Neutron Scattering for Condensed Matter Studies
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Week 5 Lecture 12B

Keywords: Ewald construction, Bragg law, Debye camera, Debye-Scherer cone

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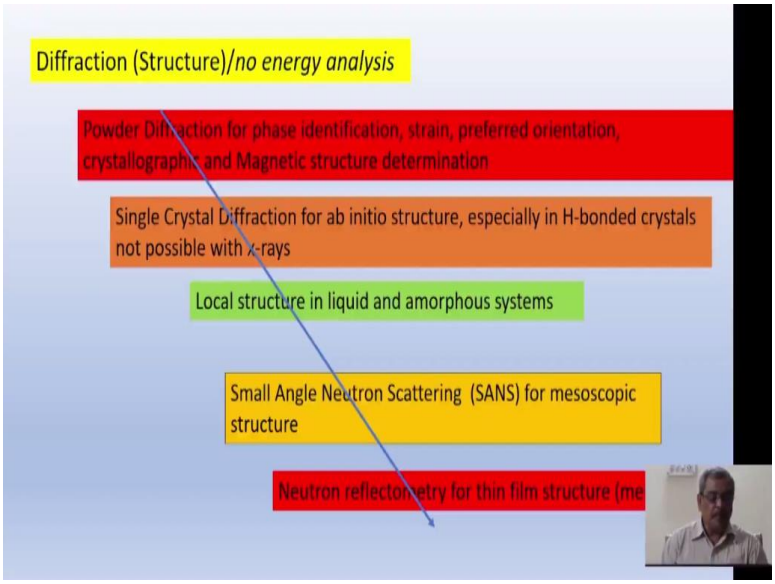

After discussing the difference in diffraction experiments between reactor and a pulse neutron source, we are back to the structure determination using neutrons. That means no energy analysis (of the scattered beam). As soon as we make a sample, most of us take it through x-ray powder diffraction and try to see whether we can identify the peaks that are listed in the international table for diffraction from powders. So, we can do powder diffraction for phase identification which is the simplest thing to do. Strain analysis, preferred orientation, crystallographic and magnetic structure, all these experiments can be clubbed under powder diffraction. It is the most commonly used technique with neutrons as well as with x-rays. I will discuss some of the techniques.

Most importantly I will introduce you to crystallography and magnetic structure determination for various samples. For study of a single crystal, a good single crystal may be required for ab-initio structure determination. In neutron diffraction not only, you need a single crystal if you want to do ab initio structure determination, but you also need to make a slightly larger single crystal because neutron intensity is poorer.

Since it can penetrate deep, we can take a reasonable dimension of a sample which will give information on the bulk liquid or a bulk amorphous system and we can find out the local order in this system. I will introduce you to this technique with large *Q* range experiments to find out local structure. Another very important technique which has become extremely popular not only with physics people, but also with chemist, metallurgists and many others, is small angle neutron scattering. Small angle can also be called as small *Q* and often we are talking about angle and *Q* that we use interchangeably. So, small angle neutron scattering is nothing, but a small *Q* neutron scattering.

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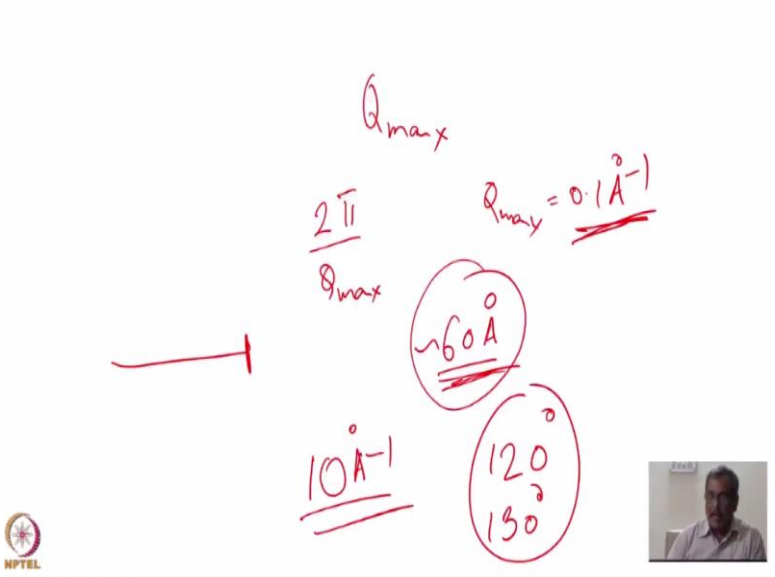

