X-Ray Crystallography

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Lecture - 29
Stress Analysis by X-Rays

In this lecture, I will deal with stress analysis by X-Rays. X-Rays provide a very elegant method of stress measurement in a material. Whenever there is a homogeneous, elastic deformation in a material, we find that when a particular set of planes within a grain in that material are properly arranged for taking a diffraction pattern, there is a change in the inter planar distances in that set of planes from the value in the unstressed body.

Now, this change in the value of the inter-planar distance will give us a measure of the stress that has been put on the material. Now normally in the X-Ray method we do not measure stress directly. We measure the strain and then from the strain, we calculate the stress. Now before I get into the techniques of stress measurement by X-Rays, it will be better to deal with the fundamentals a little bit.

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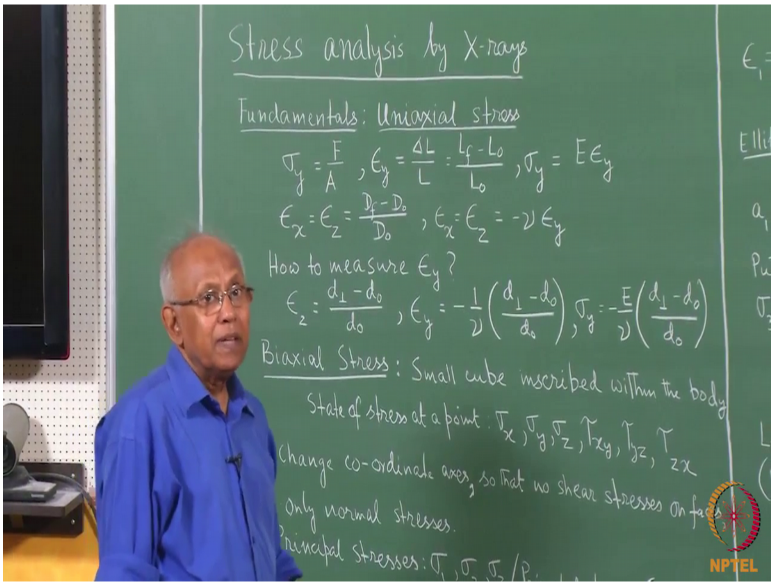

Say for example, if we have uni-axial stress as shown over here; say this is a cylindrical rod of diameter; D and length; L; say for example, we have a force; F acting in these directions as shown.

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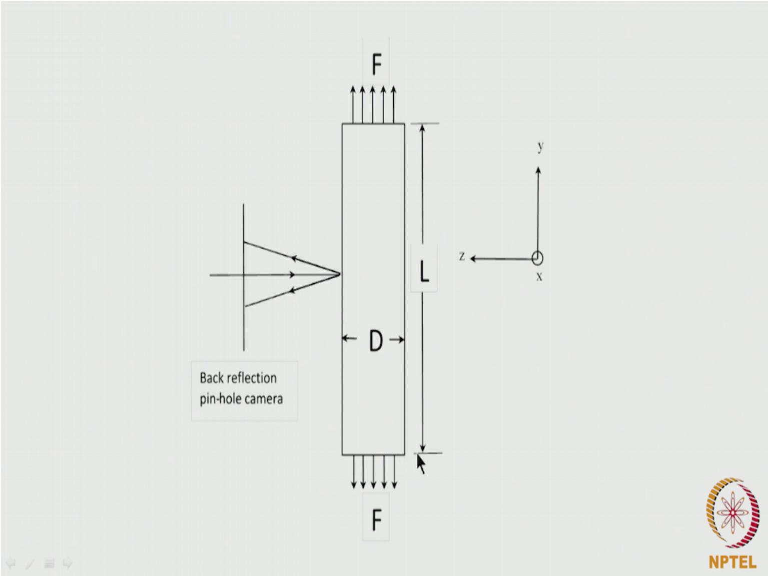

In that case, the stress will be equal to F by A. A is a area of cross section of the rod then strain in the y direction. This is the y direction; strain in the y direction will be equal to delta L by L which nothing is, but the final length minus the initial length divided by the initial length.

So, from this strain, we can calculate sigma y as E epsilon y is the Young's modulus. Now you see if we want to measure the stress in the y direction, then the methods could be that we have some gains within this rod over here and there are planes lying a perpendicular to the direction of F. So, if we have a grain over here with atomic planes lying in this fashion you know perpendicular to the direction of F, then what will happen due to the tensile stress; the inter planar distance will increase there will be an expansion in this direction.

So, because of the expansion the inter-planar distance will increase and if we can measure the inter-planar distance after applying the force and also can measure the inter-planar distance when there is no force on the body then we can calculate the strain in the y direction. However, the problem is when the rod is under tension as shown over here;

we cannot measure the inter-planar distance of the planes lying perpendicular to the direction of F because it will be difficult to put an X-Ray equipment over here.

So, instead what we do we try to figure out the variation of the inter-planar distance from grains which have atomic planes lying parallel to the side of the rod in this manner and then if we have a beam of X-Rays incident on these planes, then by the back reflection by measuring the back refection diffraction pattern, it is possible to figure out what are the values of the inter planar distances.

In fact, we can measure the strain in this direction and from that we can find out the strain in this direction. And finally, calculate the stress in this direction. So, this is exactly what we do. So, we know that epsilon x; the strain in the x direction will be equal to the strain in the z direction because it is a cylindrical body which has been under a force along the y direction and that should be equal to D F minus D 0 by D 0. D F is the diameter; the final diameter over and D 0 is a original diameter of the rod.

So, epsilon x will be F equal to epsilon z and this will turn; will be equal to minus nu epsilon y nu is the Poisson's ratio of the material which is being tested now. So, how we measure epsilon y? I have already said the problems of measuring epsilon y directly. So, what we do? We measure the value of epsilon z by finding out the value of the inter-planar distance of a series of atomic planes in a grain of the material; you know by having the X-Ray beam falling perpendicularly on those planes and then subtract D 0; the inter-planar distance in the unstressed material divided by D 0 again. So, the situation is like this.

