Neutron Scattering for Condensed Matter Studies
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Week 12: Lecture 30B

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Let me quickly summarize what we targeted and did in this entire course, for your help in understanding various aspects of Neutron Scattering experiments. This is a brief course summary. I cannot discuss everything lecture by lecture but can discuss, only very briefly.

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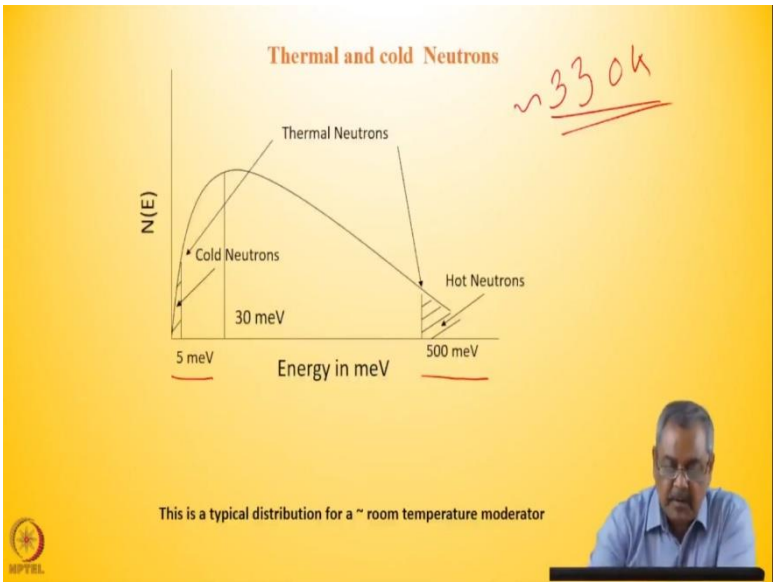

I started with the definition of thermal neutron because when I discuss neutron scattering for condensed matter, it is a thermal neutron which is useful. And I discussed with you that in the reactor after moderation, we have a spectrum which is typically at the moderator temperature of around, for Duruva let us say, is around 330 K~ 50 °C temperature of the moderator. Then you have a Maxwellian distribution of energy of neutrons and typically this has a peak around 30 meV. And lower energy neutrons are known as cold neutrons typically below 5 meV and high-energy neutron are called hot neutrons. Mostly we use for thermal neutron scattering, neutrons from this range as well as from cold and hot regions but mostly in the thermal region. These are typical distribution that we discussed.

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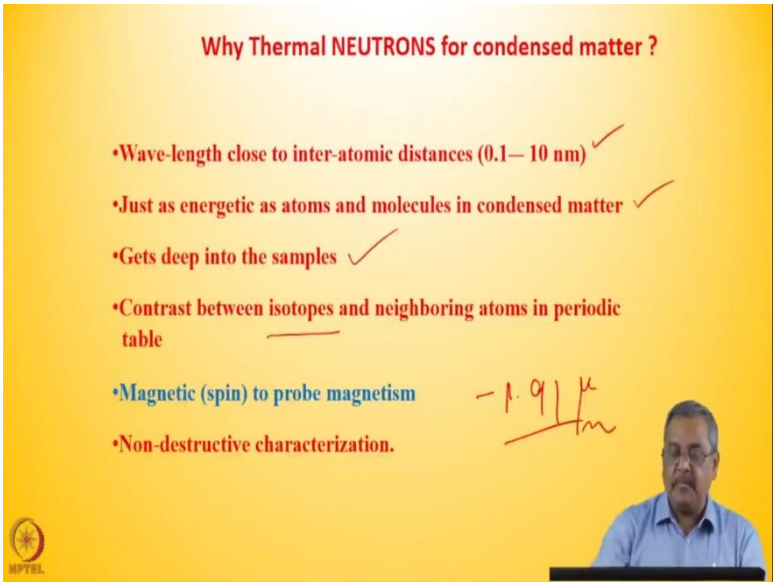

And I Justified use of thermal neutrons because they have wavelength close to inter-atomic distance. They have energies typically matching with as we saw throughout our discussion in inelastic dynamics, vibration of atoms and molecules, phonons, diffusion and also vibrational spectrum. Neutrons are neutral, so, they can get deep in the sample. They have got very good contrast between isotopes of elements and neighboring atoms unlike x-rays, as for x-rays interaction depend on the electron charge cloud and for neighboring atoms their contrast is poor. But here, not only neighboring atoms but even between isotopes the contrast is good because the neutron-nucleus interaction which is the strong interaction by nature have very good contrast between isotopes and notably between $^{1}H_{1}$ and $^{1}H_{2}$ (hydrogen and deuterium).

So, we can replace hydrogen with deuterium and vice versa depending on the requirement and we can generate very good contrast which is very important because when you replace hydrogen with deuterium, the chemistry remains same more or less, dynamics becomes marginally slower but otherwise they are same. And we can study selective parts of an object using neutron scattering. They have a magnetic moment of -1.91 nuclear magneton. And possibly they are the unique tool so far as microscopic magnetic structures are concerned. And it is a non-destructive characterization tool so it is always welcome for any samples.

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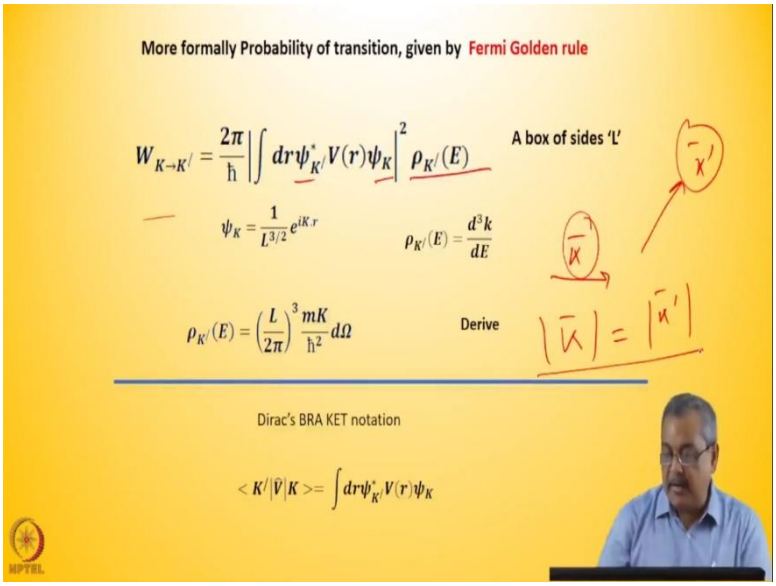

I started with the Fermi Golden Rule and described how I can work out the cross sections using this expression here $\bar{k}$ and $\bar{k}'$' are the incoming and the outgoing wave vectors. I discussed in this course density of states ρ_k and here I could use the density of states multiplied by the transition probability from $\bar{k}$ to k'' and I mentioned to you that for diffraction experiments the magnitude of k and $\bar{k}'$ remain same. We talk about structure in terms of elastic scattering or diffraction.

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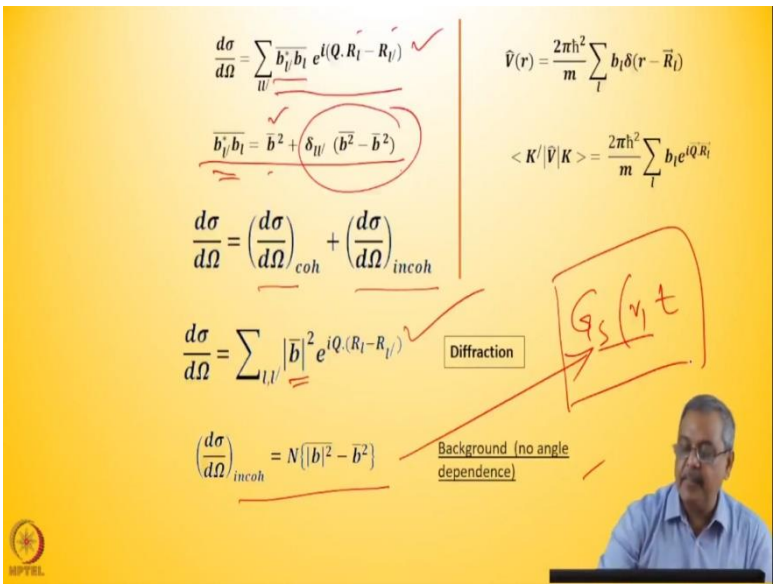

I derived the angle dependent diffraction law that is $\frac{d\sigma}{d\Omega}$, number of neutrons per unit solid angle and I showed you that there are two parts. One which takes care of the interference from atoms

at side R_l and $R_{l'}$ giving us the structure of the sample. That expression has a pre-factor which is coherent scattering length.

And also, we have got an incoherent scattering length which is $4\pi(\overline{b^2} - \bar{b}^2)$. The total scattering cross section is a sum of coherent term and the incoherent terms. Similarly, the total angle dependent scattering cross section also has got a coherent term and an incoherent term. But in case of diffraction, the incoherent part is not interesting. It is basically nuisance and it gives me the background in the diffraction data. Whereas the coherent part gives me the neutron diffraction pattern from which I try to solve for the structure.

I showed you later that when we came to self-correlation function in case of Quasi Elastic Neutron Scattering, incoherent scattering plays an important role. And we studied incoherent quasi-elastic neutron scattering, almost always using hydrogenous material that have a strong incherent scatterer viz. hydrogen.