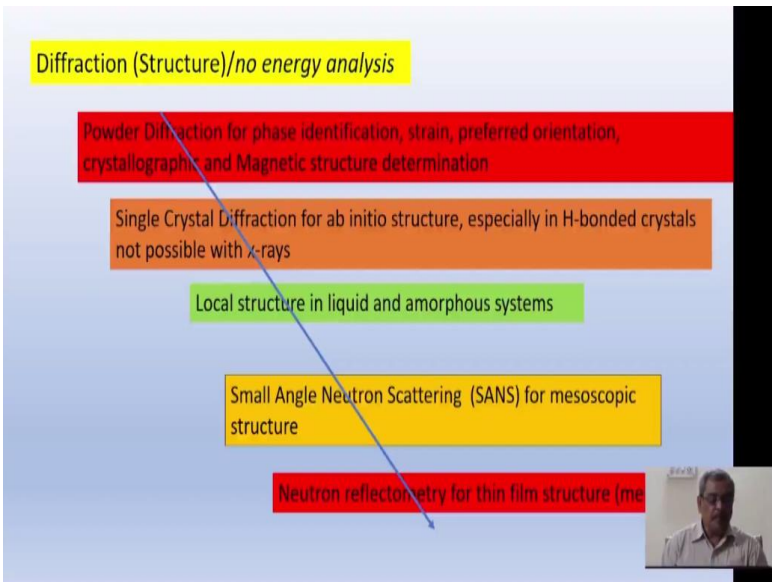

If in an experiment Q_{max} is largest value of momentum transfer then the length resolution is $\frac{2\pi}{Q_{max}}$ (by uncertainty principle). For example, if Q_{max} = 0.1 Å^{-1} then length resolution comes to around 60 Å. That means inherently if I do an experiment in the small Q range so up to 0.1 Å^{-1} then that experiment will tell me about structures with a resolution of 60 Å. With this 60 Å resolution I cannot see the crystallographic structure in a system. What I will see is an average over this length scale which is a mesoscopic length scale. To compare, a typical (powder) diffraction experiment (for crystal structure) will go up to 10 Å^{-1}. This might go to 130° in an experiment, whereas 0.1 Å^{-1} experiment is almost a near direct beam experiment.

I will discuss it in detail when I discuss the small angle instrument. Small angle (Neutron Scattering) or SANS is used heavily for determining structure at mesoscopic length scale and often many researchers are not keen to get information at crystallographic or near atomic level resolution, but at mesoscopic length resolution. For example, we may talk about micelles or, as I told earlier in my transparency, pores in solids or precipitates in metallurgical samples. They can be understood by using small angle neutron scattering.

Another technique which has come up recently is neutron reflectometry for thin film structure. Nowadays, especially for applications, thin films are very important.

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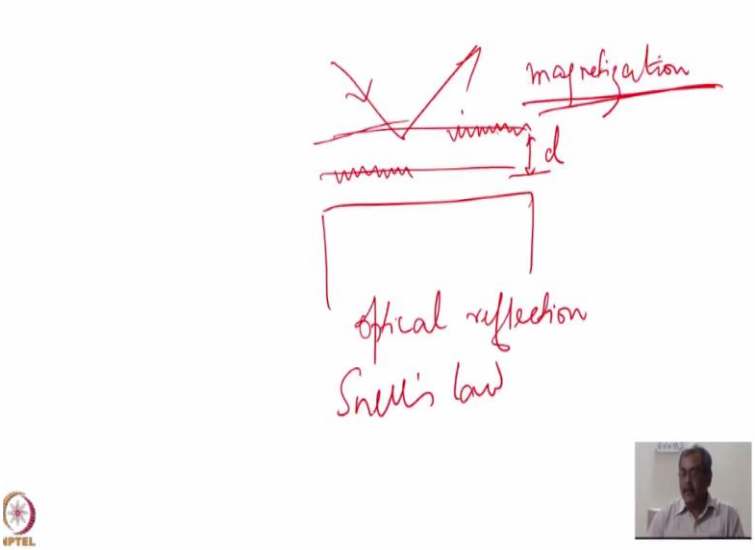