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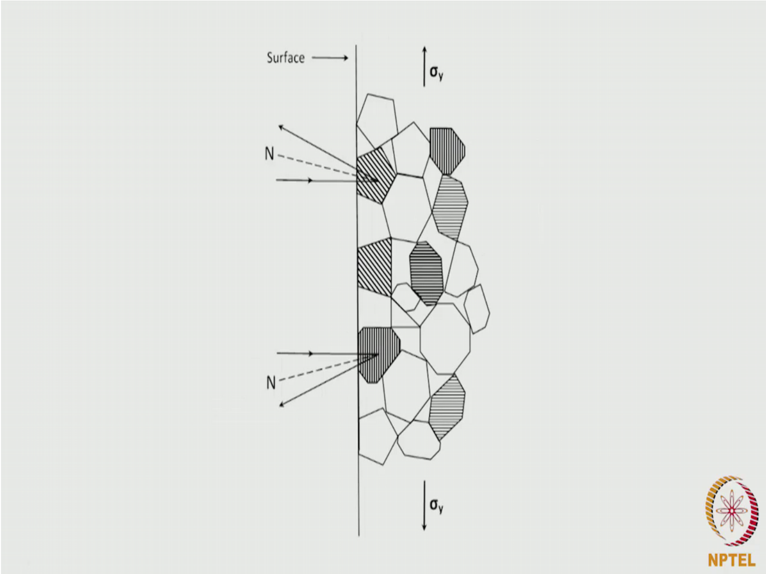

So, if we have some grains within the material, where the atomic planes are more or less parallel to the side of the sample and if we have a beam of X-Rays falling more or less perpendicularly over; you know it will make an angle small angle, but it will still be called a normal incidence.

And if we can get the diffracted beams like this; this is the normal to the planes, this is the incident beam, this is the diffracted beams although; it will be making some angle. We can consider it to be a normal incidence of X-Rays. So, by having the diffracted beam from this grain we can figure out; how much is the value of the inter-planar distance when the material is under load. So, that is the value; we call the perpendicular here and then have the body in the unstressed condition and form the same place find out what is the value of D 0.

So, epsilon z will be equal to D perpendicular minus D 0 by D 0. So, epsilon y which is you know related to epsilon x and epsilon z by this equation can be found out as minus 1 by nu into D perpendicular minus D 0 by D 0. So, once we get epsilon y the strain along the y direction, we can find out the value of sigma y; the stress in the y direction as minus E by nu D perpendicular minus D 0 by D 0. So, you say that x is provided an elegant method of stress analysis by finding out the inter-planar distance of a set of atomic planes in a particular grain which is properly arranged within the material. So, that we can find out that value of the inter-planar distance in this stress condition and also in the unstressed condition and knowing these 2 values; it is possible to figure out the value of epsilon y and from that you can calculate the value of sigma y.

In actual material; the stress condition may be quite complex; may be biaxial or tri axial stresses can exist in over there. Now you see the normally the technique that we use is what is known as a back reflection we know all method which I will come to later on. So, what essentially we do is in the; we all know about the arrangement of the specimen and the film and the X-Ray machine in case of a back reflection pinhole method.

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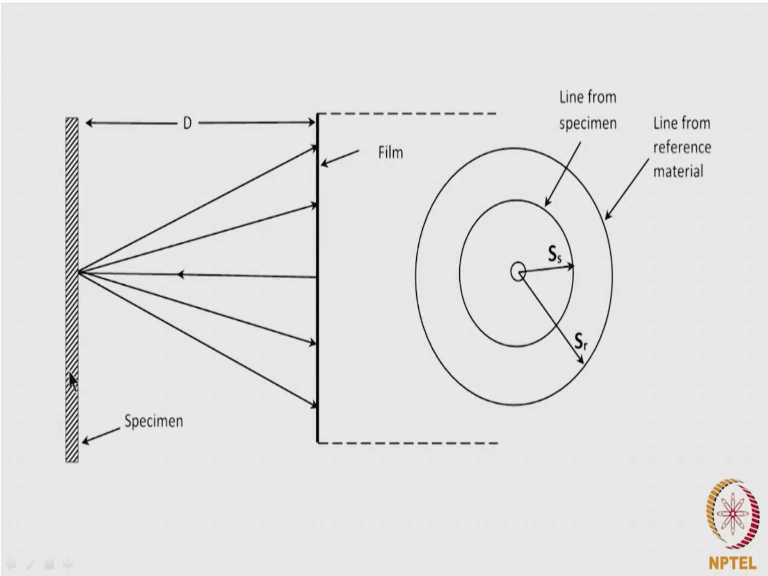

What happens in a back reflection pinhole method is we have got our specimen and X-Ray beam is allow to fall on the specimen in this direction then; we have got the recording film. Now whatever is recorded on the film is laid out in this portion and we can see what the; you know recording looks like the X-Ray beam is incident from this side through this pinhole and strikes the sample at this point.

So, what normally we do is we smear powder of a known reference material on the surface of the sample and then take a back reflection pinhole pattern from the composite. So, what will happen? We will get the Debye ring from the specimen as well as we will get the Debye ring from the reference material for which the lattice constant and everything are known. Now provided we know the value of D the distance between the specimen and the film just by knowing the; you know displacing of the reference material from this recorded Debye ring for the reference material, we can easily find out what should be the displacing for the specimen you know by measuring the radius of the Debye ring.

So, by comparison with the reference material for which the lattice constants are known we can find out from the Debye ring of the actual specimen what should be the lattice parameter lies.

So, this will be an accurate measurement of the lattice parameter. So, this is what we do over here. Now I will go to bi-axial and tri-axial stresses which normally we find in case of an actual material. In fact, in an actual material, if we inscribe a small q within the material; the state of stress at a point will be given by these components.