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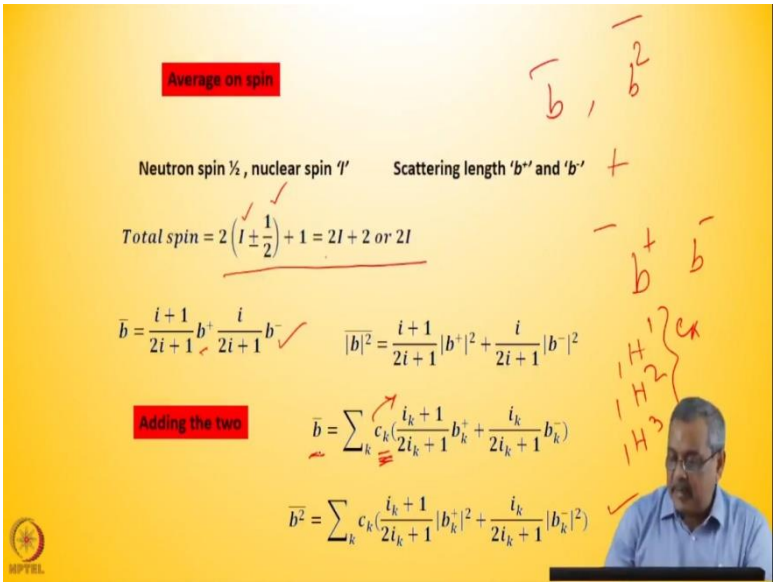

This was a task and I showed you how to calculate the coherent and incoherent scattering part of the scattering lengths and cross-sections. For that you need to find out $\bar{b}$ and $\bar{b}^2$. $\bar{b}$ comes from the fact that if I use up spin and downspin neutrons then I get two scattering lengths b^+ and b^-.

And the weighted average $of\ b^+$ and b^- is required because total spin of the neutron and the system is $2\left(I \pm \frac{1}{2}\right) + 1$ where $I \pm \frac{1}{2}$ is total spin of the system and the incident neutron and

this is either $2I$ or $2I + 2$, their sum is $2I + 2$. When it is spin down neutron, it is $2I$,the total number of possible states which the total spin has.

Then weighing according to the number of quantum projections $\bar{b}$ is nothing but $\frac{I+1}{2I+1}b^+ + \frac{I}{2I+1}b^-$. And, $\overline{b^2} = \frac{I+1}{2I+1}|b^+|^2 + \frac{I}{2I+1}|b^-|^2$ with the same weightage. And if there are several isotopes like if I talk about hydrogen, it has got a certain fraction of $^1H_1$ $^1H_2$, $^1H_3$. And if the relative concentrations are c_k then I can use the same formula with the concentrations as relative weight. I have to do it for each and every isotope and then take weighted sum over them. Here, c_k is nothing but the concentration of the isotope. For each isotope, I can calculate $\bar{b}$ and $\overline{b^2}.\overline{b^2} - \bar{b}^2$ will give me the incoherent part and $\bar{b}$ will give me the coherent scattering length for diffraction.

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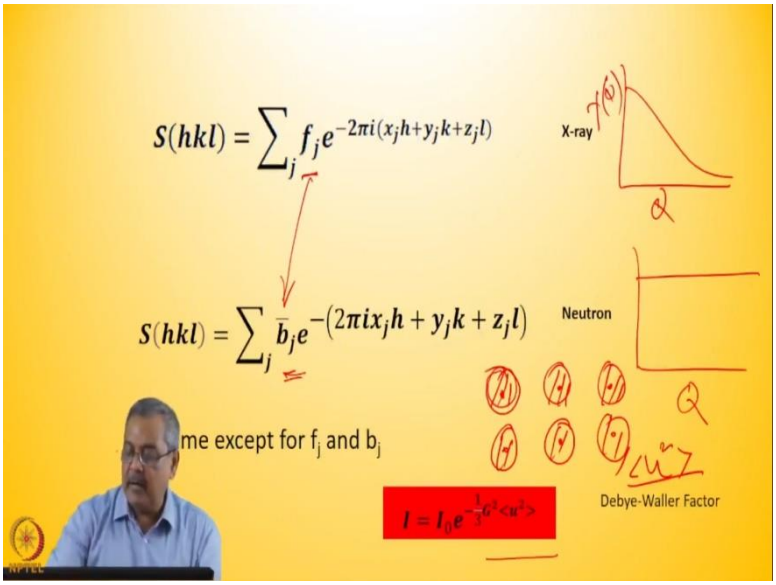

The structure factor, I mentioned for neutrons and x-rays. The only difference is here in the form factor. X-rays have form factors which fall with Q and so at high angle (or *'Q'*) we get low intensity for x-rays. In case of neutrons, instead of form factor I have got a scattering length b_j as a multiplying prefactor to structure factor and this has does not have any Q dependence.

So, you can say the form factor is constant with respect to Q for neutrons. Also, I told you that, we can estimate periodic structure at 0 K for a given crystallographic structure. When we come to a finite temperature, if these are the mean positions in a crystallographic lattice then there are vibrations around the mean positions as we go to higher and higher temperature, their amplitude increase. And that is taken care of by a factor known as Debye Waller Factor where

$\langle u^2 \rangle$ is the average deviation of the atom with respect to its mean position at a finite temperature. This is due to thermal fluctuation. So, this is due to dynamics and this is taken care of by a factor known as Debye-Waller in case of diffraction.

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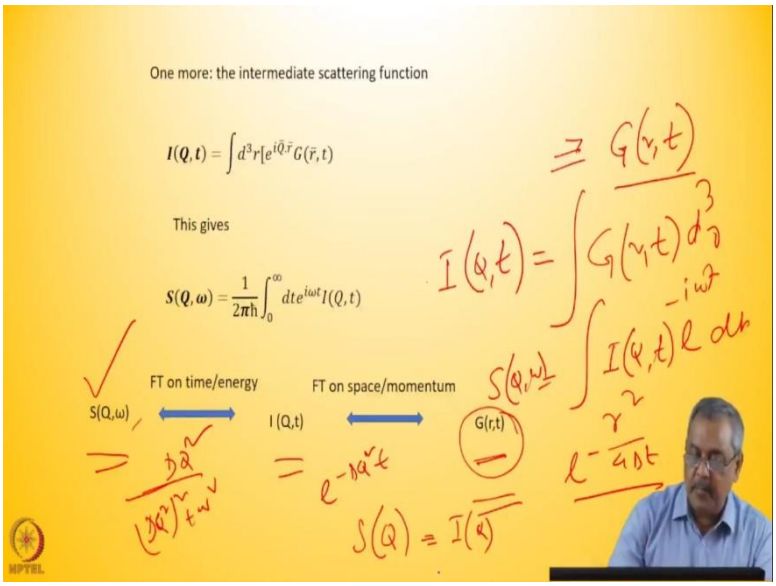

Then most importantly what I mentioned was that we are seeking information regarding spatial and temporal correlations in real space and time. $G(r,t)$ is the correlation function in real space and time, but its Fourier transform over space, $\int e^{iQ.r} G(r,t) d^3r$, takes me to $I(Q,t)$ which is an intermediate scattering law or function. And then one more integration, a Fourier transform over time takes me to $S(Q,\omega)$ which is the scattering law which we are measuring in a scattering experiment.

Now, it is desirable that I do a double Fourier transform and get all my information on $G(r,t)$ from my experiment but usually that does not happen because, the data is available over limited *'Q'* and *'ω'* values. So, I have to start with a model. For example, for quasi-elastic neutron scattering we started with a model which is $G(r,t) \sim e^{-\frac{r^2}{4Dt}}$ a Fickian diffusion model. And then $I(Q,t)$ became $e^{-DQ^2 t}$ and then $S(Q,\omega)$ became a Lorentzian in energy transfer $\sim \frac{\omega^2}{(DQ^2)^2+\omega^2}$. So, usually we have to figure out a model before we try to figure out the physical parameters. When we do only structure work then I can forget about ω. I am talking about only $S(Q)$ and this comes from Fourier transform of $I(Q)$.

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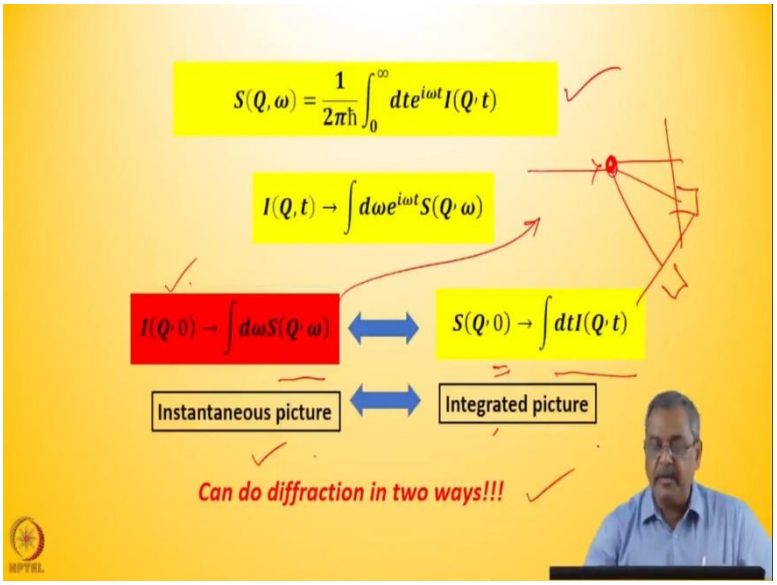

So, as I said say just now, that $S(Q,\omega)$ comes as a Fourier transform of $I(Q,t)$ over time *'t'* and $I(Q,t)$ also comes as a Fourier transform of $S(Q,\omega)$. And from these two expressions one can see $I(Q,0)$ is nothing but ~ $\int S(Q,\omega)d\omega$.This is what we measure usually. When we do our experiments; we have the incoming beam, say in a reactor a monochromatic beam and we have either a position sensitive detectors or end-on detectors looking at the sample. So, we do not do any energy analysis, which means we do an integration over energy *'ω'* and what we get is $I(Q,0)$. This gives an instantaneous picture. On the other hand, if we do an experiment where we ensure that the energy transfer is zero then what I get is a time integration of the structure, because $\int I(Q,t)dt \sim S(Q,\omega = 0)$.

