Texture in Materials
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Module - 04
Principles of texture measurements by X-ray diffraction
Lecture - 26
Texture Goniometer Components

Good afternoon everyone. And, today we will continue with module 4 a Principles of Texture Measurement by X-ray diffraction. In the last lecture we saw that, how we can measure alpha and beta?. And, obtain intensities at various alpha and beta point to measure the whole pole figure for a particular HKL plane. In this lecture this is lecture number 26; we will show various texture Goniometer components.

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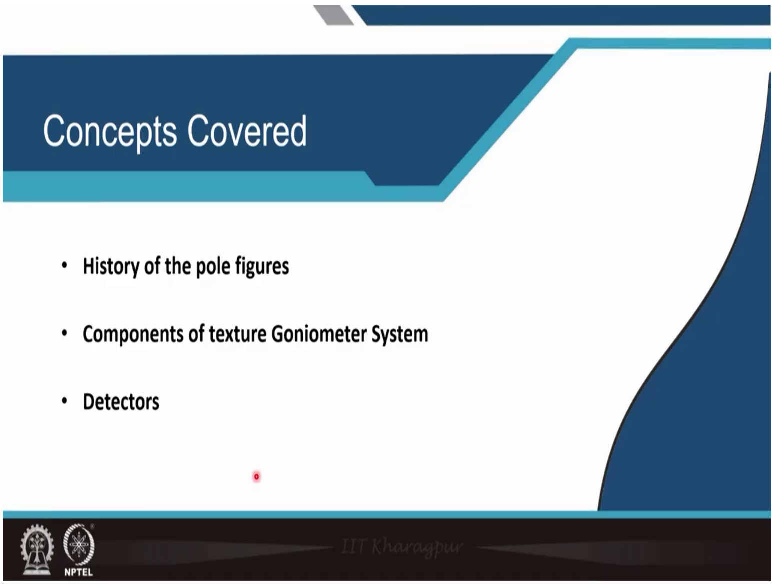

So, the concepts that will be covered in this lecture are history of the pole figure development. And, then we will carry out with various components of texture goniometer system and then we look into detectors in little detail.

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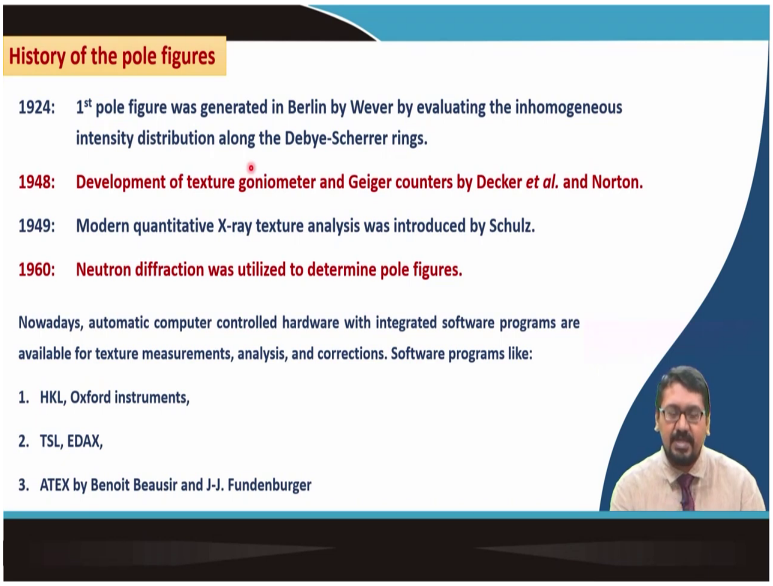

So, when we look into the history of the pole figures. It was Wever, which in 1924 generated the 1st pole figure in Berlin University in Germany. So, he did it by evaluating the inhomogeneous intensity distribution along the Debye-Scherrer rings. In 1948 Decker and Norton separately they develop , they did it separately, they develop the texture goniometer and the Geiger counters.