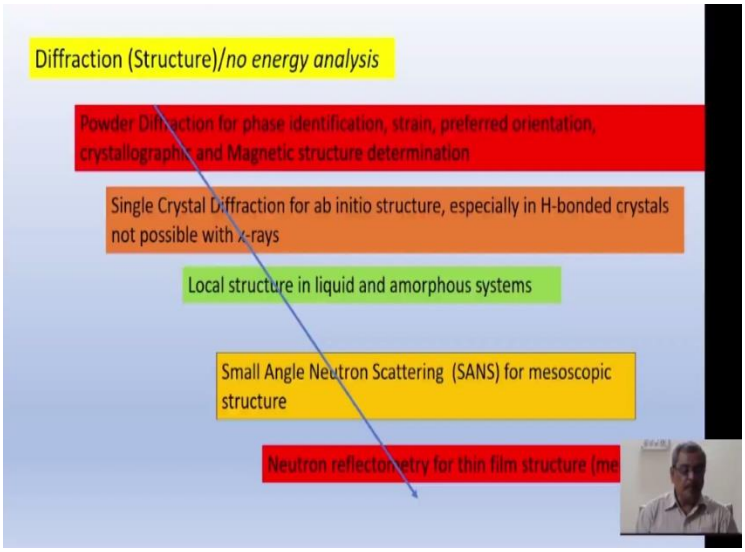

Neutron reflectometry deals with films deposited on various substrates and by reflection, I mean optical reflection following Snell's law. From reflected intensity we can understand the thickness of these layer in a heterostructure that are reflecting neutrons, their interface roughnesses and their composition. These can be determined using both neutron and x-ray (reflectometry).

Neutrons can also give you magnetization profile as a function of depth which is unique again for neutrons. All these we will be discussing while discussing neutron reflectometry which will be coming under the heading of mesoscopic length scales.

This will be my flow chart. I will start with powder diffraction techniques, briefly single crystal diffraction, powder diffraction, then liquid and amorphous systems for local structure, small angle neutron scattering (SANS) for mesoscopic structure and neutron reflectometry for thin film structure. All these are under the heading of diffraction and structure at various length scale.

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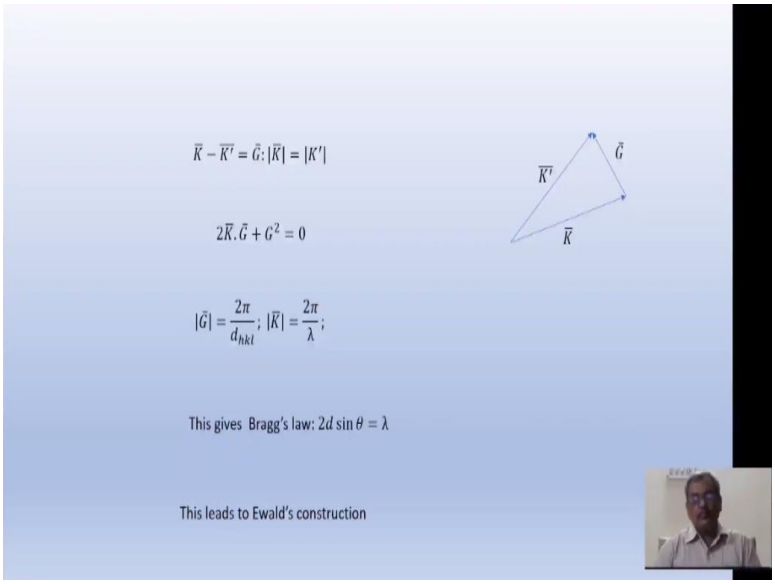

I told you earlier that for Bragg diffraction from a lattice $K - K'$ should be equal to G a reciprocal lattice vector and for an elastic experiment the magnitude of K and K' are same.