But both of them are identical because if I consider an experiment in which I find $I(Q,0)$, then I collect the data over a finite time which is much larger than the time scales in the system and ultimately, I get an average over time through frame-by-frame addition of data and this is what we also get inherently for an experiment at a zero energy transfer. So, these are the two ways we can do diffraction.

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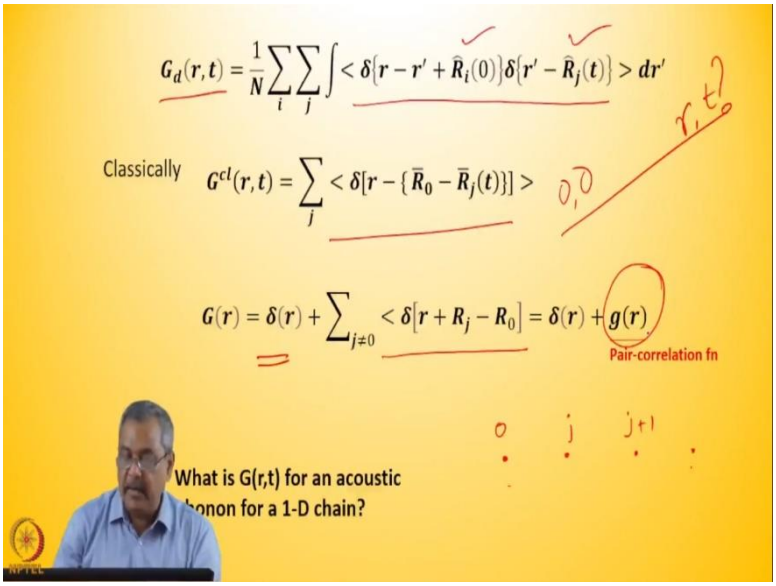

Most importantly, we try to find out pair correlation functions through various experiments. This is the proper way of writing down the pair correlation function in space, because in quantum mechanics the position operators do not commute with each other. So, we have to keep them separately and in proper sequence. This is a definition and the protocol of writing down a pair correlation function. If $R_i \neq R_j$ then it is *G(r)* for distinct pairs, that means a particle at origin at time zero then what is the probability that another particle is at position *r* at time *t* which is, $G(r,t)$.

Classically, I can write the pair correlation as a single term correlation function. If $R_0 \neq R_j$ and if I sum over all the positions, one, a particle is certainly correlated with itself at zero time, so at zero time the particle is only correlated with itself which is a $\delta(r)$ and this is at time $t = 0$. This gives me, if I just take the example of a linear chain, so, this is the 0^{th} particle, it is connected with all the particles at 0 K through the crystallographic structure, I am saying. So, this gives you the pair correlation function. This is what we obtain in a diffraction experiment.

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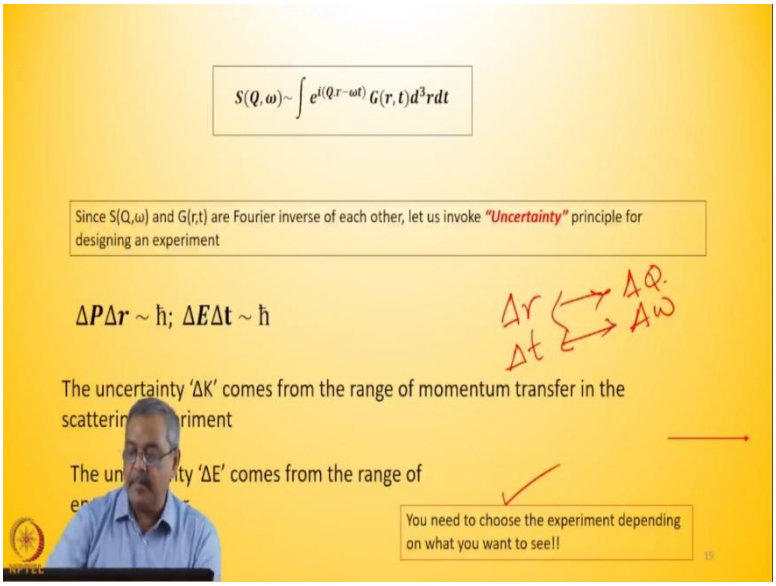

Since $S(Q, \omega)$ and $G(r, t)$ are Fourier inverse of each other so, we have to bring in uncertainty principle in our experiments. If I want certain Δr then that is related to the ΔQ in my experiment and if I am targeting certain time scale or Δt in my system that is related to the energy transferΔE in my experiment. So, we need to choose the experiment depending on what we want to see/measure. Basically, my choice of momentum transfer gives me the resolution in space, regarding structure. My resolution $\Delta\omega$ in energy transfer gives me the range of dynamics that I can see in various time scales $'\Delta t'$.

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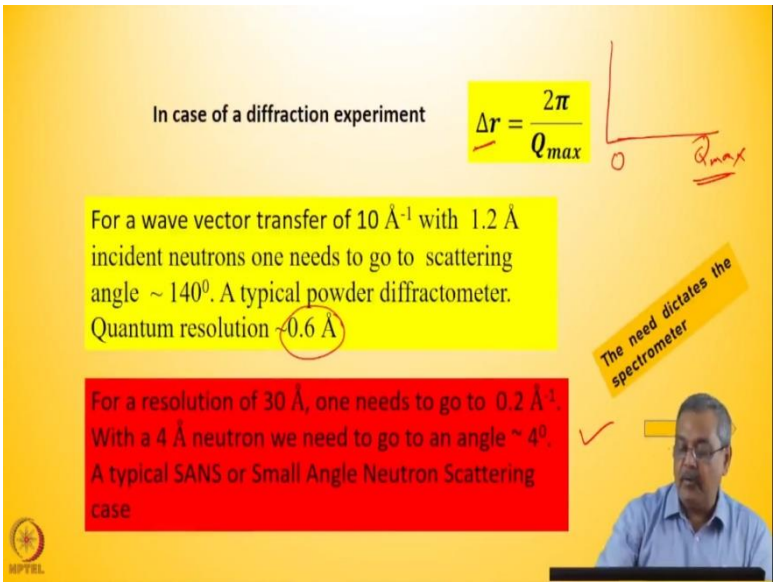

This is just an example which I used that in a diffraction experiment the overall the Δr of my experiment is given by $\frac{2\pi}{Q_{max}}$ because that is the range of momentum transfer that I am having in my experiment and this whole data is giving me my structure in the system. So, Δr is dependent on $\frac{2\pi}{Q_{max}}$.

I have given an example for a wave vector transfer of 10 $Å^{-1}$ with an incident neutron of 1.2 Å. We need to go to almost 140° for a typical powder diffractometer and then quantum resolution comes out to be around 0.6 $Å^{-1}$, typically, in the range of inter particle distances.

Suppose we want to make the resolution poorer in real space say 30 Å so that we can go to smaller Q transfer. As an example, if we go to 0.2 $Å^{-1}$ with a 4 Å neutron, we do the experiment at an angle of 4° only. This is a typical small angle neutron scattering angular range.

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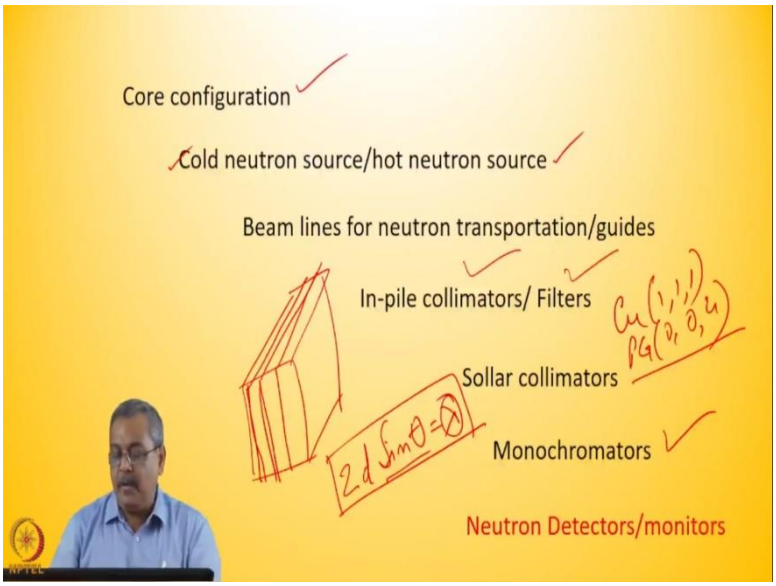

I discussed with you earlier core configuration of a reactor and how we have beam lines, various beam lines in various places reaching the core. Then various additions to the core, like cold neutron source and hot neutron source which will shift the neutron spectrum towards desirable energies. Experiments, where we want to use long wavelength or slower neutrons, I need a cold neutron source or experiments where large energy transfer or smaller wave vector transfer is required, we need hot neutron source.