So, Geiger counters are the the phosphor screens in which the intensity of the diffracted texture intensity beams are measured. So, in 1949 modern texture analysis method by Schulz was introduced and we discussed in very detailed, what is Schulz reflection method for pole figure measurements using X-rays. So, in 1960 neutron diffraction was introduced and was utilized to measure pole figure for transmission geometry goniometer geometry right. And, nowadays computer controlled and automated software's are present which are in in sync with hardware. So, hardware and integrated software programs are there, which are available for texture measurements, their analysis and all the corrections that are needed to be done.

Basically while we do this kind of texture analysis. I will we use basically HKL Oxford instruments software, which is HKL or TSL, EDAX, and we always use ATEX, which is a free software given by Benoit Beausir and Jean Jacques Fundenburger of University of Lorraine Metz France. And, at the same time there is another software, which I have not mentioned here forgot to mention here, that is the MTEX software to measure this X-ray diffraction, which is also a free software available in the internet. So, these are all different kinds of software tools, which are which can be used for the measurements mainly HKL and TSL can be used for measurements, because it comes integrated with the SEM and EBSD. And, most of both of them are mostly used for the ACME BSD case.

Whereas, for XRD texture measurements there are specific software's for from different companies like, panalytical Bruker or other companies. And, then this ATEX can be used for measuring both pole figures obtained from X-ray diffraction, and also from electron backscatter diffraction in an scanning electron microscope. And, even in I think they are developing it for the transmission electron microscope also. MTEX on the other hand are analysis software for X-ray texture pole figures.

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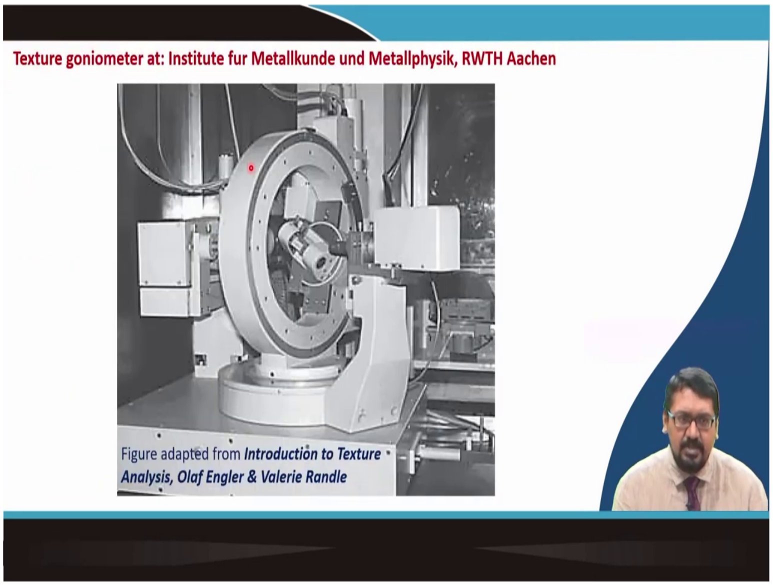

So, this figure is a typical texture goniometer of the history. So, this is a texture goniometer, this figure is adapted from the book of introduction to texture analysis by Olaf Engler and Valerie Randle. And, this is the texture goniometer which is basically was basically present in RWTH Aachen in Germany. So, institute of Metallkunde und Metallphysik. So, you can see that, how big the Eulerian cradle was and you can, we can observe that the detector and the the source of the X-ray beam and if you look into details of this texture goniometer.

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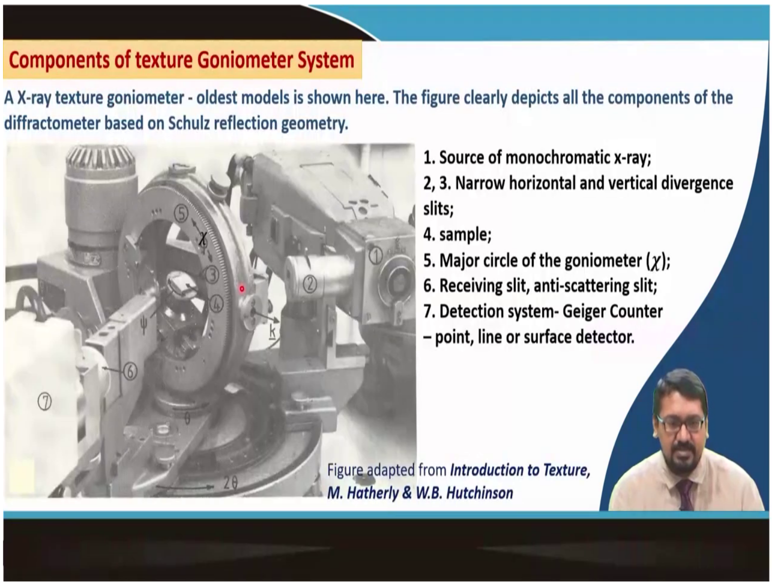