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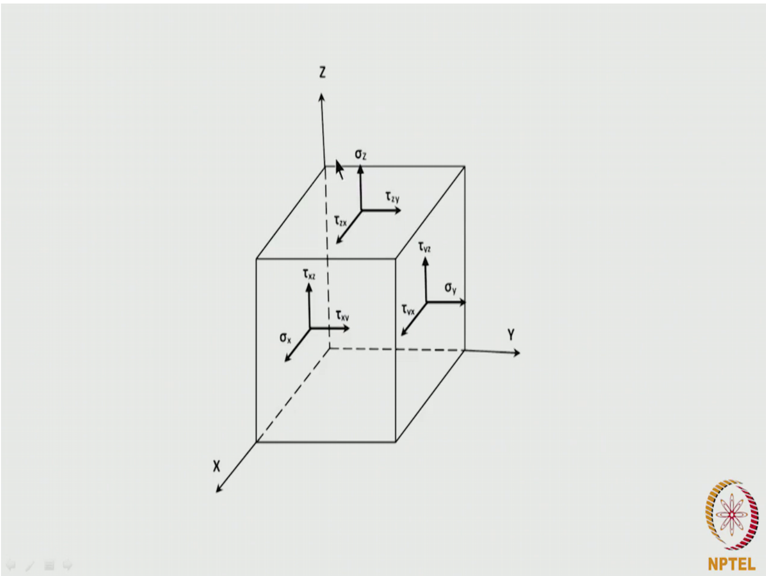

Say for example, on each phase of the cube inside within the material we will find; there will be a normal stress acting perpendicularly to the surface and there will be 2 shear stresses given by that and this will be true for all the phases. Here for example, this is sigma z. Here it is sigma y. This is sigma x and you have got all the shear stresses tau z x, tau z y, tau x z, tau x y, etcetera, etcetera. Now if we consider all this 6 phases, we will see that there will be some of these stresses are equal to some other stresses within the on the surfaces. As a result, the stress at a point within the material can be given by only 6 stresses. So, if we know the values of sigma x, sigma y, sigma z, tau x y, tau y z, tau z x, then the stress at a point will be completely described within the material.

Now, if we change the coordinate axis for example, here there will be a situation when there is no shear stress present on the surfaces and there will be only normal stresses. So, it is possible to change the co-ordinate axes of this cubic body from x, y, z to some other. So, such that they are only shear stresses present on the surfaces; only normal stresses. Now in that case, we say that the normal stresses will be sigma 1, sigma 2 and sigma 3 along the axes 1, 2 and 3. So, under that condition, the state of stress will be given by only 3 principle stresses; sigma 1 sigma 2, sigma 3 and accordingly the principle stress will be say epsilon 1, epsilon 2 and epsilon 3.

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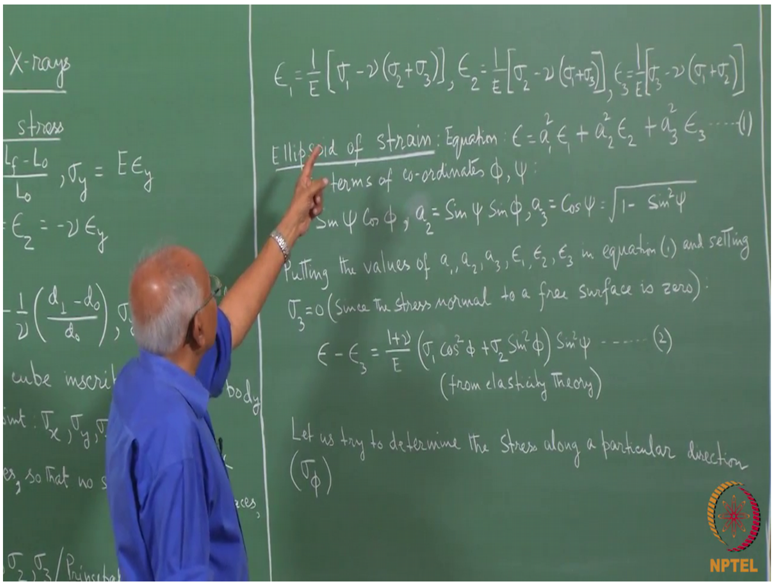

We can write down the principle strains in terms of the principle stress values as shown here. So, epsilon 1 will be 1 by E, The Young’s modulus sigma 1 minus nu Poisson’s ratio sigma 2 plus sigma 3 sigma; epsilon 2 will be 1 upon E sigma 2 minus nu into sigma 1 plus sigma 3 and epsilon 3 will be 1 upon E sigma 3 minus mu into sigma 1 plus sigma 2.

Now, we can talk about what is known as an ellipsoid of strain. Say for example, if there is a homogeneous elastic deformation of a material; then a sphere within a material; sphere element within the material will be turned into an ellipsoid and the equation for that ellipsoid of strain is epsilon equal to A 1 square epsilon 1 plus A 2 square epsilon 2 plus A 3 square epsilon 3. So, this epsilon is any strain in any specified direction and A 1, A 2 and A 3 are the direction cosines of that particular direction with the directions of epsilon 1, epsilon 2 and epsilon 3.

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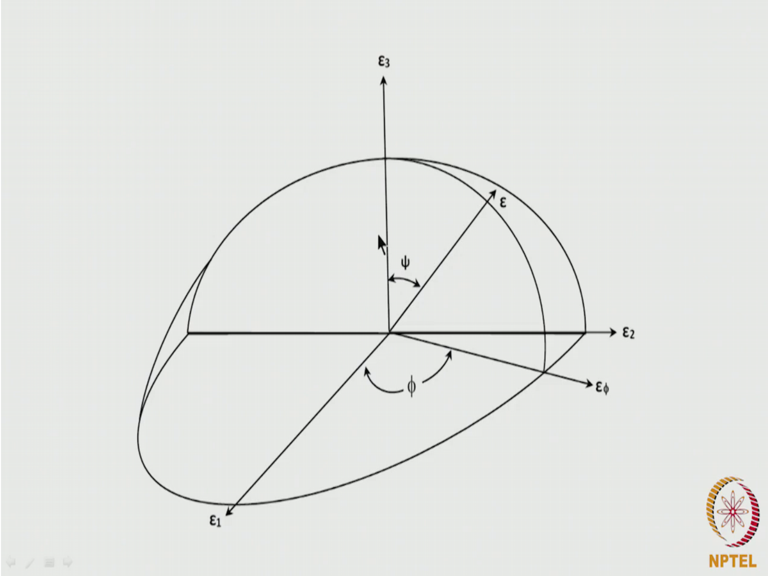

Now, if we look at the situation here. So, this is a ellipsoid of strain. So, this these are directions of epsilon 1, epsilon 2 and epsilon 3; say we are considering a direction like this for epsilon and this is making an angle psi with epsilon 3 and you know this particular direction. Here epsilon phi makes an angle phi with epsilon 1. Now in that case, this equation will be valid. So, if we look at the direction of epsilon here and the value of epsilon will be given by this.