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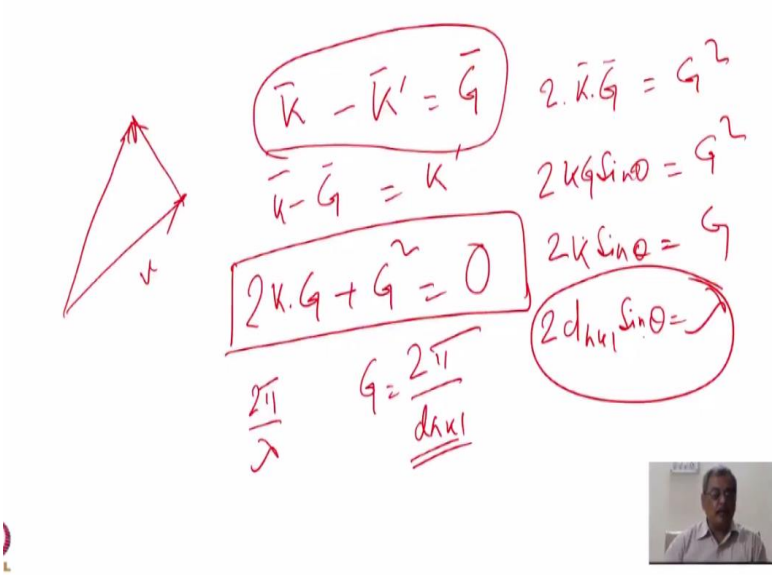

I can show you that this requirement of $K - K' = G$ translates to $2d \sin\theta = \lambda$. Squaring, this relation can be written as $2K.G + G^2 = 0$. It is equivalent to $2K.G = G^2$ or $2KG \sin\theta = G^2$. Putting $K = \frac{2\pi}{\lambda}$ and $G = \frac{2\pi}{d_{hkl}}$, it reduces to $2d_{hkl} \sin\theta = \lambda$, which is the Bragg's law.

Many of us have started with Bragg's law, but I started with $K - K' = G$ because now I want to get back to something called Ewald construction.

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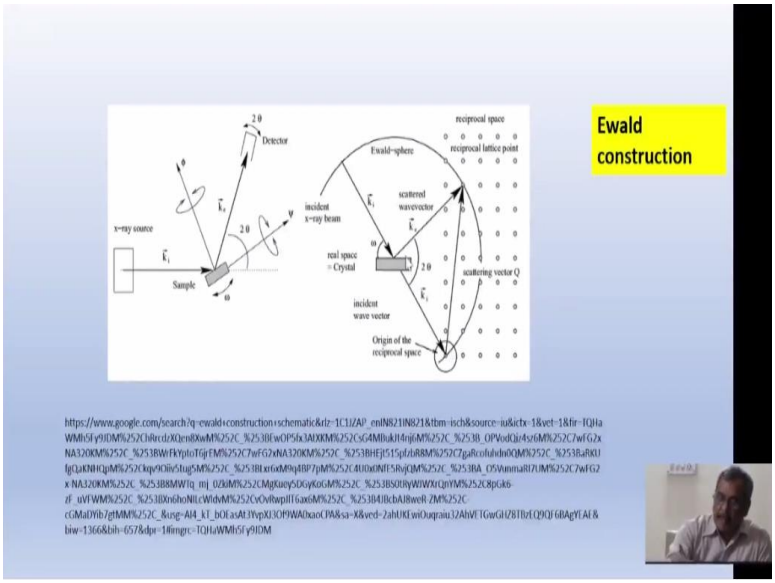

This construction was proposed by Ewald. In this construction if I consider an incident beam of length **K** then the reflected beam or the scattered beam will end on a reciprocal lattice when $K - K' = G$. And with this construction I can say that whenever the scattered beam hits a reciprocal vector then I have got a diffracted beam in this direction. This is known as Ewald construction. This is most fundamental construction in case of atomic diffraction. I have superimposed the incident beam (ending on a reciprocal lattice point) on the reciprocal space and with that I just show that $K - K' = G$.

All the points which are falling on the Ewald sphere in the reciprocal lattice signify that there is a diffracted beam in this direction. That is what a Laue pattern is, that we see from single crystals. So, Laue pattern comes out straight from the construction.