I discussed the beam lines for neutron transportation as well as neutron guides that carry neutrons by the principle of total external reflection from guide mirrors. Before we impinge

the beam on a sample, we also tailor the geometry of the beam using the in-pile collimators. We remove unwanted neutron energies by using filters. To give one compromise between size of the beam and the angular resolution, we have what is known as solar collimator, where a large beam is split by use of neutron absorbing materials like cadmium as shown in the sketch.

In case of reactors, we use monochromatic neutron beams for the experiments and we need monochromators. Monochromators are usually single crystals with some mosaic spread. We have discussed, copper (111) reflection. I think we use also discussed paralytic graphite (004) reflections. So, these are the typical crystallographic set of planes which you use depending on what wavelength we need at what angle. And it works on the principle of Bragg's reflection. But you also have monochromators based on neutron supermirrors where you literally reflect a neutron beam from a mirror optically.

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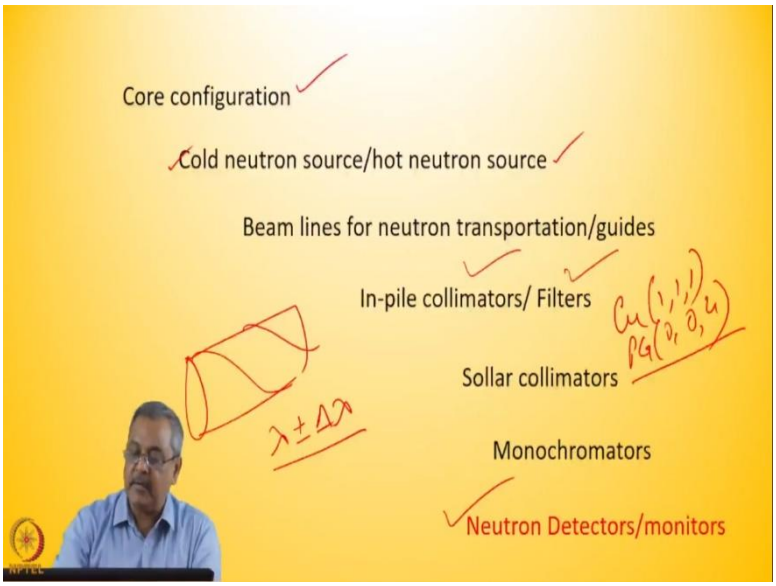

Or we also use mechanical assembles like velocity selectors where we have cylindrical bodies with helical slots on them. They allow a neutron beam of certain wavelength with a broad wavelength distribution to go through. And last but not the least and very important component, I discuss with you about neutron detectors. Neutron detectors are very important with respect to neutron intensity measurement in reactors and also in spallation neutron sources.

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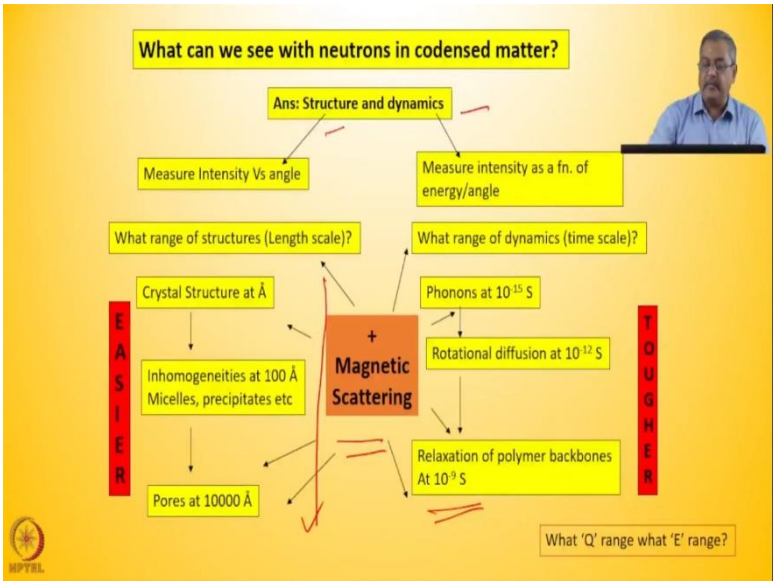

Next, I discussed with you, after these descriptions, what can we see with neutrons. There are two branches, as shown. One was the structure branch and the other one was dynamics part. And in the structure part we measure intensity versus angles. We do not do energy analysis. And on the dynamics part, we do energy as well as angle analysis of the scattered beam.

In the structure part, we went from crystal structure in Å length scale to all the way to small angle neutron scattering and reflectometry in our discussion for mesoscopic structure in tens to hundreds of Å length scale. When we discussed inelastic neutron scattering, we started with phonon dynamics. And ultimately, I discussed dynamics at nanosecond length scale using spin-echo.

And all these experiments are also having an additional advantage that neutrons have a magnetic moment of -1.91 μ_N or nuclear magneton. All of these experiments have an additional parameter which can be magnetic mesoscopic structure, magnetic dynamics or magnons or magnetic crystallographic structure.

Neutron is the most used tool for understanding magnetic structure and dynamics in condensed matter. Only which Q and which E range we need to use for a certain range of structural length scales or certain range of dynamical time scales is what our choice has to be.

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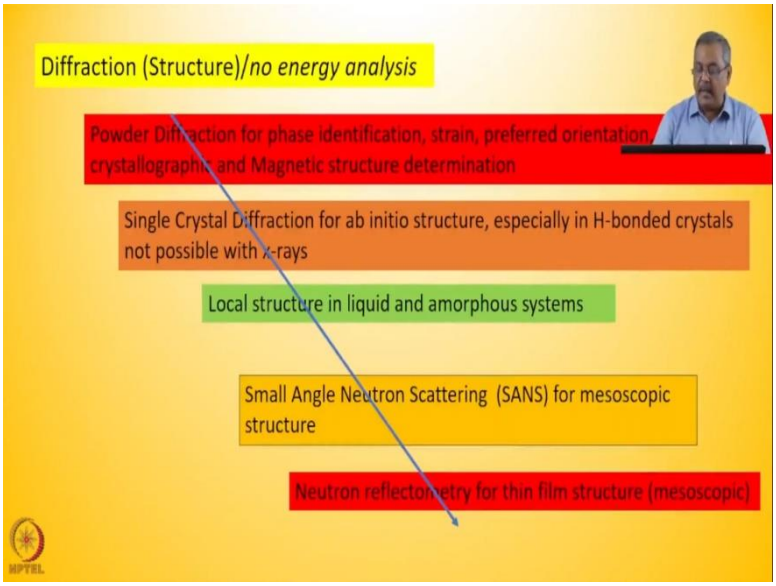

In diffraction, I went from powder diffraction to single crystal diffraction then to local structure in liquid and amorphous systems and finally small angle neutron scattering to neutron reflectrometry for mesoscopic structure. We also discussed neutron reflectometry. We discussed neutron detectors in one lecture where we discussed various types of neutron detector that one can use. Specifically, at the moment, position sensitive detector that we use in reactors are a great advantage in efficient data collection and I also mentioned scintillation detectors that can be used at spallation neutron sources.

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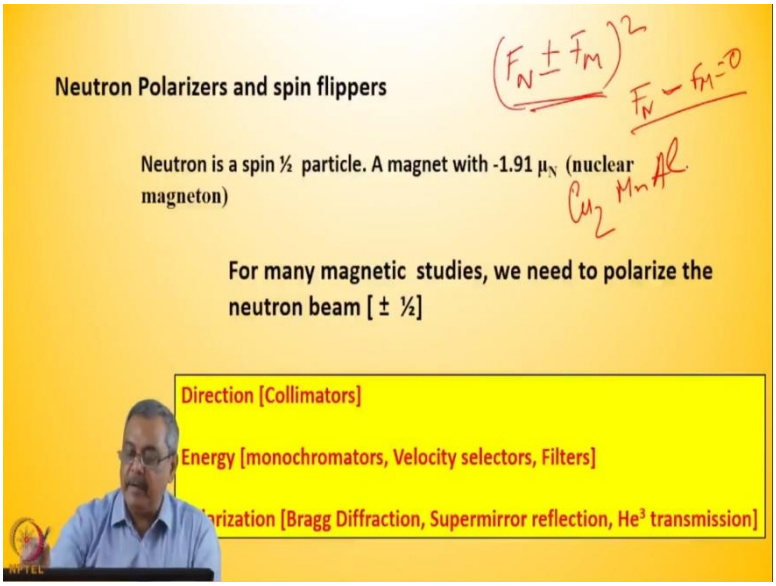

I discussed with you about neutron polarizers and spin flippers. I need to often polarize neutron beams and that can be done using Bragg scattering. Because there are some materials like Heuslar alloy where you can have the nuclear scattering length and the magnetic scattering length of almost similar amplitude so that $F_N + F_M$ for one polarization is much larger than $F_N - F_M$, $F_N - F_M$ is almost equal to 0. And after Bragg reflection from such an alloy like Cu_2MnAl, I get a polarized neutron beam with one spin polarization.

