Neutron Scattering for Condensed Matter Studies
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Week 7: Lecture 19 A

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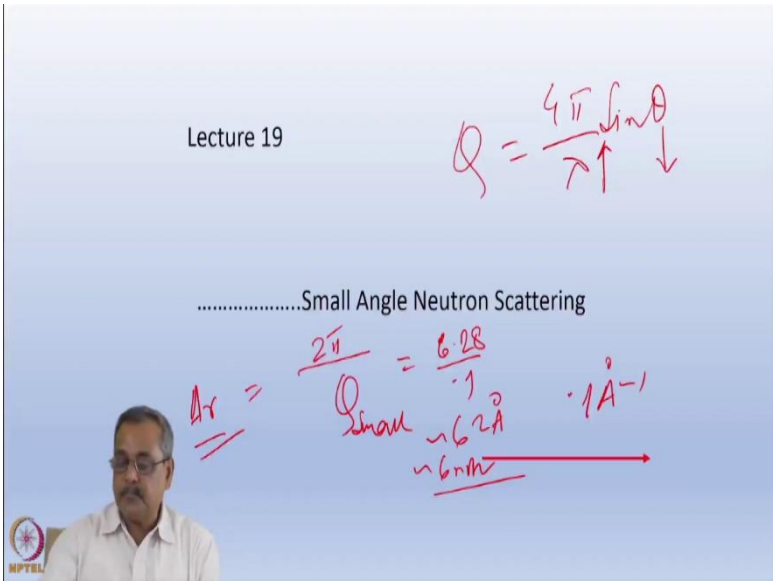

In this lecture, I will continue with our discussion on small angle neutron scattering. I have introduced you to the subject and I told you that these are basically experiments that we do at small Q. As I told you that $Q = \frac{4\pi \sin\theta}{\lambda}$, so, for smaller Q, we require either to go to smaller θ or larger λ. But in general, the technique is known as small angle neutron scattering, and I had just introduced you to the subject and I told you that, when the Q is small, the inherent resolution in an experiment is given by $\Delta r = \frac{2\pi}{Q}$. For example, if Q =0.1 Å^{-1} then Δr is around 62 Å or around 6 nm. So, I have intentionally reduced the resolution of my experiment to look at my sample [particles] in a broader scale. This is a very important technique used in soft condensed matter and also for studying precipitates in metallurgical samples and pores in rocks. I will explain them with examples because that is important.

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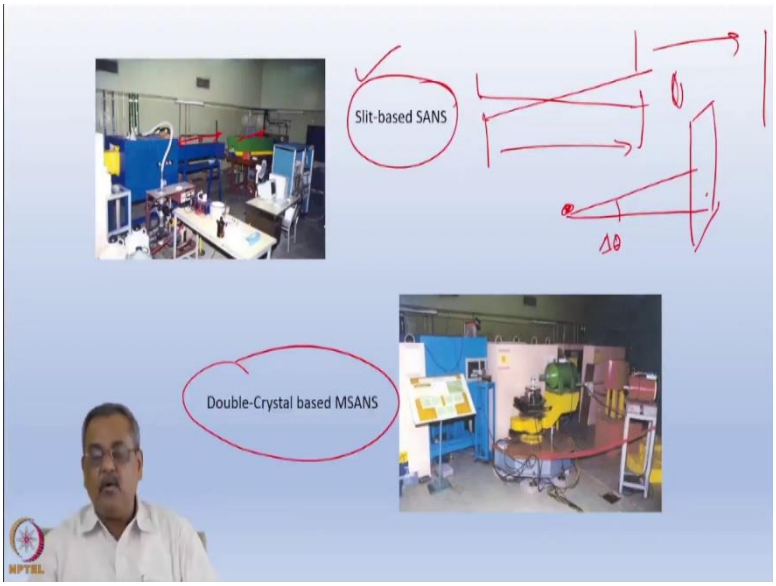

I also discussed with you the two instruments that we have in Dhruva, one is the slit-based SANS, other one is double-crystal based SANS. Slit-based SANS is the commonly used instrument in most of the sources, because here we use primarily a long flight path. So, you have got a large distance between two slits, that dictates the resolution, then you have a sample, then you have a further large flight path before it goes to detector.