And now in terms of the coordinates as we have shown here; the psi and the phi angles in terms of this coordinates A 1, A 2 and A 3 can be written in this fashion. For example, A 1 can be written as sin psi cosine phi. A 2 will be equal to sin psi sin phi and A 3 will be equal to cosine psi. So, will root over 1 minus sin square psi

Now, if we put the values of A 1, A 2, A 3 and epsilon 1, epsilon 2, epsilon 3, in equation 1 and also put the value of sigma 3 is equal to 0; as we know that the stress normal to a free surface is 0 then equation 1 becomes epsilon minus epsilon 3 equal to 1 plus nu by E into sigma 1 cosine square phi plus sigma 2 sin square phi. The whole thing multiplied by sin square psi or equation number 2. This is from; this can be derived from the elasticity theory. Now; our job now is to try to determine what should be the stress along a particular direction as we have shown here say for example, you know along a particular direction what will be the value of sigma phi.

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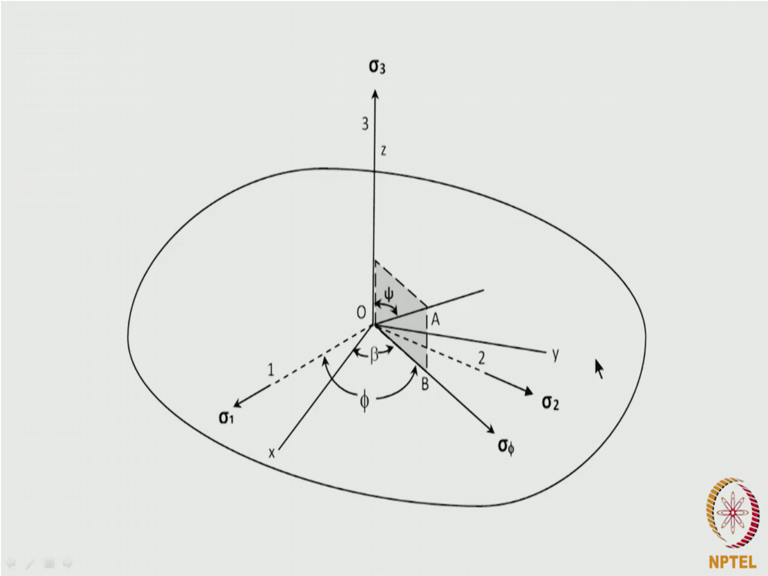

So, this is what we are trying to find out. Now let us suppose that we have a free surface as shown over here and the angular relationships between x, y, z and the direction 1 for sigma 1, 2 for sigma 2 and 3 for sigma 3 are all shown here. Let us suppose that we want to find out the value of the stress sigma phi lying in this plane and also lying on the surface. So, we have got this direction which is the direction of sigma phi; say we want to figure out the value of sigma phi stress. In this particular direction and the direction is specified by the angles phi as well as psi.

Now, how to find out the stress in this particular direction that is what we are going to figure out now just as we saw the equation for a strain ellipsoid we can write down the equation for a stress ellipsoid in this fashion where sigma equal to A 1 square sigma 1 plus A 2 square sigma 2 plus A 3 square sigma 3, A 1, A 2 and A 3 just as before are the direction cosines of the direction of sigma with respect to the directions of sigma 1, sigma 2 and sigma 3.

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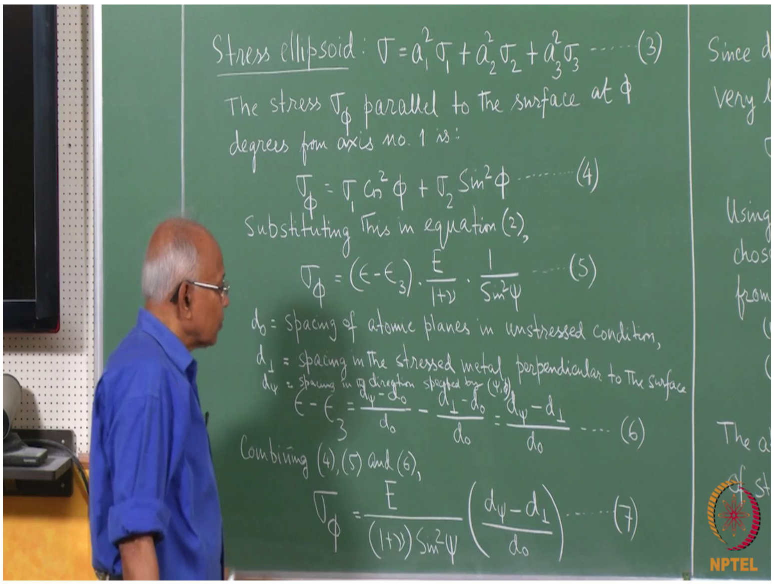

So, this is our equation 3 now the stress sigma phi parallel to the surface at phi degrees from the axis number one; that means, this one over here is sigma phi equal to sigma 1 cosine square phi plus sigma 2 sin square phi. This is our equation 4. Now if we substitute this in equation 2 then you find sigma phi is equal to epsilon minus epsilon 3 into E divided by 1 plus nu; the whole thing multiplied by 1 upon sin square psi. This is our equation phi.

Now, say for example, if we take D 0 as the spacing of atomic planes in a suitable grain in the unstressed condition and D perpendicular is the spacing in the stressed metal perpendicular to the surface and D psi; the spacing in the direction specified by psi phi then we can write down this equation you know as epsilon minus epsilon 3 will be equal to D psi minus D 0 by D 0. This is the value of epsilon minus epsilon 3 can be written as D perpendicular minus D 0 by D 0 that it equal to D psi minus D perpendicular by D 0.

So, essentially what we are doing here? if this is the free surface; if this is the free surface and if we have the x X-Rays incident on the surface in a perpendicular fashion and if from a suitable grain with the atomic planes; we can measure the inter-planar distance then that will be D perpendicular. Now if we do the same thing in the material when the material is in the unstressed condition; then it will be D 0 and if again we allow the X-Ray beam to be incident on the surface in this direction as shown over here; then that will give us the value of D psi the spacing of that we planes in that particular direction specified by psi and phi.

