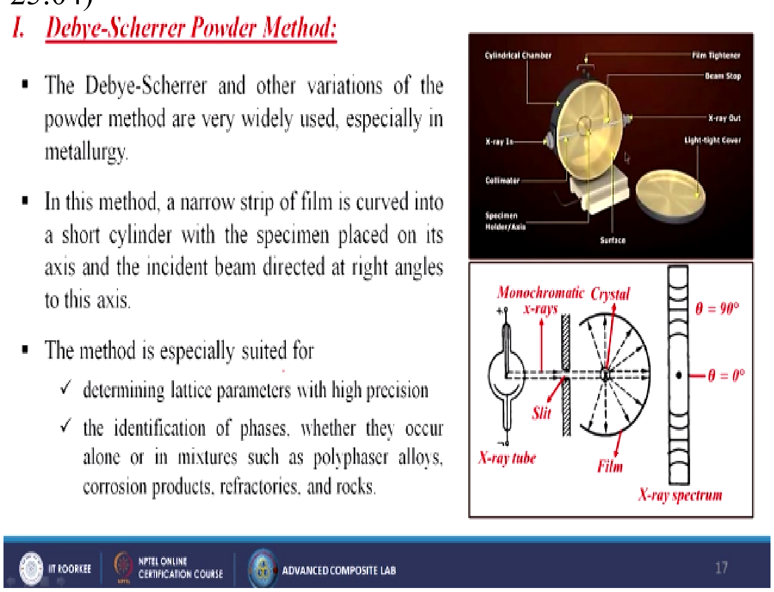

So what is the Debye-Scherrer Powder so the Debye-Scherrer and other variations of the powder method are very widely used especially in metallurgy that means for the metallurgical samples, so in this methods, a narrows strip of film is curved into a short cylinder with the specimen placed on it is axis and the incident beam directed at right angles to the axis itself this methods is especially suited for determining the lattice parameters with high precision, the identifications of phases whether they occur alone, or in mixture such as polyphascr alloys corrosion products refractories and rocks.

So in this particular case we are having the X-ray tubes, X-ray beam incident is going we are having that slit and then we are having the film and here are we are going to put our crystal structure and we are getting g the x-ray spectrum in this particular manner this is X θ is equal to 0 degree and in this particular line the θ is equal to 90 degree.
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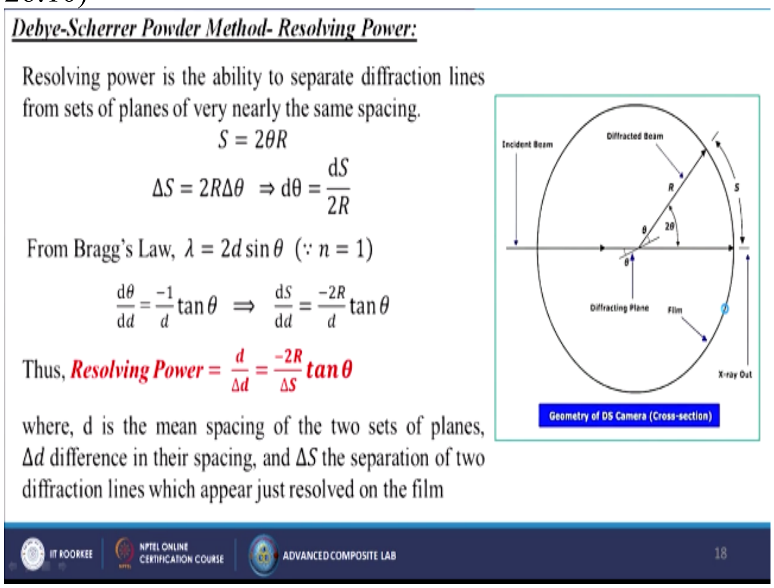