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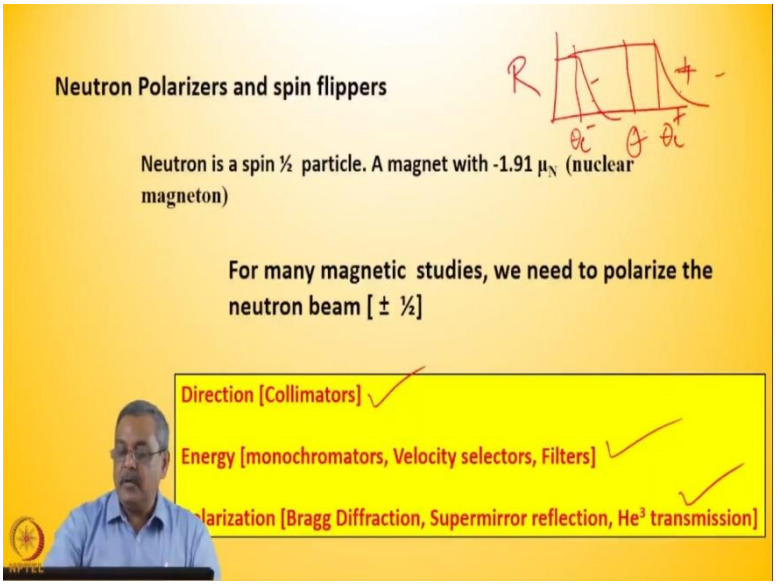

It is also possible to have polarized neutron beam by use of supermirrors where we have critical angles for reflection, much different for two spins. And I can have a polarized beam by

choosing an incident angle on the supermirror which is between these two values of critical angle. This is θ_c^- for say down spin and this is θ_c^+ for up neutrons, as indicated in the sketch. So, I can use an ordinary monochromator first to get a monochromatic neutron beam and then I can reflect it from a supermirror to get a monochromatic polarized neutron beam.

We define the direction by using collimators, we choose the energy by monochromators, or velocity selectors, filters etc. We do polarization using Bragg diffraction, supermirror reflection and of course, we can also do using helium-3 transmission, not available everywhere and only in some of the sources. By transmission through a polarized helium gas, we can get a polarized beam of neutrons in transmission mode.

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This is a typical powder diffractometer which is used for magnetic neutron diffraction at DHRUVA.

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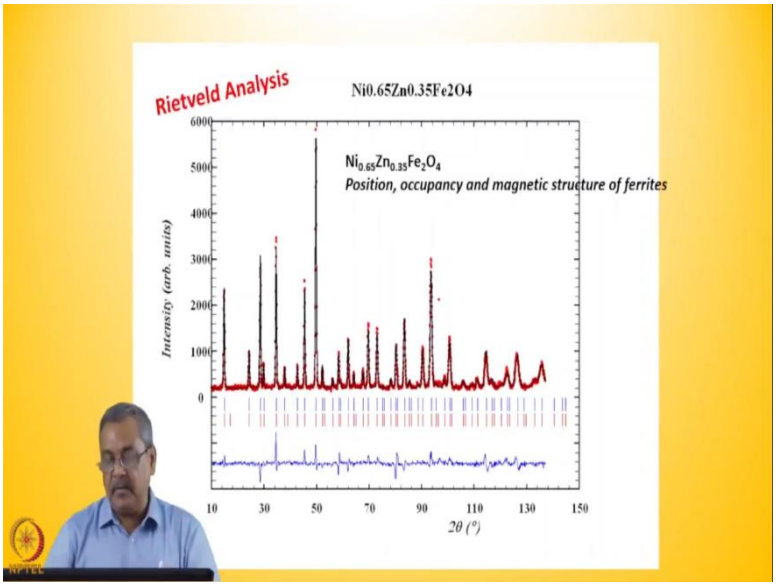

This is typical data which has been collected on this instrument and these fits are from Rietveld analysis of the data.

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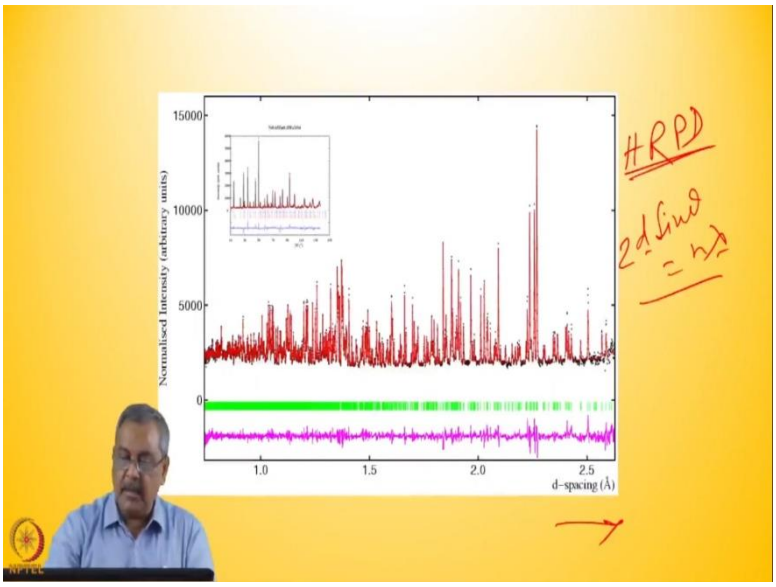

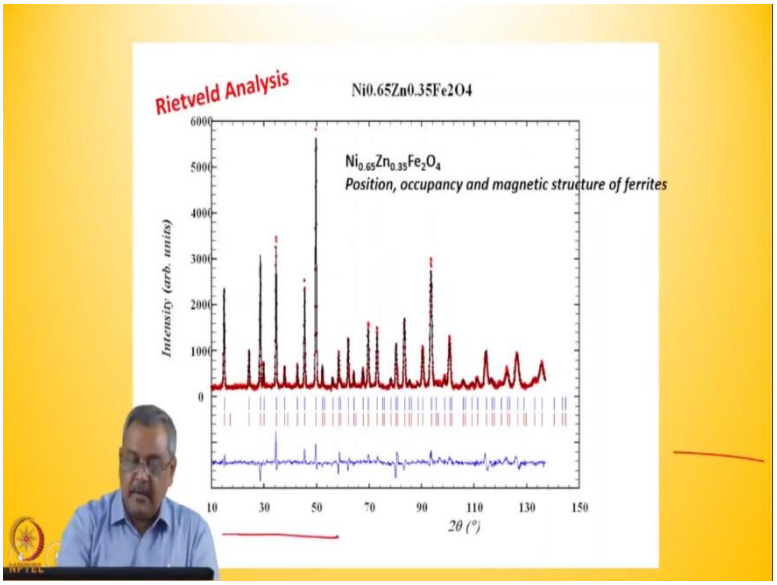

This is a data from the HRPD, High Resolution Powder Diffractometer, at ISIS, Rutherford Appleton Laboratory. Please note the way the peaks go as a function time-of-Flight. Here, because it is with respect to *d* spacing, so, large *'d'* spacing is the low Q part here in ToF. And this part, the shorter TOF part or the the high Q part in the low *d* spacing range because $2d \sin\theta = n\lambda$, You see, as the *d* spacing becomes longer or the time of flight becomes longer, we go into the lower range of θ in the other intensity vs *'θ'* or 'Q' plot in a monochromatic reactor source. The diffraction data in a reactor and in a spallation neutron source ToF plot are mirror images of each other.

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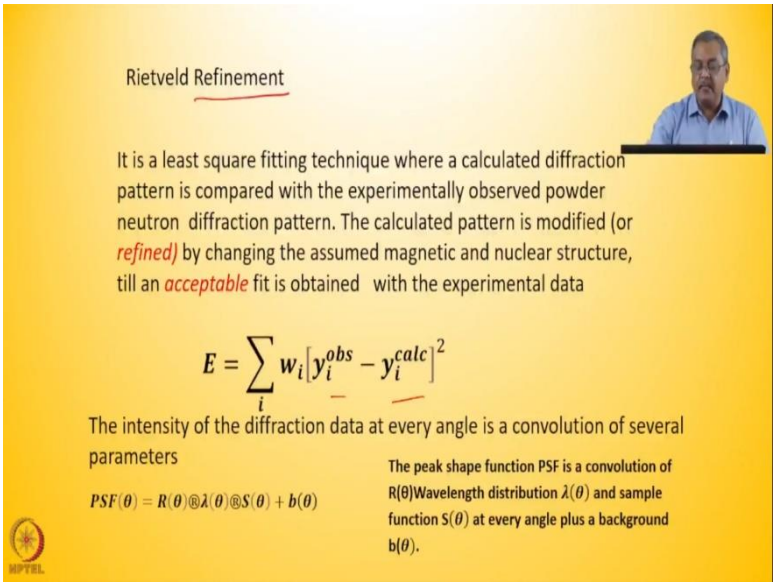

Rietveld refinement is a very commonly used tool for fitting diffraction data and actually most popular tool possibly with x-rays and neutrons. It is basically a χ^2 minimization technique.

Like all other techniques, we try to reduce the difference between the y-observed (data) and y-calculated (fit). But it is a refinement of a given structure, as I told you earlier. Let me just remind you that you start with a known structure and refine it to get a good fit to the data and that way we can get microscopic structure, magnetic as well as physical structure of materials.