So, I will repeat in that case; what will happen? The epsilon will be the epsilon here is the strain corresponding to this condition defined by psi and phi. So, this epsilon here will be equal to D psi minus D 0 by D 0 and epsilon 3 will be equal to D perpendicular minus D 0 by D 0. So, after subtraction it becomes D psi minus D perpendicular by D 0 this is our equation 6.

Now, if we combined the equations 4, 5 and 6 then we get sigma phi equal to E divided by 1 plus nu sin square psi the whole thing multiplied by D psi minus D perpendicular by D 0. This is our equation 7.

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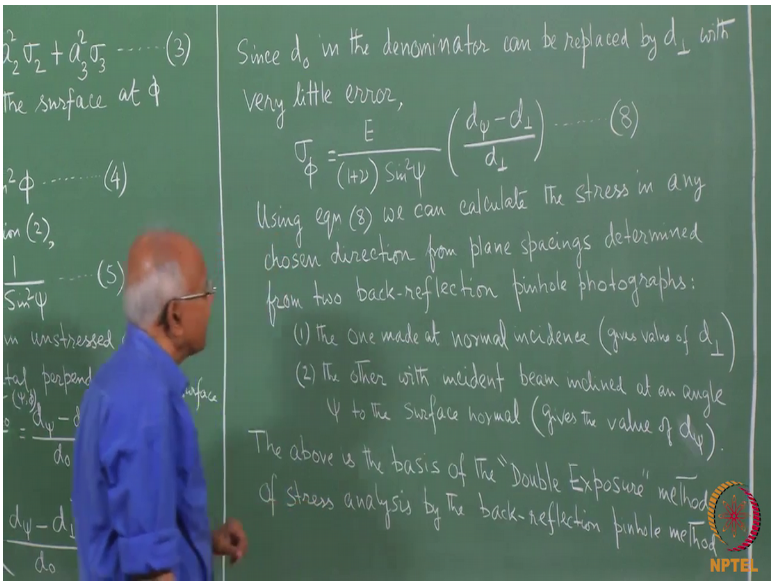

Now this D 0 in the denominator can be replaced by D perpendicular with very little error. So, this equation 7 can then be rewritten as sigma phi is equal to E upon 1 plus nu sin square psi. The whole thing multiplied by D psi minus D perpendicular divided by D perpendicular. This is our equation A. Now this is a basic equation. This equation 8 is the one by using which we can calculate the stress in any chosen direction from the plane spacing determined form 2 back reflection pinhole photographs as I have already mentioned.

And what were those the one might at normal incidence the one might with normal incidence perpendicular to the surface; which will give us the value of D perpendicular the other with incident beam inclined at an angle psi to the specimen surface. So, what we can do? We can make 2 back reflection pinhole photographs 1 by allowing the X-Ray beam to fall on the surface in a direction perpendicular to it and that will give us the value of the perpendicular and again a second back reflection pinhole X-Ray exposure in which the X-Rays are incident on the surface at this particular angle psi and that will give us the value of D psi.

Now, putting the value of D psi and D perpendicular over here; it is possible to figure out what will be the value of sigma phi because you know psi is a known value. In most of the cases, we keep it at 45 degrees. So, whatever the angle of psi that is kept 45 degree; so, this value will be known nu is the Poisson's ratio that is also known. E is a elastic modulus that is also known.

So, we can find out the value of the stress in a particular direction phi. From this equation, simply by doing I by having 2 back reflection pinhole photographs in one case; the photograph X-Ray photographs made by having the X-Ray incident in a normal direction or to the surface and the second case by having the X-Ray incident at an angle psi to the normal of the surface considered.

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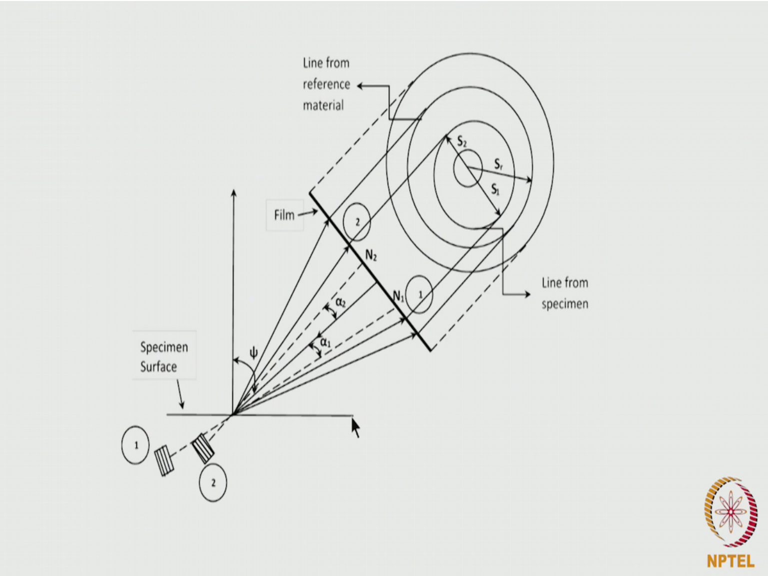

So, from these 2 values, it is possible to figure out what should be the value of sigma phi. Now the above is the basis of what is known as the double exposure method of stress analysis by the back reflection pinhole method.

The figure here illustrates the set up for the double exposure method. Say for example, this is the specimen surface and this is normal to the specimen surface. So, what we do? we take the experimental stressed material and smear on that stressed material powder of a reference material. So, that when you do the back reflection pinhole photography we will have Debye rings from both the stressed material and the known reference material for which the lattice constants and other parameters are known. So, in that case, what happens is we do 2 exposures the first one by giving the X-Ray, then allowing the X-Rays to fall normally on this specimen surface in this particular direction. So, the first exposure is by allowing the X-Rays to fall on the specimen surface in this direction normally and prepare the pinhole photograph.