Now what we are getting from the Debye-Scherrer Powder Methods Resolving power, if you are going to calculate the resolving power, so resolving power is the ability to separate the diffractions lines from sets of planes of very nearly the same spacing, so here in this particular case what we are going to do we are putting the incident beam and then it is falling on to the samples and the reflected beam is falling on to the film in this particular point.
So this is the two θ which is the nothing but the diffraction angle and this is arc and here the incident bean angle is θ which is nothing but the diffracting plane, so this is the film and if we go, make a straight line from this point to this point which is nothing but the known as the S, so here the S is equal to two θ into R, so what is the Debye so if we do the diffractions is equal to two R which is constant in dl θ which is nothing but the D θ is equal to DS by QR.
So from Braggs law what we know, that λ is equal to two D sin θ where N is equal to 1, so D θ by DD is equal to – one by D tan θ is equal to DS by DD is equal to-2R by D tan θ, so thus the resolving power D upon dl D is equal to – 2R by dl is tan θ, so where D is the mean spacing of the two sets of planes dl D is the differences in their spacing so in the last slides you have seen that so many lines are coming so what is the distances in between those spacing which is nothing but the know as the dl D and dl S is the separations of two diffractions lines which appear just resolved on the film itself.
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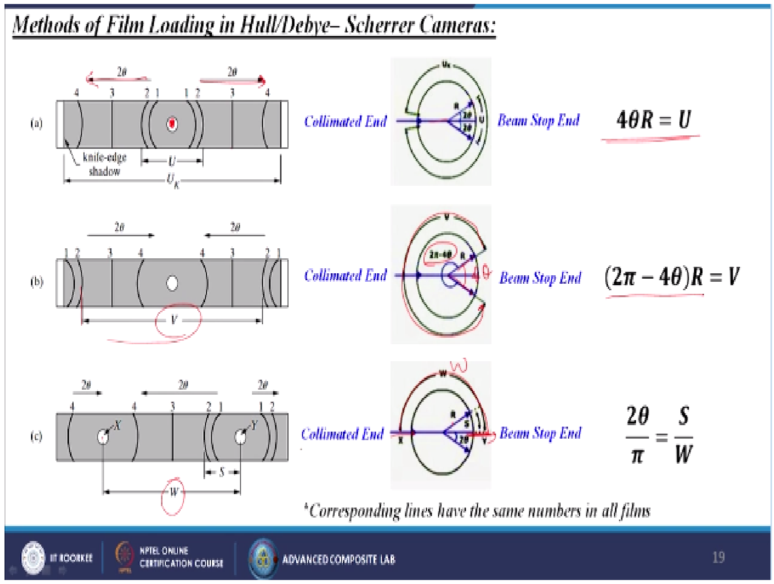

Now there are so many methods of film loading on the Hull Debye-Scherrer Cameras so first when we are talking about these say suppose we are having that here the θ is equal to 0 degree here the θ is 90 degree, two θ is increasing in this way, two θ is increasing in this way from 0 both the sides so this is known as the knife-edge shadow, so these distances is known as the capital U.

So one we are talking about the collimated end, so this side is the collimated end over here and then when the incident beam is going and then it is reflecting and it is making an angle of four θ, so here the four θ R is the U, so from here to here, this distances to this distances when we are talking about the capital V means here how to calculating the spacing's in between the lines so if we want to calculate the spacing's in between these two points, number two to this number two points.

So in this particular case which is nothing but the V, so how to calculate the V so V is what the V is the total length of these one so this is the R and this the 4 θ so now rest of the thing is two π-4 θ so 2 π- 4 θ into R is equal to capital V, now if we are measuring the distances in between this centers and this centers which is nothing but we are denoting as a capital W so now capital W means from this point to this point which is nothing known as the capital as the W, so incident, we was going and then we are thinking that it is going through fully transmitted, so now this angle is totally again it is 4 θ so 2 θ by π is equal to capital S by W, corresponding lines have the same numbers in all films.