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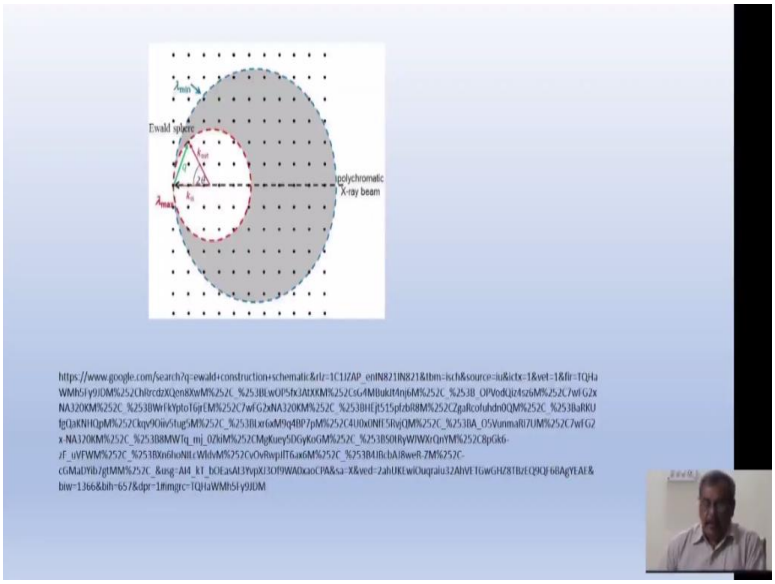

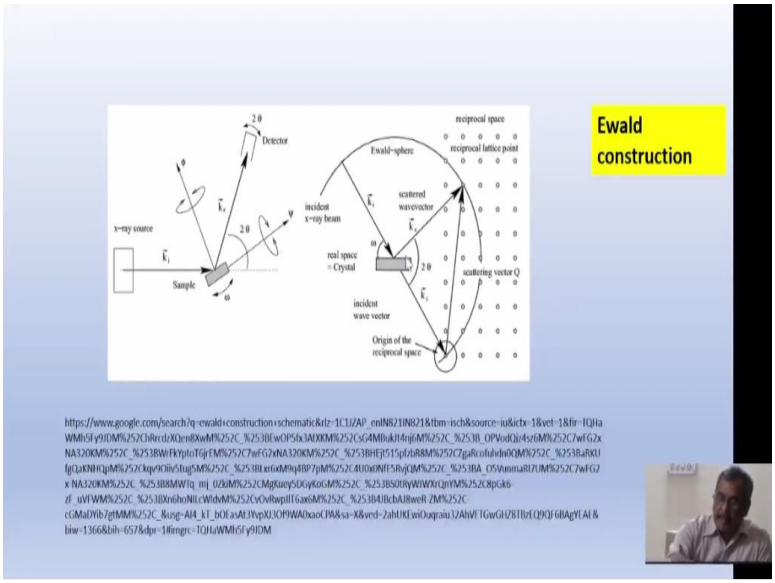

I must add here that in case of pulse neutron sources since you have a polychromatic beam with λ_{min} and λ_{max}. λ_{max} means K_{min} and vice versa. So, I have a range of wavelengths and you can see I have superimposed these two spheres with radii K_{min} K_{max} with reciprocal lattice. This is the origin of the circle and they are superimposed on the reciprocal lattice. All these reciprocal lattice vectors intercepting will satisfy $2d \sin\theta = \lambda$ for one of the wavelengths in this beam and will have a Bragg reflection in that direction.