In the second case we allow the X-Rays to be incident at an angle psi to the normal to the surface as over here. So, X-Rays are allowed to fall in a direction at a direction psi to the normal to the specimen surface this is the film and on the film whatever you get is showed over here on a magnified scale. So, this is the pinhole through which the X-Rays come and go through and falls on the specimen now this particular ring dividing is due to the specimen sake and this particular Debye ring is from the reference material.

Now, if we look at the Debye ring from the specimen, we will find that it is not perfectly circular. The reason is this; you see when we do this incidence angle pinhole photography; then say for example, we have got 2 series of atomic planes. Now this is one series and this is another series. Now what happens is the strain in the direction normal to the atomic planes will vary with the angle from the normal direction. So, you say that in this particular case and in this particular case; the strains will not be the same and in fact, we will see that in this particular case, here you know when we talk about this particular case the plane normal is given by n 1 and in fact, this plane normal will not be at an angle phi, but at an angle phi plus alpha 1.

Again on the other hand when we talk about this series of planes, in another grain giving raise to the back deflection pinhole pattern you know the normal is n 2 and this is making an angle phi minus alpha 2 with the specimen normal now because of this we are not going to have a perfectly circular Debye ring from the specimen. So, in this case we will find that the measurement of the radius of the Debye ring S 1 is not going to be the same as S 2. So, normally when we want to measure the Debye ring radius; we measure it from S 1 because from this side we know the value of S is more susceptible to strain. So, this is much more sensitive to sensitive to stress or strain and that is a reason why we always measure the Debye ring from the specimen from S 1.

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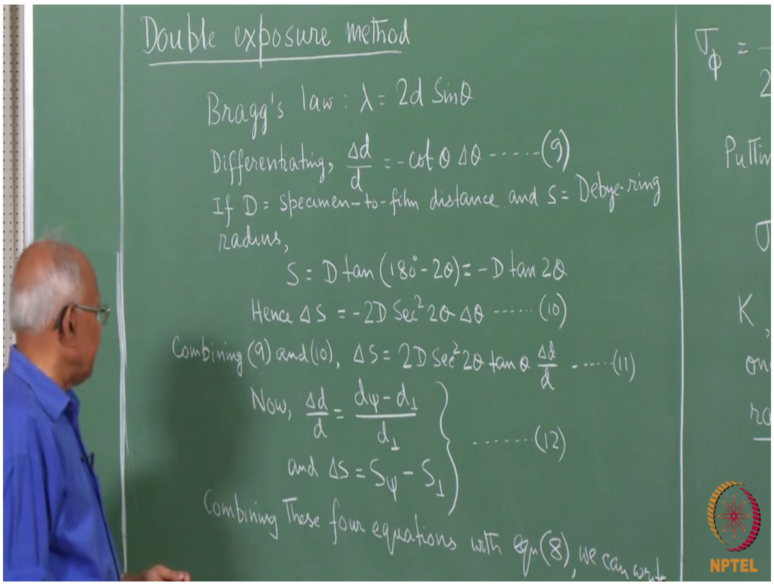

So, this is what we do now if we go back to Bragg's law lambda is equal to 2 D sin theta now differentiating we get delta D by D this will minus cotangent theta delta theta our equation nine now if D is the specimen to film distance and S is the Debye ring radius then you can write for the back deflection pinhole method S is equal to D tan 180 degree minus 2 theta is equal to minus S tan 2 theta hence delta S will be equal to you know by differentiating minus 2 D sec square 2 theta delta theta this is our equation 10.

Now, if we combine the equations 9 and 10 then delta S is equal to 2 D sec square 2 theta tan theta delta D by D this is our equation eleven now we already know that delta D by D will be equal to D psi minus D perpendicular divided by D perpendicular and delta S is equal to S psi minus S 1; now combining these 4 equations with equation 8.

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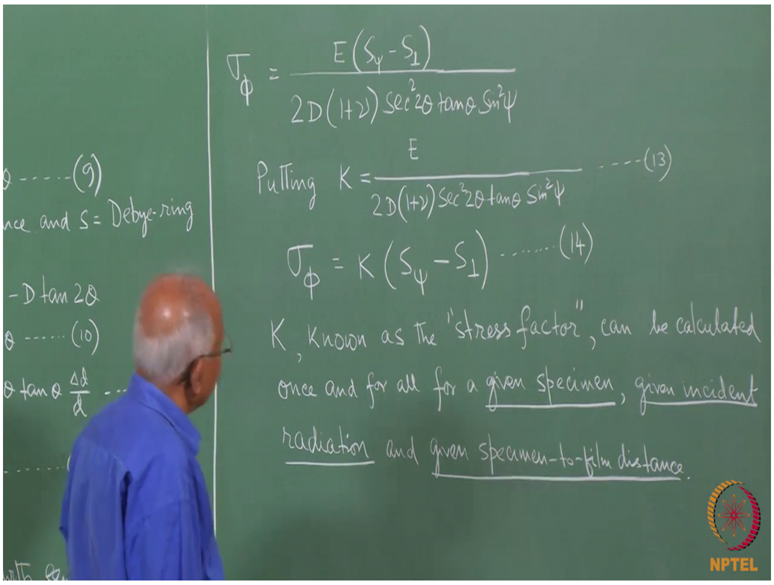

We can write it down as sigma phi will be equal to E multiplied by S psi S psi means what S psi means the Debye ring from the specimen during inclined incidence x radiation. So, this is the situation when we are having an inclined incidence photography that refers to the radius if the Debye ring the minus S lambda what is S lambda S lambda stands for the Debye ring from the specimen when there is normal incidence photography from the material. So, now, when we have a normal incidence photography; that means X-Ray falls on the surface in a normal way and we produce the Debye ring. So, S perpendicular refers to that and S psi refers to the value of this S 1 over here during inclined incidence X-Ray photography.

Now, if we put K is equal to E by 2 D 1 plus nu sec square 2 theta tan theta sin square psi you see because all the quantities are fixed. So, if we do that we can write down sigma psi phi is equal to K multiplied by S psi minus S perpendicular equation 14. So, this is basically the most important equation in order to find out the stress in a particular direction in a stress material by doing back deflection pinhole photography in 2 different ways one by you know the normal incidence another by inclined incidence of X-Rays. So, you say that here if we measure the Debye ring radius S psi.