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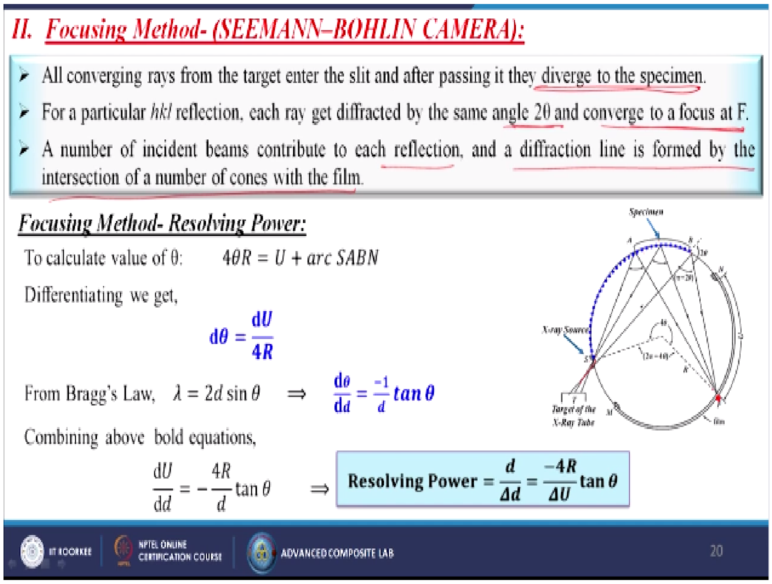

Now we are going to discuss about the Focusing methods which is nothing but the SEEMANN-BOHLIN CAMERA it is also known as, all converging rays from the target enter the slit and after passing it they diverge to the specimen, from HKL reflections each ray get diffracted by the same angle 2 θ and converse to you are focus at F, a number of incident beam contribute to each reflections and diffractions line is formed by the intersections of a numbers of cones with film.

So in this particular case you can see that target of the X-ray tube is this one then it is passing through to the materials then again it is diverging and then after that diverging it is falling on to the specimen and then again it is converging on to the film itself on a particular point, so here from that last slides lectures PPt's we have already gone through that here the S and R so here it is known as the 2π – 4 θ because this is known as the 4 θ so here to calculate the value of θ, 4 θ R, 4 θ R so 4 θ R into R which is nothing but the U+ arc SABN.

So the total this area so differentiating we get D θ is equal to DU by 4R from Braggs law λ is equal to 2D sin θ which is D θ by DD is equal to – one by D tan θ combining our two equations this is number one equations and this is number two equations so DU by DD is equal to- 4R by D tan θ so the resolving power D upon dl D is equal to – 4R by dl U tan θ.

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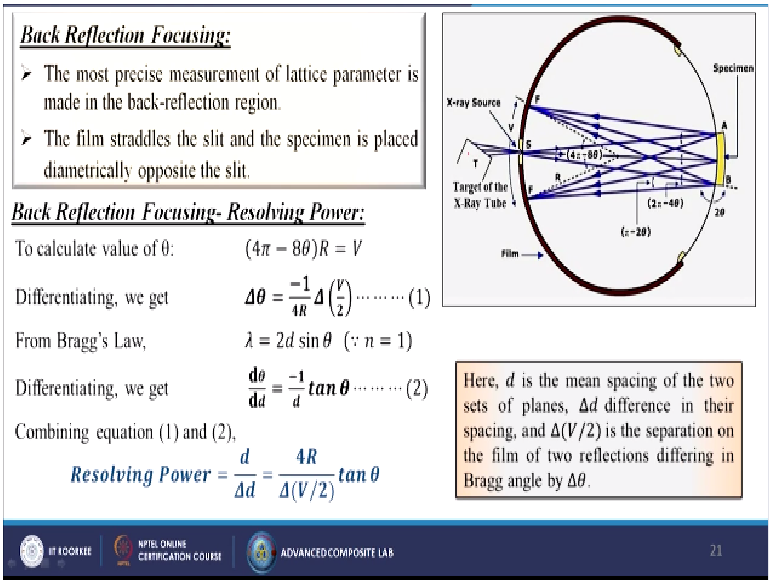

Now we are going to discussed about the Back Reflections Focusing so in that back reflections focusing what happen the simple thing the target is here the X-ray is coming then it is falling on to the specimen then back reflections is talking places and through that all the beams is falling into these two points into the on to the film itself so the precise measurement of lattice parameters is made in the back-reflections region, the film straddles the slit and the specimen is placed diametrically opposite the slit.