Here is another figure that, I would like to show and this figure is basically a picture taken and adapted from introduction of texture book by Hatherly and Hutchinson. So, this is basically a very old model, the may be the oldest model of texture goniometer. The figure can be used to clearly depict all the components in the in this diffractometer and this is the diffractometer, which is based on reflection mode, that is Schulz diffraction method right.

So, this part of the goniometer is basically the source of the monochromatic x-ray beam. And, so, the monochromatic X-ray beam basically comes out here and it goes to two slits, which might be present inside here. And, they are very narrow horizontal and vertical divergence slits. So, this goes and it goes to the sample which is kept here on the sample holder 4 right. And, you can see that it consist of a major circle, which is basically known as the goniometer circle and is given by zeta rotation right.

We must remember that in the reflection mode the zeta rotation is equal to 90 degree minus alpha, which is we actually done along T D. And, it is done zeta is the right hand thumb rule based rotation right. So, zeta rotation is there and then it goes, if the X-ray beam goes and hits the sample and it is diffracts and it goes to the detector here, the detector here. So, the detector system could be a Geiger counter and nowadays it could be a point detector or a line detector or a surface detector right. And, before it goes to the detector it has to pass through the receiving slits and anti scattering slits. So, that the width of the beam reduces.

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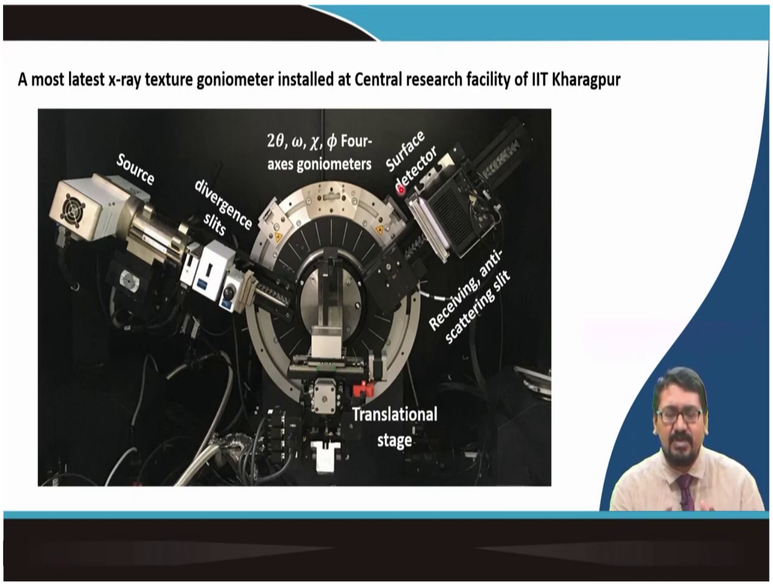

Now, this figure is the photograph taken of the most latest x-ray texture goniometer that has been installed recently in the Central Research Facility of Indian Institute of Technology, Kharagpur. You can see the difference between the oldest model and the newest model here. And, you can see that in the newest model, the source of the x-ray has become so, small and so, sophisticated. Of course, it has the divergence silts and the technique or the mechanism remains the same.

It has the 4 axes 2 theta omega zeta phi goniometer and you can see that. It has the sample holder with a a translational stage; once translational and in this direction and another translational outside and inside of the screen right. You can see the presence of the receiving and the antiscattering slit. And, in place of point detectors which was present in the old models. Now, surface detectors are being present which makes the pole figure measurement faster. So, with the advent of the technology the pole figure measurements as that we have to measure each pole figure HKL for a particular theta fixing omega in such a way. So, that for that particular theta making that omega equal to 0. And, then measuring that HKL plane like that all 2 or 3 or 4 HKL planes has to be measured.