So, in this case as I already said the Debye ring radius is normally shown by measuring the S 1 because this side you know is more susceptible to strain and that is a reason why you know S psi is nothing, but this value S 1 and exact value of S 1 can be easily found out with comparison with the line from the reference material for which the lattice parameters are already known. So, we can accurately measure S 1 by comparing with S R and that value is put over here and S perpendicular is the value of the Debye ring which is obtained by taking a normal incidence x radiation and measuring the value of Debye ring radius they are from.

Now, this K is known as the stress factor and this can be calculated once and for all for a given specimen a given incident radiation and given specimen to film distance once we do it; we can use it over and over very often the double exposure method of stress analysis by back reflection pinhole method is not used instead we can have a single exposure method. So, to say which takes much less time than the double exposure method now in the single exposure method we get rid of the pinhole photograph using the normal incidence of X-Rays. So, if this is the specimen surface in the single exposure method; we do not use the normal exposure normal incidence of X-Rays, but instead we have only one pinhole photograph using the inclined incidence of X-Rays. So, in this particular method it is necessary to measure not only the value of S 1, but also the value of S 2,

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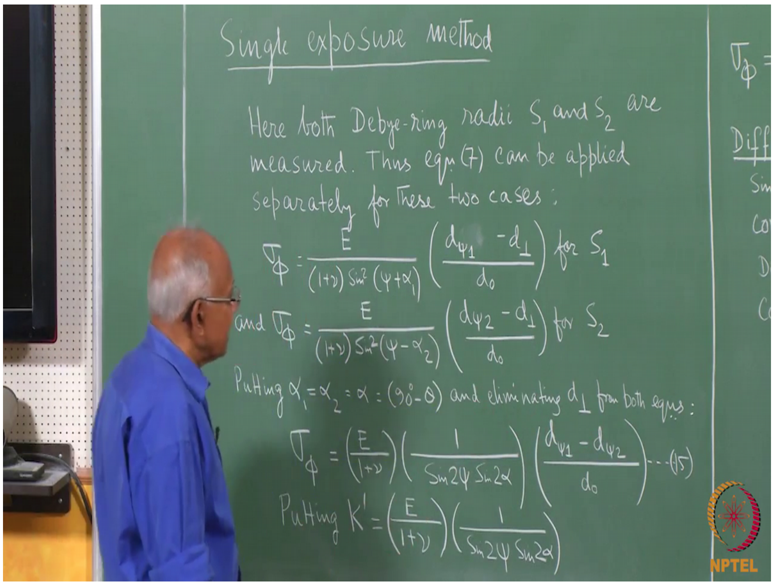

Now if that is the case then equation 7 can be applied separately for these 2 cases of S 1 and S 2.

Therefore we can write sigma phi equal to E divided by 1 plus nu sin square psi plus alpha 1 as we can see here for S 1 the angle; angle from the specimen normal is psi plus alpha 1 then D psi 1; that means, the inter-planar distance which has been measured from S 1 side minus D perpendicular divided by D 0 you know the equation 7 can be written in this manner for S 1. We will see that the D perpendicular will be eliminated. So, that you know there is no need of having the normal incidence X-Ray method and sigma phi for the second case for S 2 will be E by 1 plus nu sin square psi minus alpha 2 because here the angle from the plane normal is this. So, it will be psi minus alpha 2 multiplied by D psi 2; that means, the value of inter-planar distance calculated from the S 2 side minus D perpendicular by D 0, this is for S 2. Now since alpha 1 alpha 2; these are very small; we can write alpha 1 equal to alpha 2 equal to alpha and this is equal to 90 degree minus theta the Bragg angle.

And if we eliminate D perpendicular form both these equations, then we find the sigma phi can be written as E by 1 plus nu; the whole thing multiplied by 1 upon sin 2 psi sin 2 alpha into D psi 1 minus D psi 2 by D 0. This is our equation 15. Now if we put K prime equal to E by 1 plus nu multiplied by 1 upon sin 2 psi sin 2 alpha; you know all these things can be calculated and we can find out the value of K prime; we can write sigma psi is equal to K prime multiplied by D psi 1 minus D psi 2 divided by D 0

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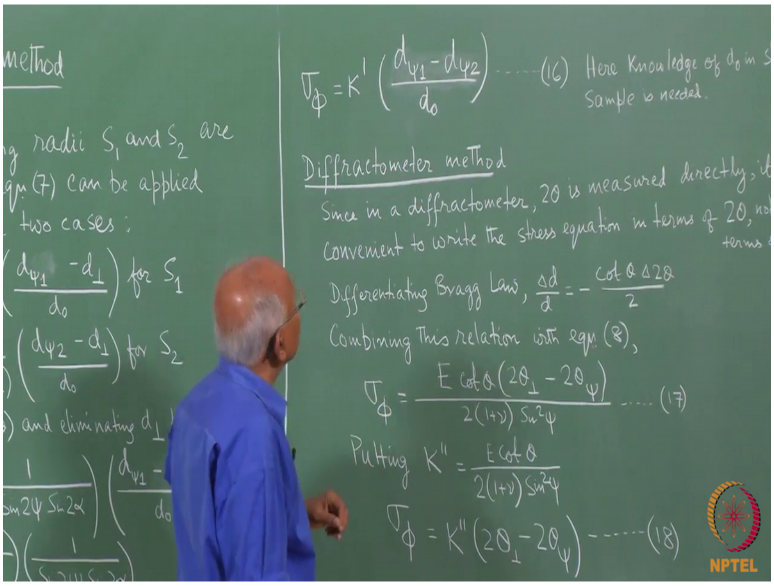

So, in this method, we need to know the value of D 0 in order to find out the value of sigma phi this is important; that means, D 0 is the plane spacing in the unstressed material. Normally if we know the lattice constants of the material it is possible to figure out the value of D 0 in any case. So, the advantage of this method is here; you know the total time taken for the measurement is roughly half of the time taken in the double exposure method because here we are not carrying out the normal incidence X-Ray back reflection pinhole photography we are using only the inclined X-Ray method for the back reflection pinhole photography.