This large flight path is used because your sample scatters in a very small angular window in which you are interested and to expand that angular window onto let us say 2-dimensional detector or a linear detector, I need to move it outward. So, these distances are long. In this old module, we had a lot of reasonably long flight path you can see it in this diagram. And also then there is a large detector shielding at the back with the one dimensional detector, this is a normal slit-based SANS.

We have also something called a double-crystal based SANS, we call MSANS, medium resolution SANS.

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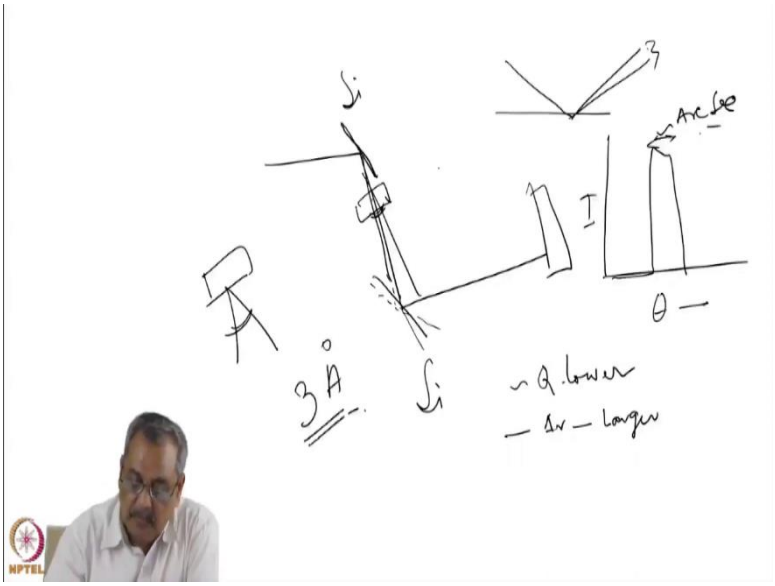

Here basically what we have are two perfect silicon crystals in parallel geometry. Now, please understand that if I have a perfect single crystal here it gives a rocking curve width which is known as Darwin width, which is few arc seconds in intensity versus angle plot for a Bragg angle. Now, the beam of this width falls on the second crystal and then if I rock the second crystal it will trace the width of the first beam onto the detector. So, if it is a perfect single crystal, then the rocking the second crystal gives me the width of the beam in the first silicon single crystal or any other perfect crystal used.

Now, if I put a sample between the two crystals, then this sample causes a widening of this very narrow beam. And when that happens, then the rocking of the second crystal actually catches the widening of this beam. Unlike other instruments, here two single crystals cleverly manipulate a very low *Q* region. We can go to lower *Q* range and study the sizes that are larger.

In this instrument which I am discussing, the incident wave length is around around 3 Å.

In picture, you can see the double crystal monochromator-based system.

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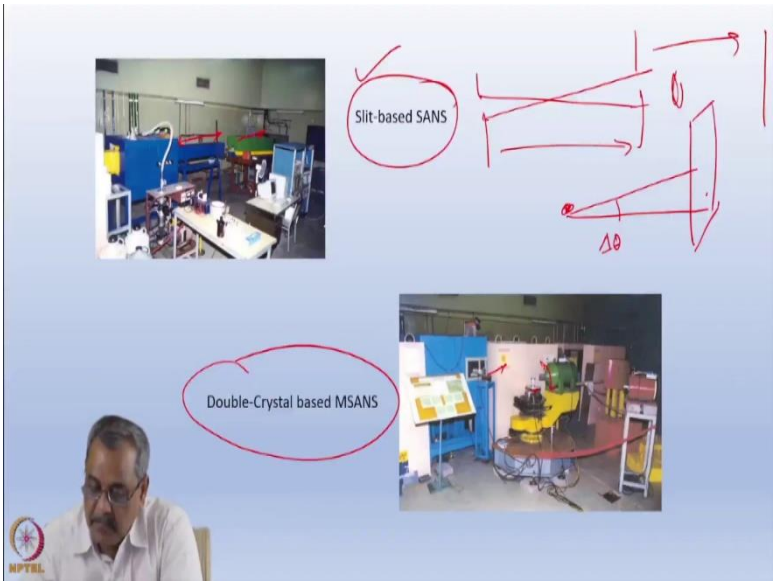