And, all the measurements basically goes from 0 to 70 degrees and it cannot go from 0 to 90 degree in alpha that is the zeta rotation. But, we will show why it is not up to 0 to 90 degree, because of the defocusing status, where the incident beam becomes almost parallel to the sample surface; we will talk about that later. So, this is what about the different kinds of components that are present in the x-ray texture goniometer.

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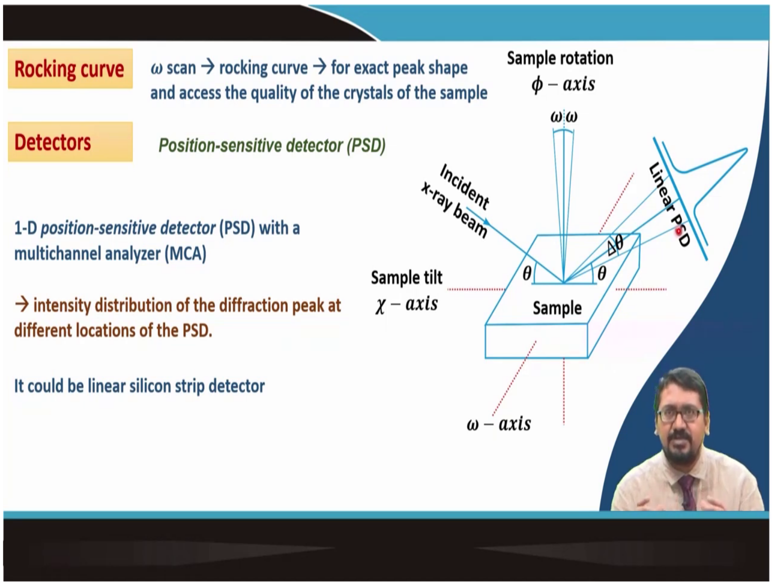

Now, that in order to obtain the peak as I said the omega is basically rotated. So, that it goes to that theta or 2 theta for that particular HKL plane to get that Bragg angles reflection right. that, usually what happens? When we do a different kind of samples? And, we have samples which have may have residual stresses or which have some stresses. So, that its d is somewhere elongated or it is compacted.

So, in order to obtain, the to see the quality of the crystal means where actually the theta lies for that particular HKL plane a rocking curve is usually done. The rocking curve is basically nothing, but there along R D. The omega is given a slight rotation along R D is given to see the exact peak position of the sample. And, that if this is given then the theta basically changes a little by delta theta. And, that is how we get the exact peak position of the sample for the any particular HKL plane, which may be required. Now, this figure basically shows this rocking curve. At the same time we are showing this along with a linear position sensitive detector .

A linear position sensitive detector can detect has a larger it has a length, it is one dimensional it has a length. So, it can take intensities from more than 1 HKL planes, which are diffracting at different thetas right. And, this diffraction may occur and this may not follow the Bragg Brentano focusing condition, but still it will diffract we will come to that.

But, that here the incident beam, remember that this incident beam is shown to be a single line, but it is coming like a point parallel, but it is not like that it is a divergent incident beam. And, then it is it seems like it is divergent diffracted beam, but it is not like that it is a convergent diffracted beam. But, why the different angle has been shown, this different angle has been shown to show, that if the diffraction can occur in 3 different planes, three different HKL planes. Which are closer and maybe in a range of 5 to 10 degrees from each other? And, then the linear position sensitive detector may detect all these three intensities and may be able to separate out these three intensities.

So, at a single go one can measure three pole figures at the same time, but there could be inherent difficulties that we will talk about in the later slides. Now, the 1-D position sensitive detector has comes with a multi channel analyzer. And, the intensity distribution of the diffraction peaks in different locations of this position sensitive detector is basically noted down by this multi channel analyzer. At the same time in few cases linear silicon strip detectors are also used.