Therefore this methods single exposure method about 50 percent of the time taken in the double exposure method, but the error in the single exposure method is 2 to 3 times the error that we encountered in the double exposure method. Normally if we use a steel sample by double exposure method; the amount of error is about plus minus 32; 35 MPA for steel samples, but as we can see that in the constant K values you know; there is always this factor E the Young's modulus. So, naturally the error will be much less for a material which has lower Young's modulus value; therefore, say if we take aluminum alloys the error in the measurement of stress by the double exposure method will be much less than in case of measurement on steel samples. So, to say that is another method by which we can measure stress and that is the diffractometer method.

Now, since in a diffractometer 2 theta is measured directly it is convenient to write the stress equation in terms 2 theta not in terms of D. So, if we differentiate the Bragg law delta D by D is minus cotangent theta delta 2 theta by 2 combining this relationship with equation 8, we can write sigma phi is equal to E cotangent theta 2 theta perpendicular minus 2 theta psi divided by 2 into 1 plus nu sin square psi. Now you see due to the presence of stress in the material; you know they are; there will be a shift in the position of the lines; you know there is a change in the D values and that will automatically lead to a shift in the 2 theta values. So, this is A 2 theta value when we do it under normal incidence or X-Rays and this is A 2 theta value which we find out under the inclined incidence condition.

Now, if we put K double prime is equal to this value which can be calculated; we can write the sigma phi is equal to K double prime 2 theta perpendicular minus 2 theta psi. So, in this case in the difractometer method it is simply to figure out what is the; you know what is the shift in the value of the 2 theta angle.

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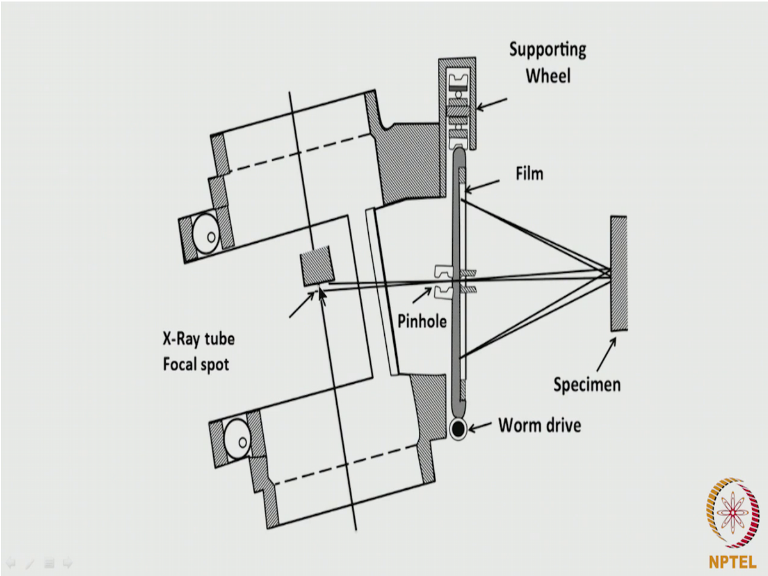

Now, here you know it is a schematic of a back reflection pinhole camera this is the X-Ray tube focus spot X-Ray tube this is a pinhole this is the specimen and this is the film. So, the back reflected rays are obtained in this manner.

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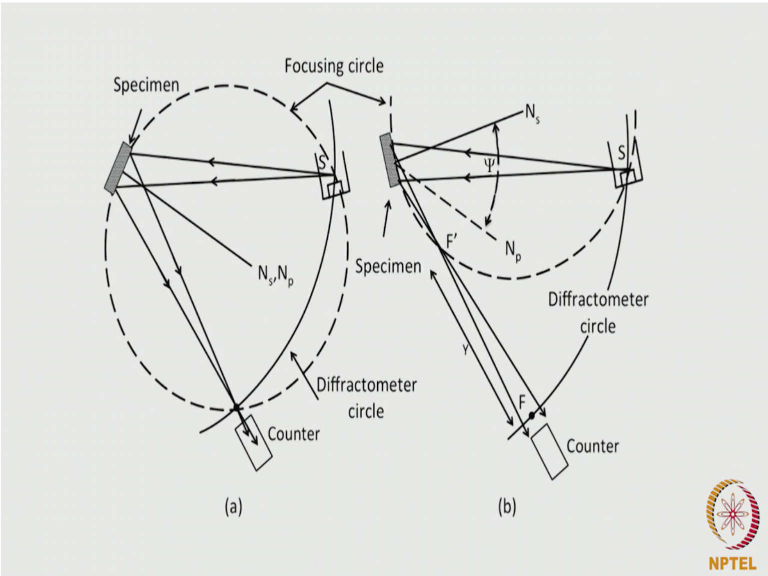

So, this is you know the schematic of a back reflection pinhole camera and this shows the angular relationships between the specimen the counter you know the focusing circle etcetera in the 2 cases.

Here this is for in the diffractometer; here in the diffractometer, this is the condition you know for normal incidence diffractometry and this is for the inclined a incidence diffractometry. So, we can see here under normal incidence; if this is the specimen and this is the diffractometer circle; you know, this is the focusing circle focusing circle is always tangent to the specimen and this is the location of the counter. So, in the normal incidence method you know the perpendicular to the specimen and perpendicular to the plane which is diffracting you know it is the same.

But what happens when we have the incidence you know inclined incidence X-Ray diffraction there the specimen is rotated by an angle psi as we can see here. So, the N P the normal you know and the N S N P; the normal to the plane and N S the normal to the specimen they are quite different here. So, N S changes by an angle phi. Now in this case, the diffracted beam you know gets focused at F phi you know much before it reaches the counter. So, what normally is done you know; if in this particular case, if it is focused at F prime then in the counter; the intensity will be much less. Now in order to avoid that we can use a bigger slit here, but in that case the resolution will be very poor.

So, the ideal case will be; if we have a small slit over here and you can put the counter some over above there, but normally what is done we put the slit somewhere in between F dashed and f. So, that will you know in that method we make a compromise between the resolution and the intensity. So, this is this explains as I said how stress is measured by the diffractometer.

Now coming to a conclusion; stress measurement by X-Rays is quite useful when we deal with lot of high localized stresses in a material which varies over a distance from place to place. There X-Ray method is very useful compare to the; you know electrical or mechanical gauges. In fact, for residual stress measurement the X-Ray method is undoubtedly better than the electrical and the mechanical strain gauges.