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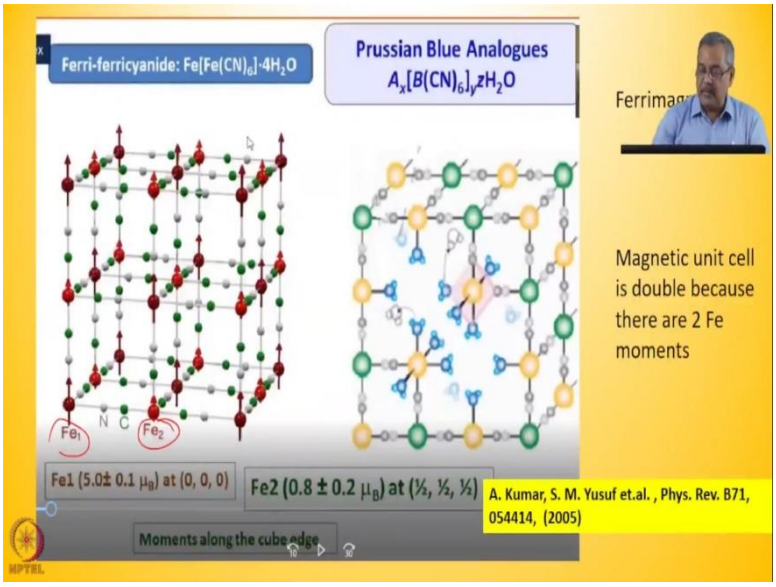

I just showed you one magnetic structure where you can see we can not only obtain position of the atoms by Rietveld fitting but I can also get magnetic moment values at various sites. In this example, for iron there are two iron siteshaving two different magnetic moments in this Ferri-ferricyanide sample taken from this reference.

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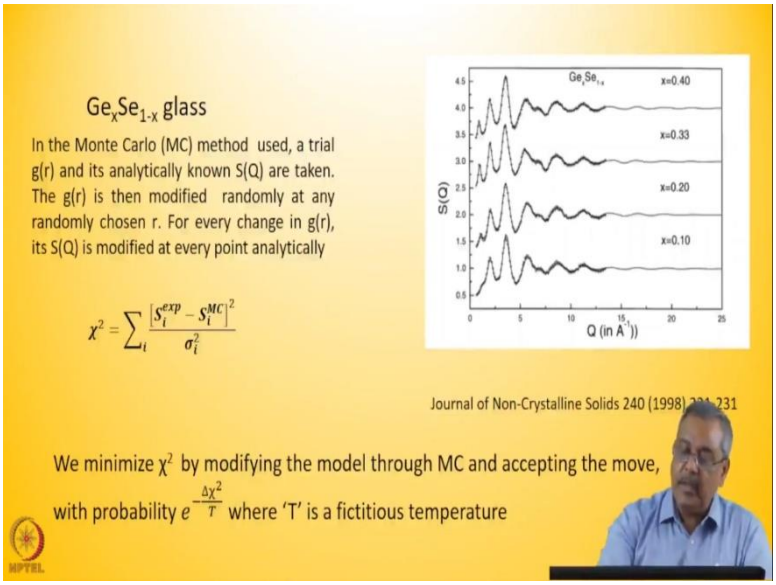

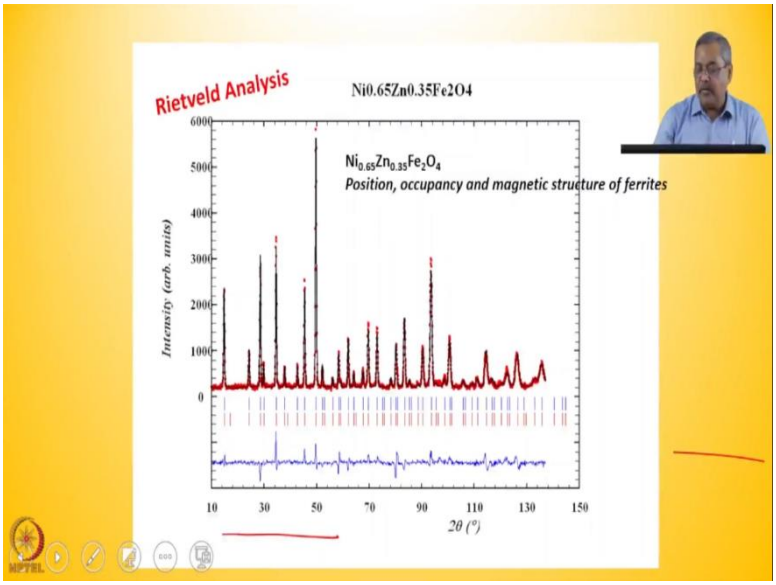

Next, I discussed with you the local structures in liquid and amorphous systems. Just as an example, please, see the difference here between crystalline diffraction and short-range order in liquid and amorphous structures. This is what a long-range order crystallographic material, this is diffraction from a powder crystal and how it will look like in a diffraction experiment. You have sharp peaks all over Q.

But when we talk about a liquid and amorphous system, you can see that $S(Q)$, it gives first sharp diffraction peaks or second sharp diffraction peaks. But it goes to a flat line because in case of liquid and amorphous material, you have only local structure and we do not have a long-range order.

And the consequence of that is, in $S(Q)$ you get this kind of few peaks in low Q region and then data goes into flat background. And from here, we can find out the local structure in glassy materials. Here, we have found out the structure. You can see the fit we obtained using a reverse Monte Carlo technique which again uses the χ^2 minimization process. But the minimization is done through a Monte Carlo technique here.

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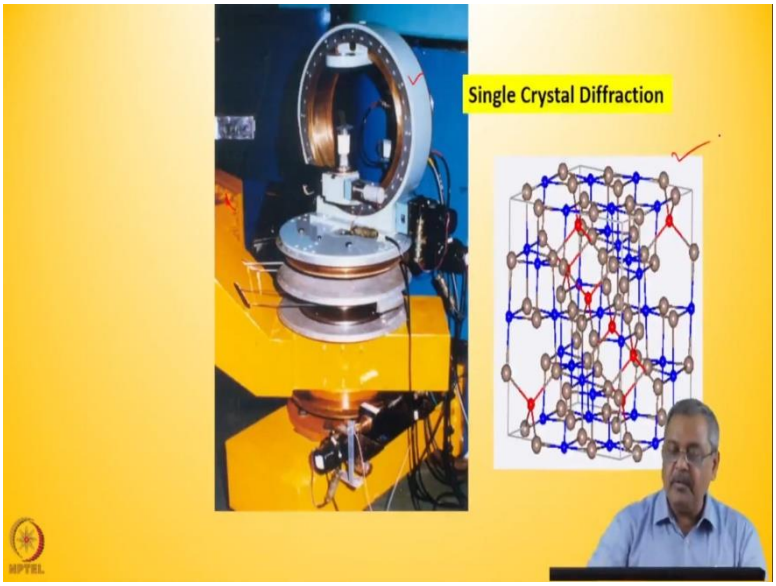

We also discussed very briefly single crystal diffraction studies. It is difficult to get single crystals. But if you have organic single crystals, these studies can provide ab initio structure unlike Rietveld fitting. Regarding the diffratometer, the difference is that we have a large goniometer on the sample table. Because the single crystal needs to be oriented, so that the Bragg reflection comes onto the horizontal plane in which the detector is rotating around the sample. This is the detector arm here. You can see the detector in this photograph. One can find out the structures ab-initio using single crystal diffraction.

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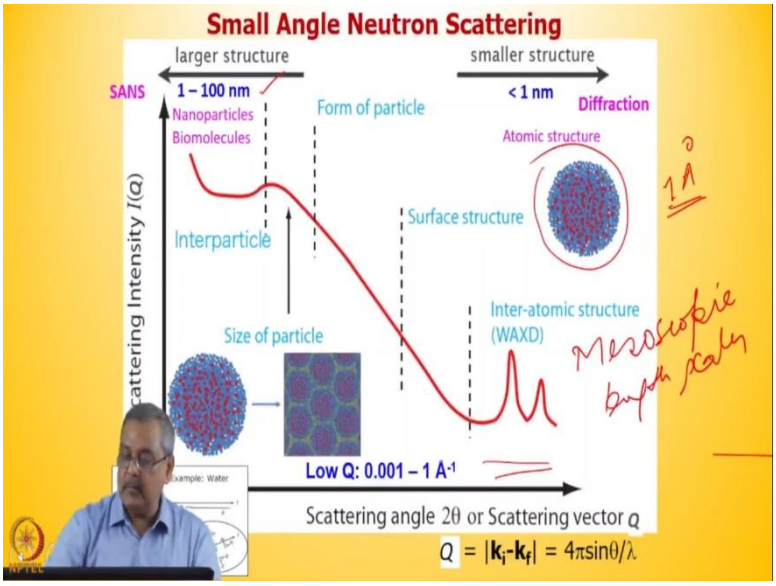

It was followed by small angle neutron scattering (SANS). In a small angle neutron scattering, you can see that the length scale being probed drastically changes. In case of crystallographic material, we talked about structures with 1 Å resolution. Now, we are talking about sizes which are large, 1000 Å or 100 nm to close to crystallographic structure. This entire range covers what we call as small angle neutron scattering.

In these SANS studies, we do not look at the crystallography structure of a medium but rather at inhomogeneities like this one that you can see as shown here in the schematic. Inhomogeneities and the size of this inhomogeneities in a medium, which are often of lot of interest in industries, for chemists, for biologists, metallurgists, a large class of material scientists. And small angle neutron scattering is an important tool, if we want to look at mesoscopic length scales.