So in the back reflections focusing resolving power to calculate the θ value, so in this case what is that this angle is 4 π- 8 θ sop 4 π- 8 θ into R so we are talking about this one, so 4 π- 8 θ into R is equal to capital V so differentiating we get dl theta is equal to – 1 by 4R into dl V by 2 so from Braggs law λ is equal to 2D sin theta where N is equal to one so differentiating we get D theta by DD is equal to – 1 by D tan theta so combining equations 1 and 2 resolving power D up an dl D is equal to 4R by dl V by 2 tan theta so here D is the mean spacing of the two sets of planes 1 dl D is the differentiations in their spacing and dl V by 2 is the separation on the film of two reflections differing in Braggs angle by dl theta.

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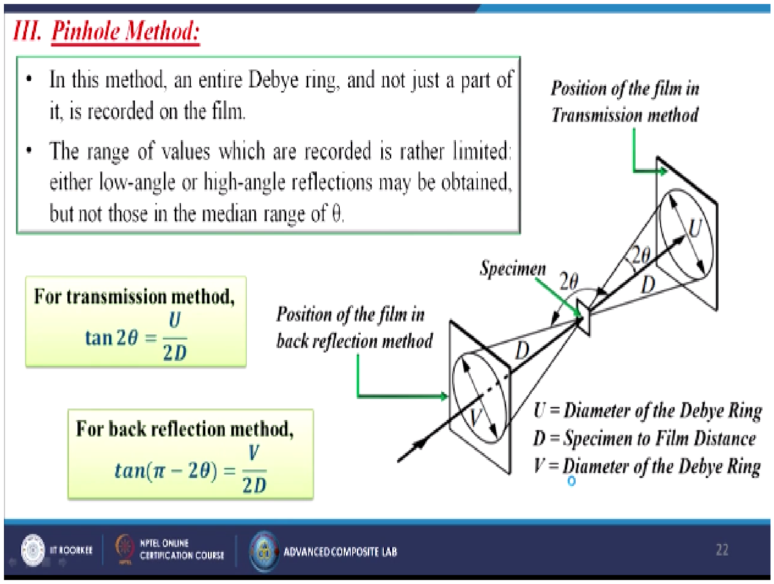

So now we are going to discussed about the Pinhole methods so in this methods, an entire Debye ring and not just a part of it, is recorded on the film, the ranges of values which are recorded is rather limited either low-angle or high angle reflections may be obtained but those in the median range of theta so in this particular case we are putting one spacing over here, and now some rays maybe it has transmitted through samples or may be it has been reflected through samples.

So both the diffracted beam are recording in this particular case so here whatever the shape are forming that these total distances is known as the capital U and here it is known as the capital V so U is the diameter of the Debye Ring D is the specimen to film industry which is nothing but the this one and in this case is this one and capital V is the diameters of the Debye Ring into the back reflections methods so here this is the film for the back reflections methods and this is the film for the transmission methods

So for the transmission tan 2 theta is equal to capital U by 2D this is number one and for back reflections methods tan π-2 theta, so this angle is totally 2 theta so 2 theta is angle over here diffracted beam so here π-2 theta is equal to capital V by 2D so this is now as for the Pinhole method.