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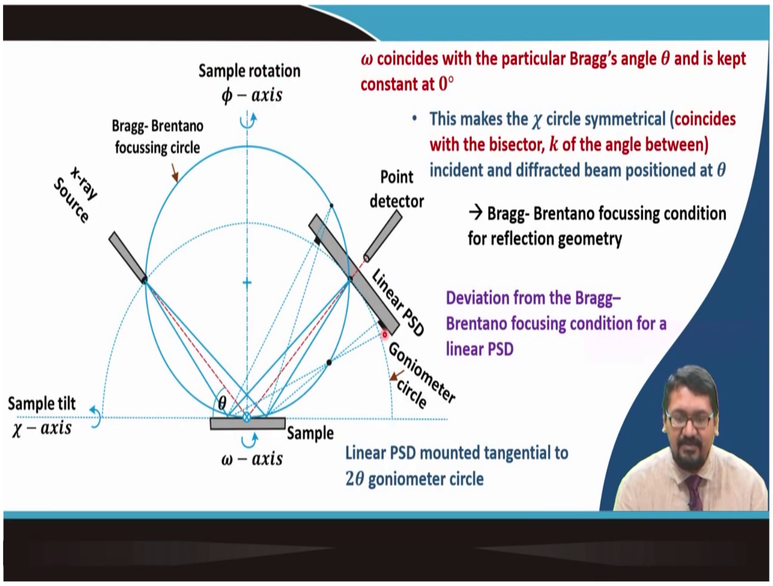

Now, here is a a figure which we have shown, though we have discussed this in quite a detail in the last lecture, but still I think that it is needed to have a little bit of more understanding, in terms of presence of a linear position sensitive detector. So, if that if there is a x-ray source and it gives a divergent beam like this. And, then there is a divergent beam which is sorry convergent beam, diffracted beam, which goes to the detector and if it is a point detector it goes exactly to this point. So, that it is detecting this.

Now, that the incident beam and the diffracted beam has a bisector and this bisector is exactly like this. And, this bisector is at the initial position when alpha is 0 and beta is 0; that means, zeta is 0 and phi is 0. Then at this position k that bisector is basically parallel to the end right. And, when under such condition the this we should always remember that when we measure a certain plane by rotating alpha beta, we always measure that plane which is perpendicular to that bisector k. And, therefore, we get the information for that HKL plane for every alpha and beta possible.

So, if this is a divergent beam and it is a convergent beam, then if this is the and if this is always kept constant. In such a way that we are always measuring that particular intensity, which is corresponding to this k bisector, then this position this point always comes in the Bragg-Brentano focusing circle right. So, the Bragg-Brentano focusing circle is such a way. So, that in a point detector the position where the the intensity of the X-ray beam basically get focused is the Bragg-Brentano focusing circle. Now, that if this is the goniometer circle, if this is the goniometer circle. This goniometer circle basically represents the circle where and if you say that, if this is R D. The along R D the omega axis omega is rotated. Now omega is rotated in such a way so, that we first fix it for that particular theta.

So, that now the Bragg-Brentano focusing circle is being fixed for that particular theta, for that particular HKL plane, which we will get focused here right. Now, the point is that when there is a presence of a linear position sensitive detector then, what will happen? That even if there are other HKL planes say H 1 K 1 L 1 plane; H 2 K 2 L 2 plane, then also these two planes may get intensity peaks here.

So, at a same time a linear position sensitive detector can be used to measure the positions of three simultaneous plane; the one which is having the Bragg Brentano focus and the two others, which does not have. So, that see the omega of this 4 axis goniometer is is rocked or rotated and so, that it gets to the particular Bragg angle theta and then it is made equal to 0.

And, this makes this this makes this k the bisector, which is basic, which lies in this zeta circle. And, this zeta circle becomes symmetric means becomes the bisector which consists the k of this incident and the diffracted beam right. So, this zeta circle this one, if you think that a circle is there right in this axis which is the k. So, if we use a linear position sensitive detector, then there is a deviation from the black Bragg-Brentano focusing condition for this case. Because, the linear position sensitive detector is actually kept tangential to the this goniometer circle and therefore, that this H 1 K 1 L 1 and this H 2 K 2 L 2 are not in the focusing condition.