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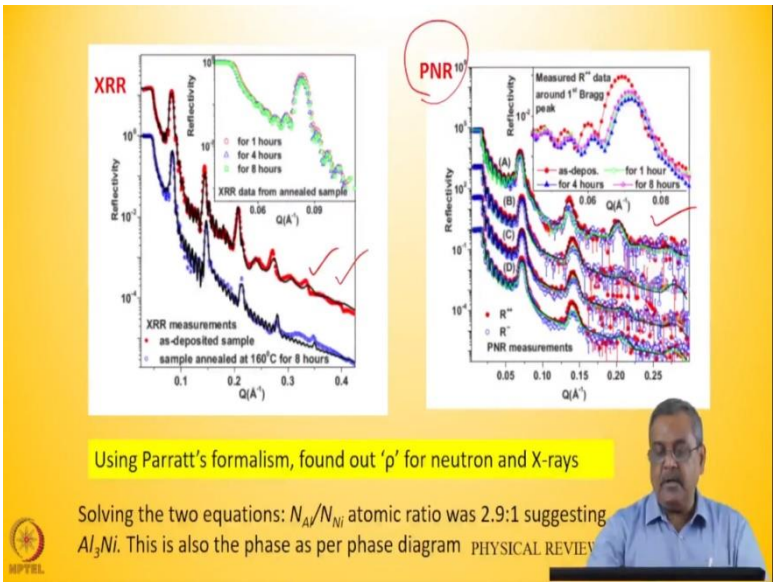

Then in the same mesoscopic length scale we can use neutron reflectometry for understanding structure and magnetism in thin films. And we discussed the techniques of neutron reflectometry. There are two kinds of reflectometry, specular and off specular. In case of specular reflectometry as you see here, the results are shown.

In case of specular reflectometry, the experiment follows Snell's law; the incident and the outgoing angle after reflection are same [$\theta_i = \theta_r$. Incident angle is equal to angle of reflection. And here also by solving for the structure, you can extract data regarding thickness of layers, interlayer roughness, magnetic moment density in thin films and magnetic materials.

This is a very important tool for understanding magnetism in thin films. There is a technique called Parratt's formalism, using which we can actually fit a given structure to the experimental data. And these fits are obtained from PNR data. The Polarized Neutron Reflectometry for a sample is shown here. This is a nickel-aluminum alloy and this is the x-ray refrectrometry data [left side panel]. The two techniques are very similar. Only advantage in case of polarized neutron reflectometry is that we have an added advantage of finding out the magnetism at mesoscopic length scale.

In case of XRR we can find out the physical structure like density and thickness of thin films. Avantage is that, XRR is a technique which is much higher intensity often compared to PNR. So, any thin film sample, before we carry out any PNR on that, it is desirable that we do an XRR measurement to find out the structure of the film.

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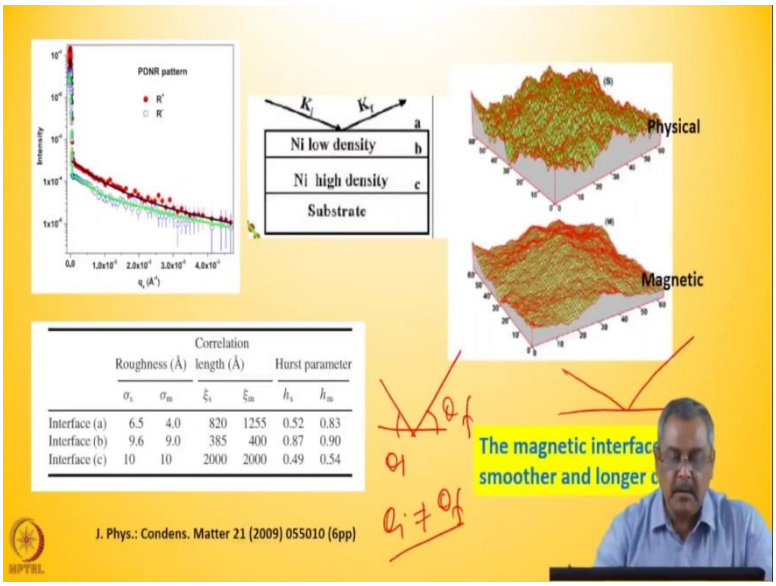

And we also discussed off-specular neutron scattering where the angle of incidence and an angle of reflection are not same.

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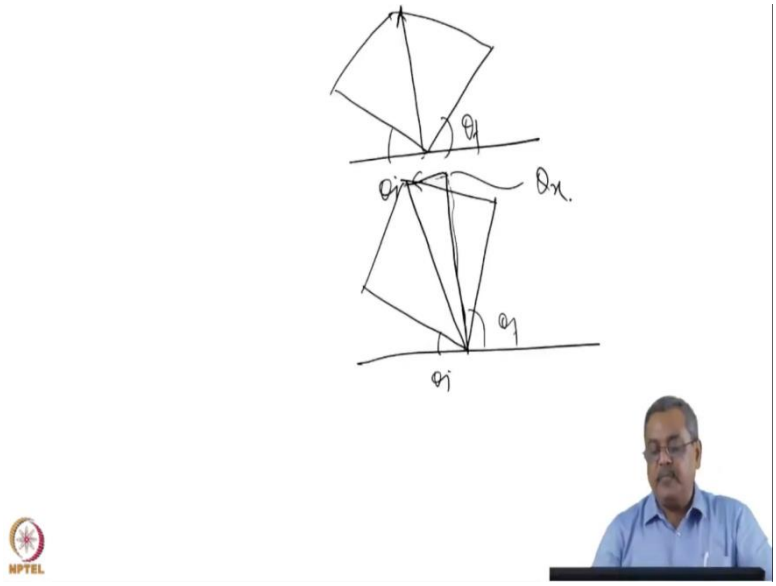

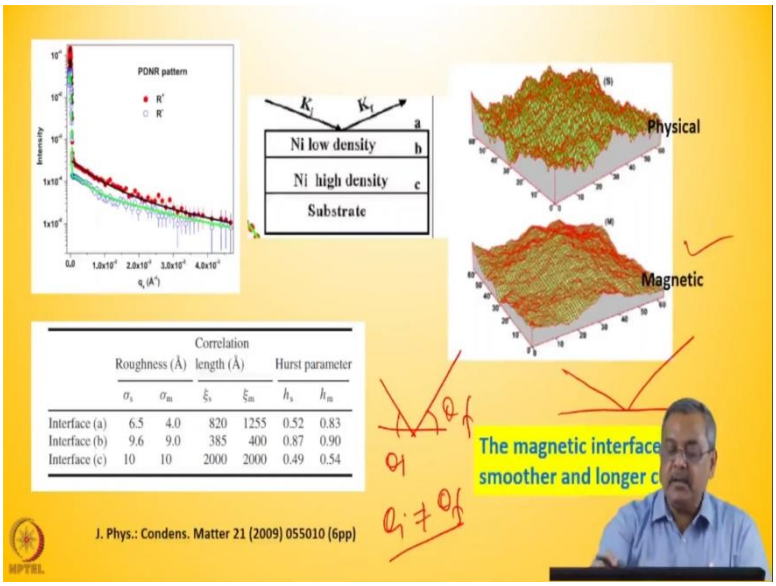

Let me just remind you, when these angles are same then the wave vector transfer is normal to the film surface. But when they are not same then the wave vector transfer, *'Q'* is at an angle to the normal to the film surface and we have got a component which is along the film surface. We have a normal component and a component which is along the film surface which can be called Q_x. And this study gives me height-height correlation on a surface. So, here, in the shown example, I talked to you about off-specular neutron reflectometry, especially for magnetic interfaces. And I showed you how magnetic interface is smoother than a physical interface.

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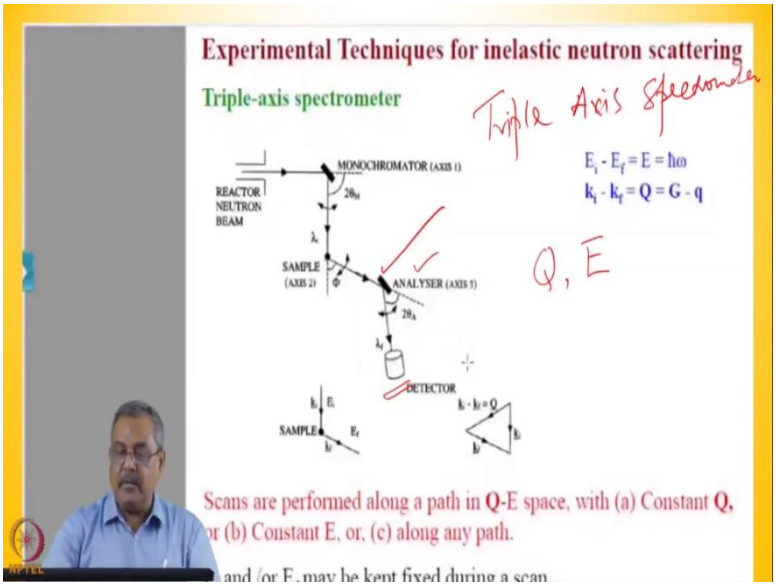

At this point, I switched over to inelastic neutron scattering and I discussed with you how we can do inelastic neutron scattering. Here in a reactor, we have a monochromatic neutron beam prepared by a monochromator. After that there is a sample followed by an energy analyzer which is important in inelastic nuetron scattering as you need to know not only momentum transfer *'ΔQ'* but also the energy transfer *'ΔE'* in the experiment. And that energy transfer is measured using an analyzer crystal after the sample, followed by a detector. This is known as a triple axis spectrometer. It is very commonly used, in almost all reactor sources for studying inelastic neutron scattering.