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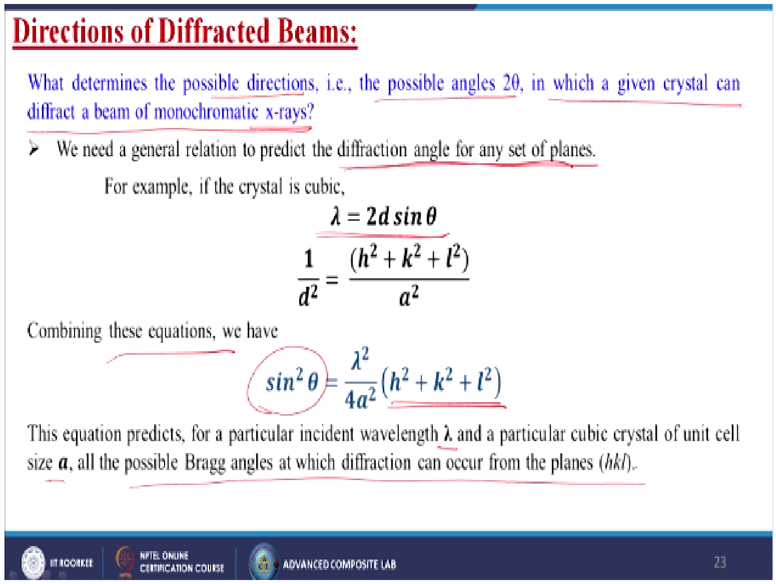

So now we are going to discussed about the Directions of the Diffracted Beams so what determines the possible directions that is the possible angle 2 theta, in which a given crystal can diffracted a beam of monochromatic X-rays we need a general relations to predict the diffractions angle for any set for planes so for example if the crystal is cubic so generally λ is 2D sin theta so one by D^2 is equal to $H^2+k^2+L^2$ by A^2, combining this equations we have $\sin^2$ theta is equal to λ^2 by $4A^2$ into $H^2+K^2+L^2$ the equations predicts for a particular incident wavelength λ and a particular cubic crystal of nits cell size A all the possible Braggs angle at which diffractions can occur from the planes which is nothing but the HKL lengths.

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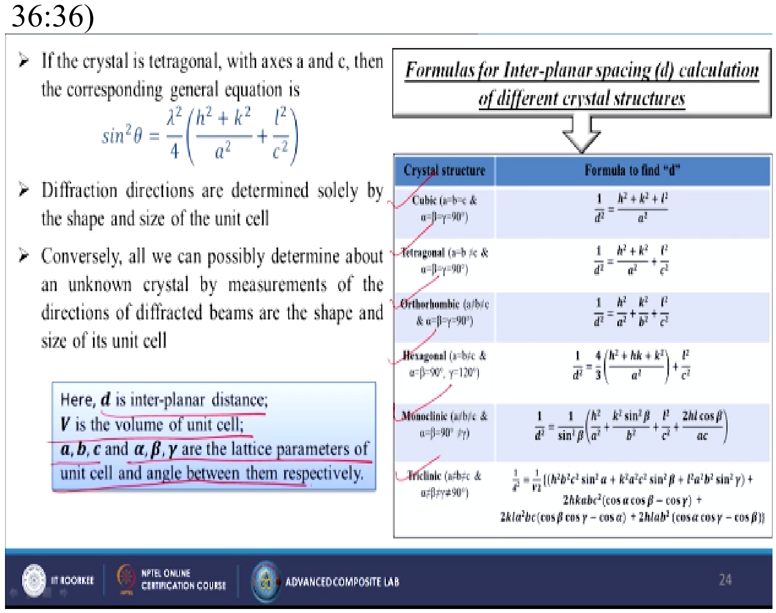

Now if the crystal is tetragonal with axes A and C, then the corresponding general equations is $\sin^2$ theta is equal to λ^2 by 4 into H^2 +K^2 by A^2+ L^2 by C^2 diffractions directions are determined solely by the shape and of the unit cell, conversely all we possible determine about an unknown crystal by the measurement of the directions of the directions diffracted beams are the shape and size of it is unit cell so here in this particular case we having formulas for inter-planers spacing D calculations of different crystal structure.

So for Cubic's so generally where A is equal to B is equal to C and alpha, beta, Gama all are 90 degree so what is the D formula on by D^2 is equal to H^2 +K^2 +L^2 by A^2 so like this way we can calculate for cubic, tetragonal, oribarbambic, hexagonal, monoclinic, or may be the triclinic structure and here all are the their assumptions and right hand side is the how to find the D or may be the calculations of the D value, so here D is known as the inter-planer distances capital V is the volume of unit cell small a,b,c and alpha, beta gama are the lattice parameters of unit cell and angle between them respectively.