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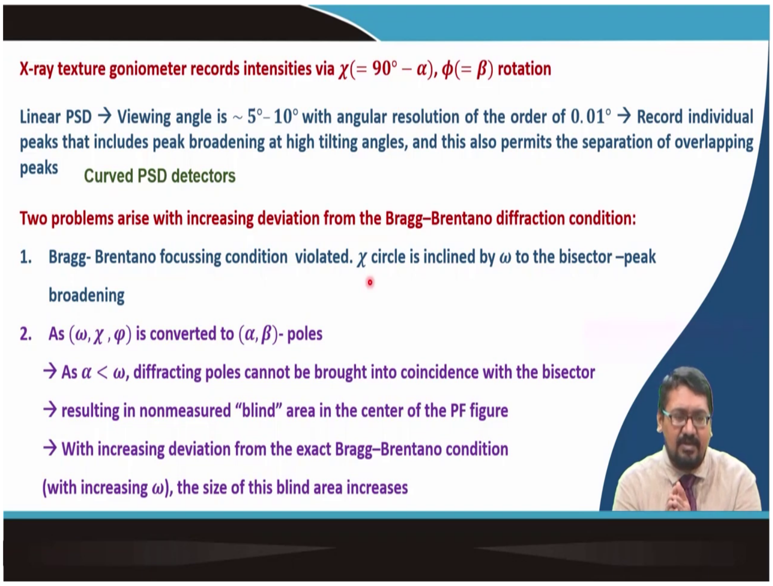

Now, one can say that one can use a curved linear not linear 1 D position sensitive detector, which should go through the Bragg-Brentano focusing circle. But, then also that even if it is instead of linear, even if it is what you called a curved position sensitive detector. The Bragg-Brentano it all the three focused diffracted intensity beams from the three different HKL planes of three different thetas, may lie at the Bragg-Brentano focusing circle and the detector also may lie at the Bragg-Brentano focusing circle.

But, then also the k between this three different beams may not remain same and the goniometer circle, may not follow the all the time the Bragg-Brentano focusing condition. So, as I have stated earlier that that the X-ray goniometer basically rotates zeta and phi that is alpha and beta.

So, basically a linear position sensitive detector has a viewing angle of 5 to 10 degrees and it must have a very good angular resolution of 0.1 degrees. And, so, that it can it can record individual H 1 K 1 L 1, H 2 K 2 L 2, H 3 K 3 L 3 for theta 1, theta 2, theta 3 all different Bragg's angles for these three different HKL planes. And, this must this must include the pre sorry this much include the this must include the peak broadening at high tilt angles. And, also should have it should provide the separation between the overlapping peaks. So, as I said the curve PSD detectors are also coming up in this case, but that as I said that two problems arise, when we basically increase the deviation from the Bragg-Brentano focusing condition.

The first condition is that the first problem is that the Bragg-Brentano focusing condition is violated. So, zeta circle is now inclined to the omega. And to the bisector of the bisector and then it will lead to peak broadening right, peak broadening. Secondly, as the omega zeta and phi is has to be converted into alpha and beta poles. And, now the alpha is always less than omega and the the diffracting poles cannot be bought in coincidence with the bisector right.

So, the diffracting poles and is not in coincidence with the bisector. So, it may it will give the correct diffraction and software's can be used to correct it, but it may result in non measured blind spot area, at the center of the pole figure. And, with the increasing this deviation of alpha and omega what will happen; that means, with the increase in the omega, the size of the blind spot in at the center of the pole figure will increase right.