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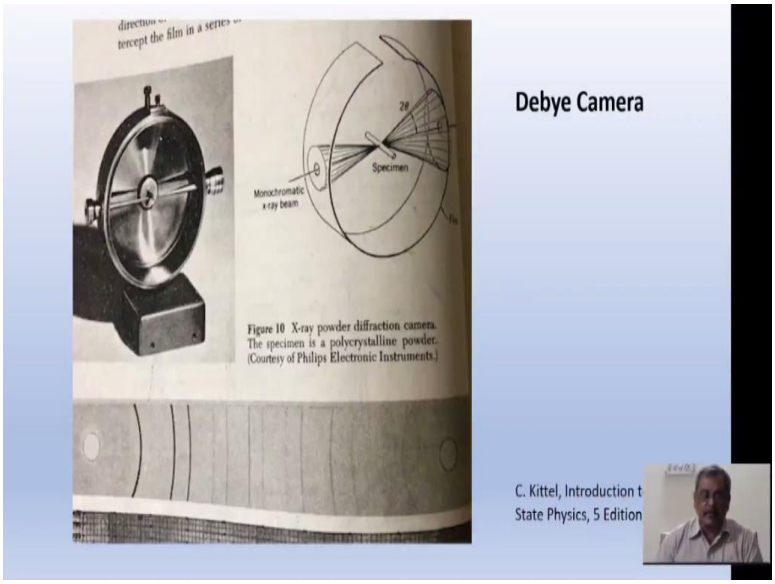

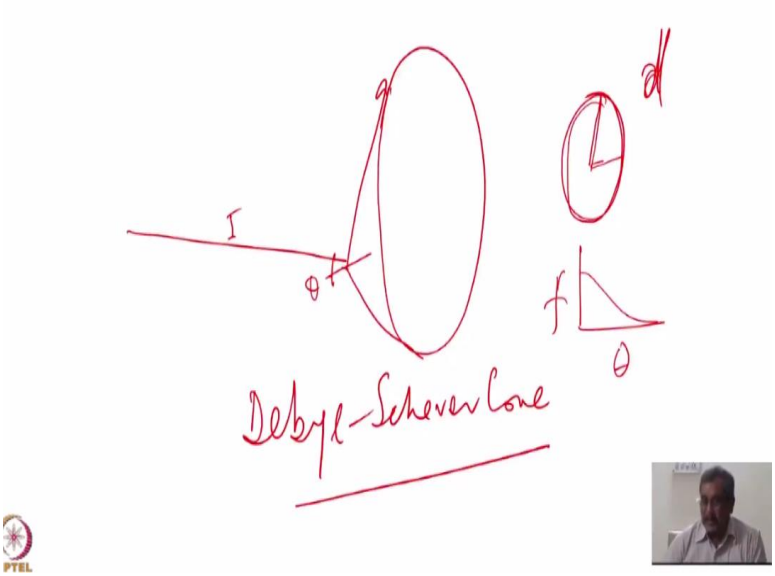

This slide (Time: 13:26) is taken from the Introduction to Solid State Physics by Kittel. Actually, here I hope that some of you have done this experiment. This shows how (x-ray) diffraction is done for a powder crystal. This is called Debye camera where a photographic plate was wrapped inside this circle. In the figure, this is the incoming beam; this is the outgoing beam and the powder crystal is kept at the center. I myself did such experiment on a copper crystal 40 years back. In the picture, instead of a single crystal I have got a powder crystal.

Now imagine I have got a powder crystal. I have got a powder crystal and there is an incident beam.

For a powder crystal there is this incident beam. In the powder crystal, if one crystallite satisfies Bragg relation in this direction there will be other crystallites equally oriented with the same

theta, but at a different orientation (with respect to the incident direction). That means I will be rotating the crystal orientation around the incident beam and the reflected beam will describe a cone in the reciprocal space.

For a powder crystal there will be a Debye-Scherrer cone. What I showed you in this photograph is basically a photographic plate that is intersecting this Debye-Scherrer cone. Now this is the incident beam hole, there is a hole in the strip, and there is the outgoing beam hole and you can see that this is the smaller *d*-spacing and I can calculate the *d*-spacing from the radius of this circle, because this gives me the angle from the radius of the Debye camera and from λ of the x-ray. I can find out from this radius of this cone the d-spacing of the plane. This experiment is done to find out various *d*-spacings. You can see that various *d*-spacings will satisfy different angles and in this case, it was a monochromatic beam of copper K_α of 1.54 Å

Usually, laboratory sources use 1.54 Å Cu k_α radiation so that λ is known, knowing the radius of this beam around the incident direction, using Bragg relation we can find out d-spacing.