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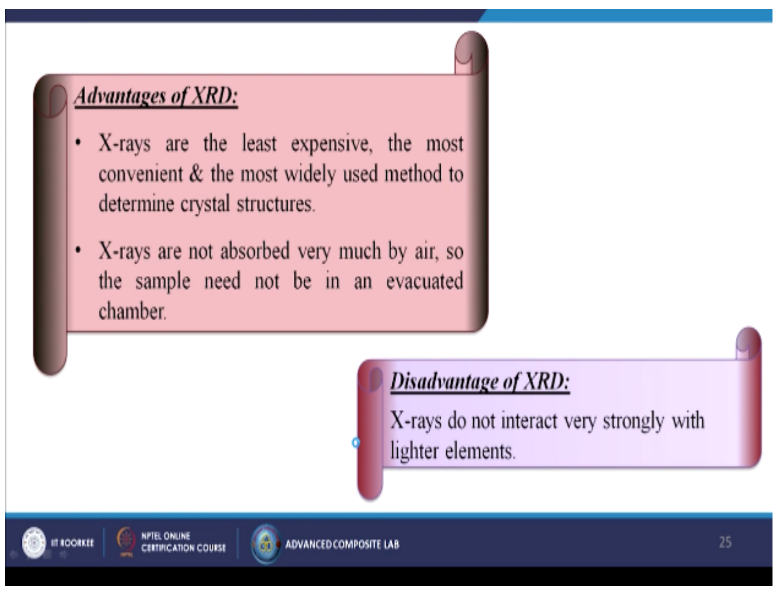

So now we are going to discussed about the advantage of the XRD, so X-rays are the least expensive, the most convenient & the most widely used method to determine the crystal structure X-ray are not absorbed very much by air, so the sample need not be in a evacuated chamber, but of course there are been certain disadvantages also so X-ray do not interact very strongly with lighter elements.

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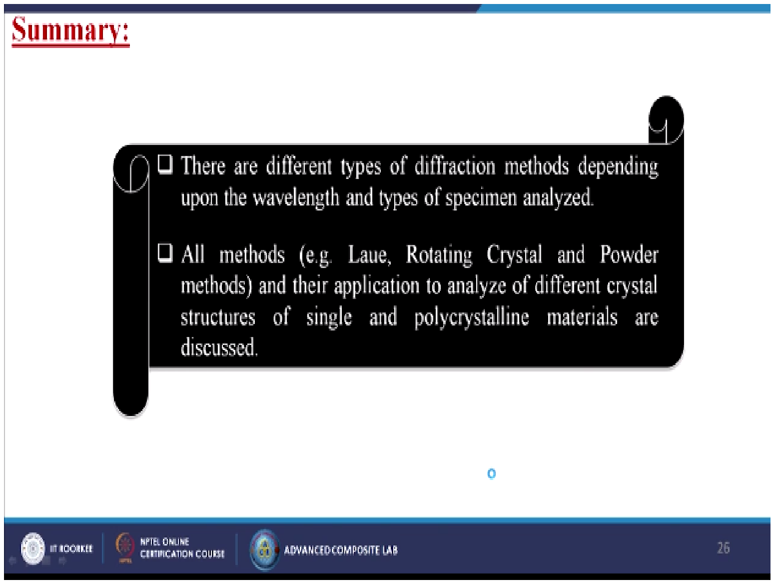

So now we are going to the whole study of this particular lecture, so there are different types of diffractions methods already we have discussed depending upon the wavelengths and the types of specimen analyzed say for single crystal or may be for the powder samples all methods example like Laue methods Rotating Crystal, and Powder Methods and their applications to analyzed of different crystal structure of single and for the polycrystalline because we have discussed about the powder samples which we are thinking that is a combinations of different so many types of single crystals materials has been discussed in this particular lecture, Thank you.

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