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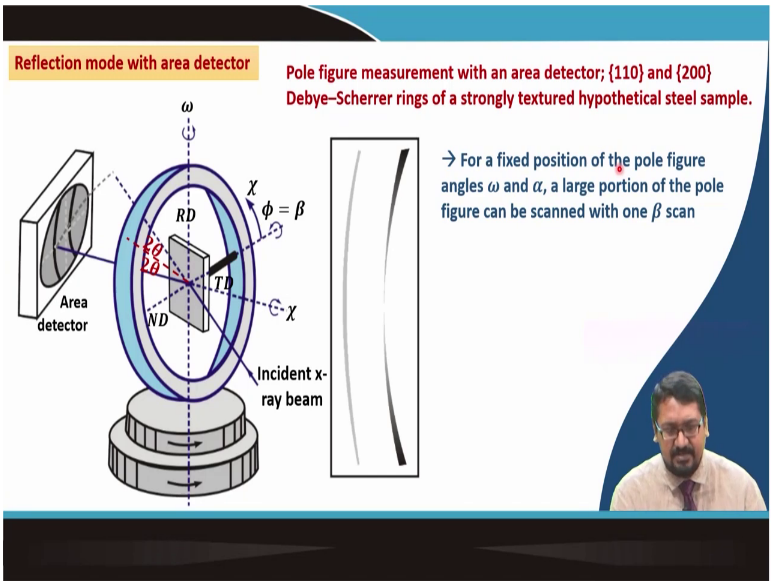

So, ok on the other hand in the reflection mode area detectors can means are coming up into with coming up to measure 2 or 3 different HKL planes simultaneously, with the more and more advent and the development in the science and technology. So, you can see that the pole figure measurement, this is an what you call hypothetical diagram, which shows a steel samples and a sample which has R D above N D here and T D this side.

In the reflection geometry same, but this time the incident beam is coming from this direction and here is the area detector present. And, in this case what has happened that one can measure a hypothetically, this is just not to measure by scale diagram, which shows intensity measured by 110 and 200 all together in a case in using an area detector.

So, for this intensity points may look something like this in the phosphor screen so, for a fixed position of the pole figure angles omega and alpha. A large portion of the pole figure can be scanned with 1 beta scan using such kind of an area detector right.

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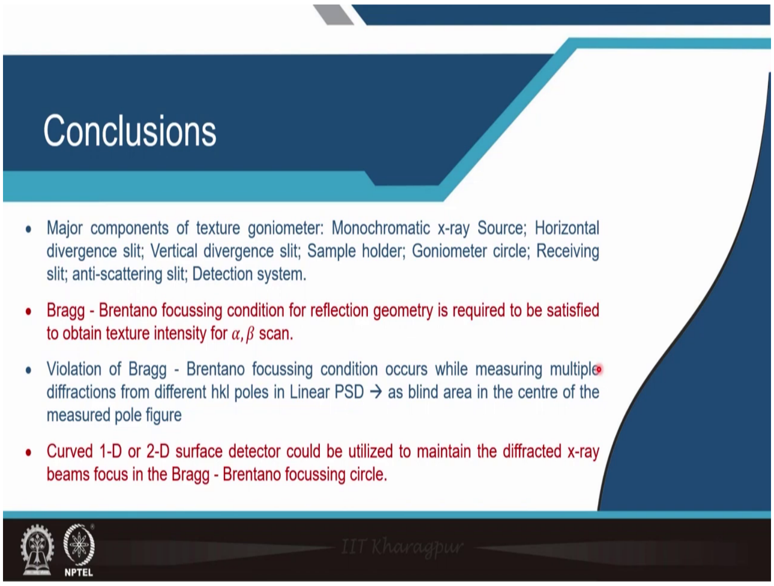

So, we can conclude here that the major components of texture goniometer, that we studied today is the monochromatic x-ray source; horizontal divergence slit; vertical divergence slit; sample holder a goniometer circle right, Eulerian carrier receiving slit; anti-scattering slit and the detection system.

The Bragg-Brentano focusing condition for reflection geometry is required to be satisfied to obtain texture intensity for the alpha beta scan right. Violation of Bragg-Brentano focusing condition occurs while measuring multiple diffraction from different HKL planes in Linear PSD as blind areas at the center of the pole figure, will start to evolve and it will increase as omega is increased compared to alpha.

So, the curved 1-D and 2-D detectors means the 1-D detector curved position sensitive detector and 2-D means the surface, but curved position sensitive detector. Could be used to means can be utilized to maintain the diffracted x-ray beam focused in the Bragg-Brentano focusing circle. But still there would be prominent problem to maintain it in the goniometer circle right.

So, thank you thank you very much for this class.