If I reduce this to a strip detector or a one-dimensional position sensitive detector, this gives one particular *Q* or theta and what I showed you earlier, this is x-ray Debye camera, but what you see here is one part of the Debye-Scherrer cone from a powder crystal. This is the very beginning of our knowledge about x-ray diffraction. Basically, this is a monochromatic x-ray beam as I showed you for this specimen. The back reflected beam has high angle (of scattering) and the forward reflected beam in the low angle. This is around this hole this particular radius this is the at the center of this circle and this is near the backscattered beam and you can see the backscattered x-rays because of the atomic form factor are of much lesser intensity then the forward scattered beam because the form factor almost all atoms in case of x-ray falls.

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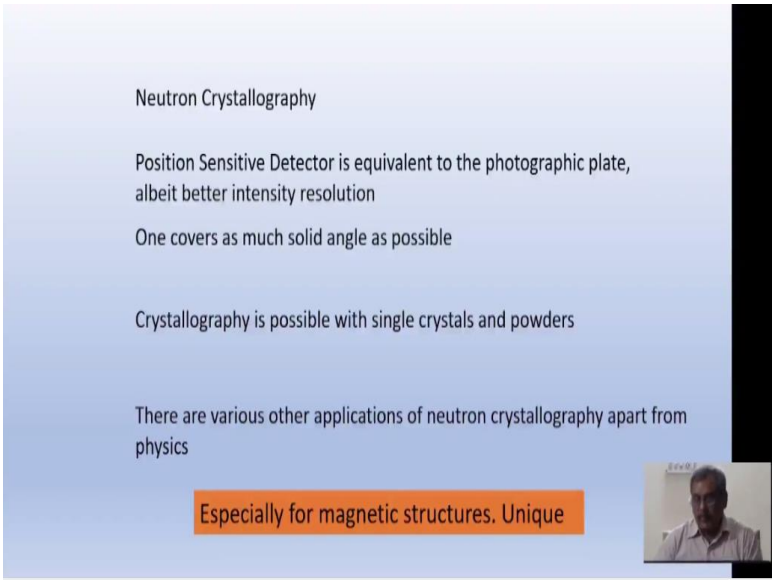

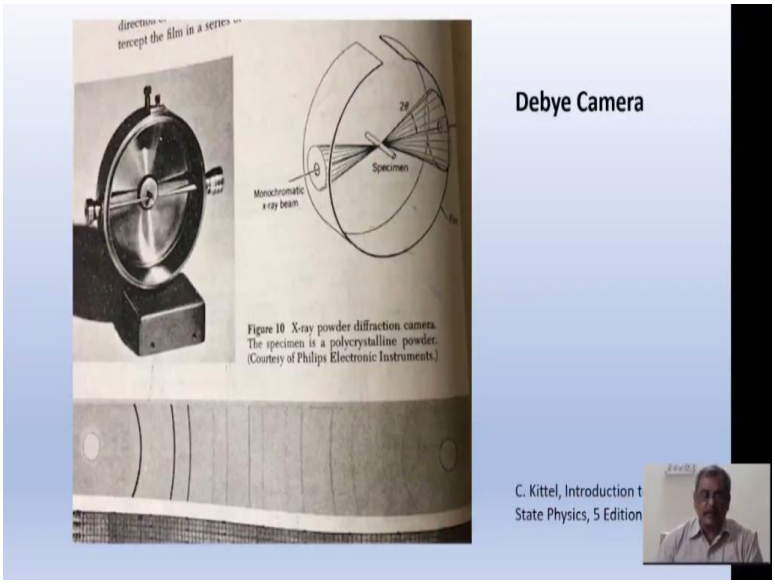

We do exactly the same thing with neutron. In neutron crystallography a position sensitive detector is equivalent to the photographic plate, only the size dictates how much of the cone that you are intercepting, but they have better intensity resolution. The instrument that I showed you can identify the *d*-spacing from the radius of the cone around this incoming and outgoing paths.

But in case of neutron there is better intensity resolution and we can carry out phase determination through Rietveld fitting. Ab initio crystallography is possible with single crystals but not with powders. There are various applications of neutron crystallography apart from phase identification. I will also tell you how strains can be determined using diffraction,

especially, neutron diffraction because neutrons can penetrate deep inside the material which is not possible by using any other radiation. Neutrons are also unique for magnetic structures. We will discuss all those in the next lecture.