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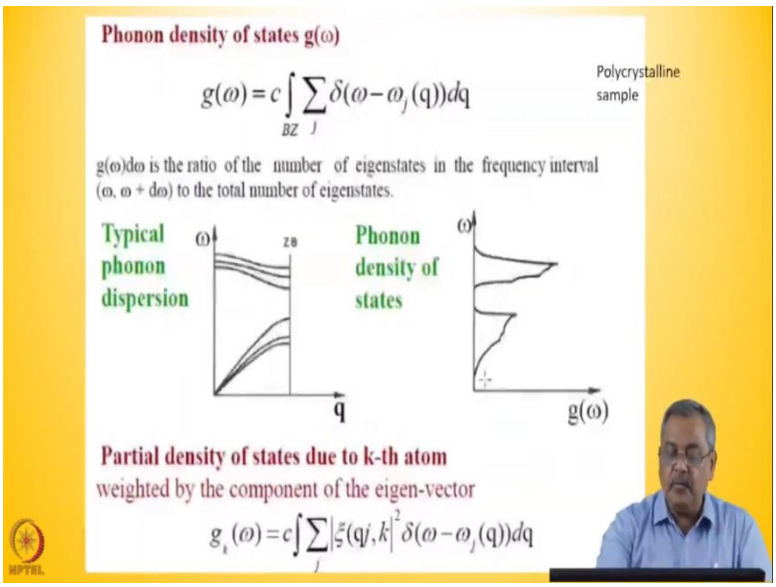

By using inelastic neutral scattering, we can find out phonon dispersion relation in crystals and also phonon density of states in powder samples as I discussed with you.

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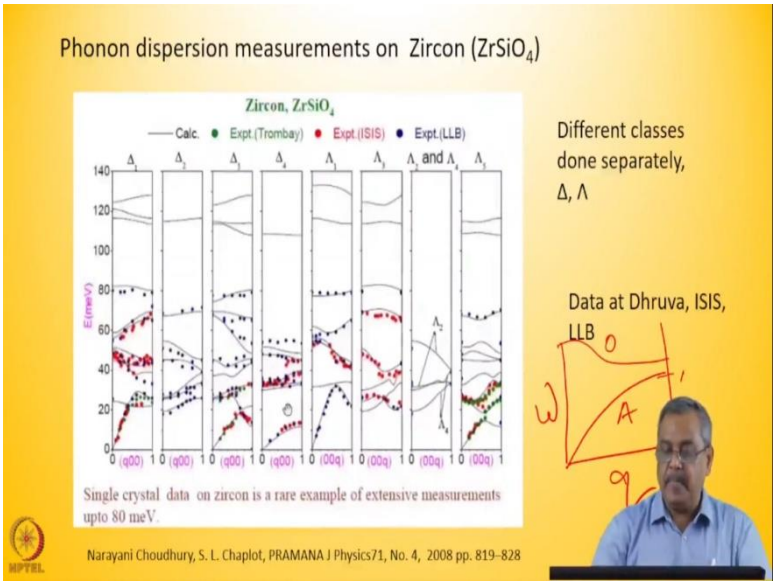

This is an example of measurement of dispersion relation in zircon. Basically, dispersion relation means we have omega (ω) versus wavevector (q) inside a Brillouin Zone. And we talked about acoustic phonons as well as optic phonons and transverse and longitudinal phonons. This data is all about the phonon dynamics in zircon. This is important because by understanding the phonon dynamics, one can understand many thermal and thermodynamic processes in the solid.

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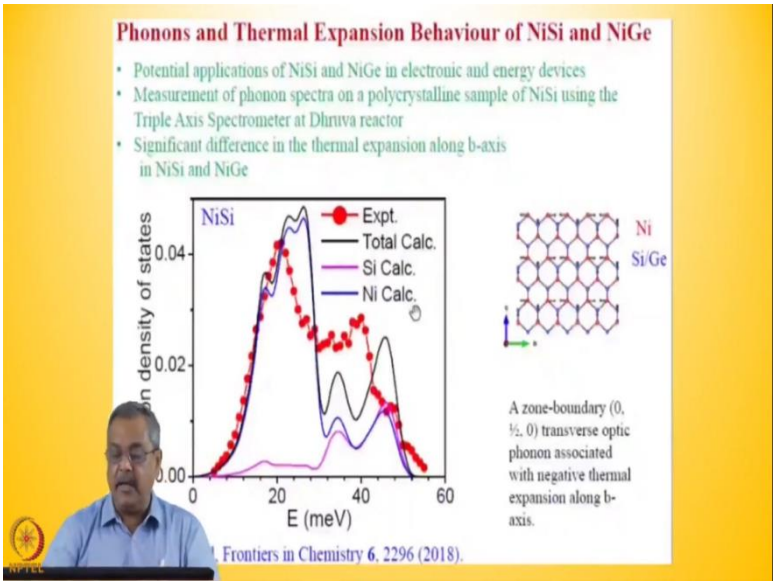

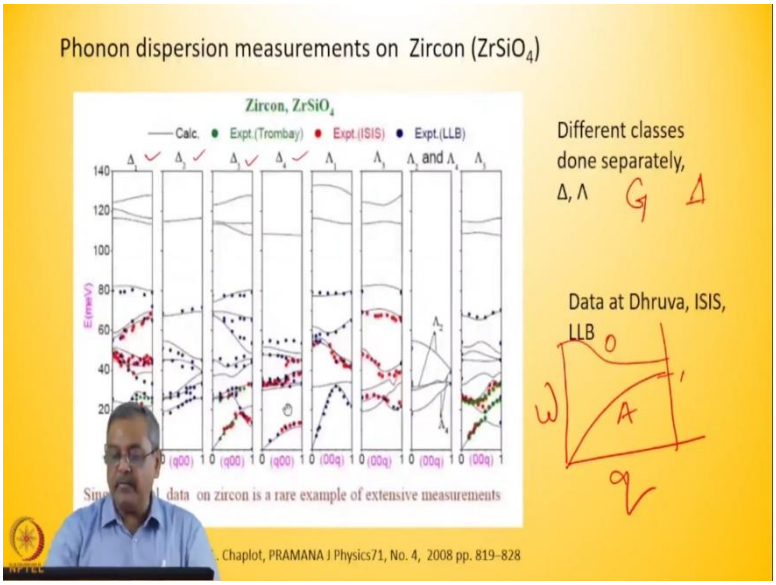

This is an example of measurement of density of states. You can see here we have found out *E* as a function of *Q* and this is as I mentioned to you earlier while discussing it in details that we need to take care of the crystal symmetry in our calculations. And from the crystal symmetry and from group theoretical calculations, we find out at which reciprocal lattice vector *'G'* I will do the measurement, for a particular class of movements in a phonon dispersion relation.

Mentioned here are the various group theoretical class of motions that have been measured and this is a combination of data from various sources. In case of phonon density of states, since, we use a powder sample, so there is an averaging over the *Q* range and this is how the data looks like.

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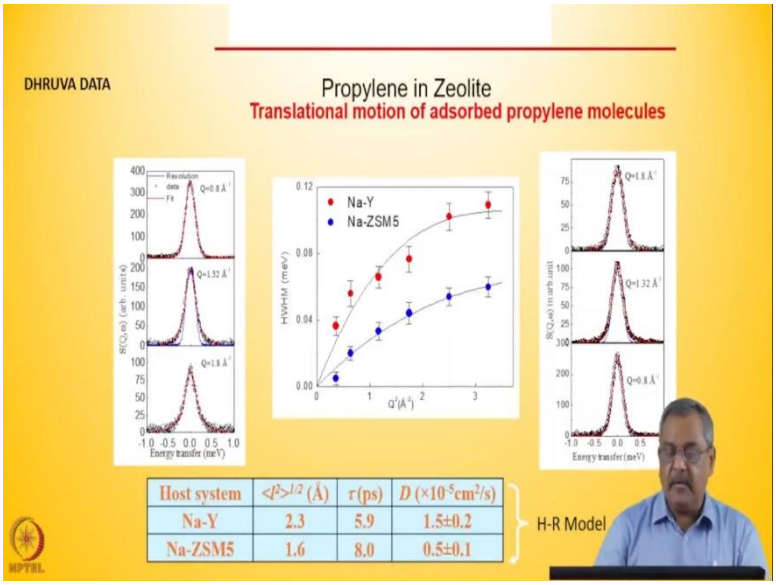

Host system	$\langle r^2 \rangle^{1/2}$ (Å)	τ (ps)	D ($\times 10^{-5}$ cm^2/s)
Na-Y	2.3	5.9	1.5±0.2
Na-ZSM5	1.6	8.0	0.5±0.1

We followed it by quasi-elastic neutron scattering echnique, which I discussed in last two lectures. This is for understanding diffusion in material and this I discussed with you with respect to various organic materials in zeolite cages when I chose the examples for Dhruva. Also, I showed you how such slow dynamics can be measured using spin-echo spectrometer, at the beginning of the lecture today.

This is a quick summary of all the things that we did in neutron scattering during these lectures delivered by me. Thank you.