You can see one crystal is here, and this is the second crystal, which is rocked with a very precise angular movement. The sample comes right after the first crystal in the beam path. This rocking curve is arrested by θ-2θ movement, this is the detector which is rotating locked with the second crystal in a θ-2θ mode. There are two instruments which we have got.

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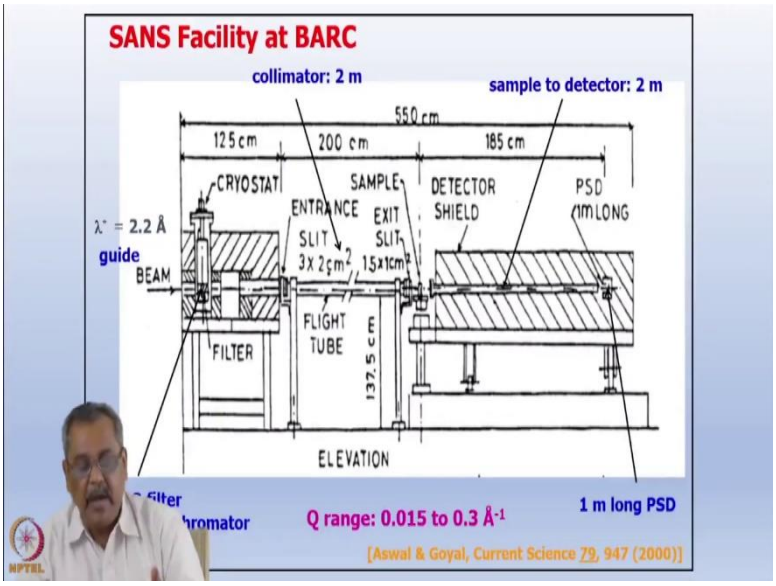

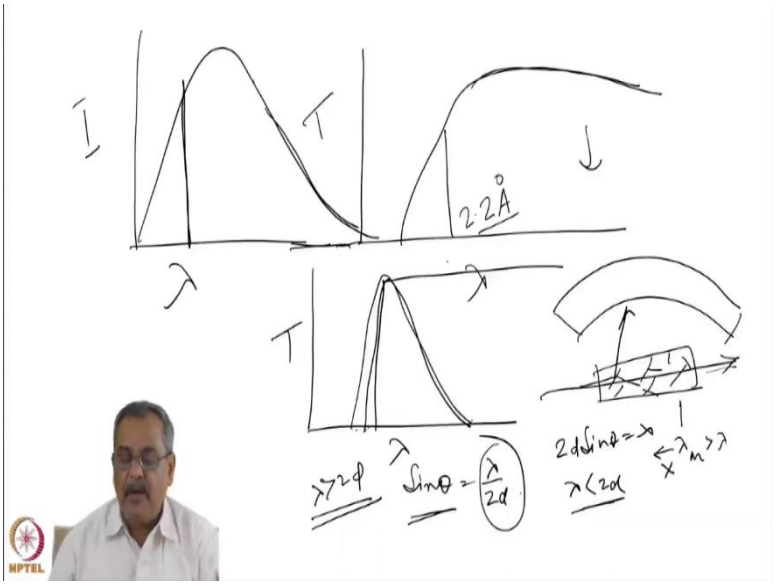

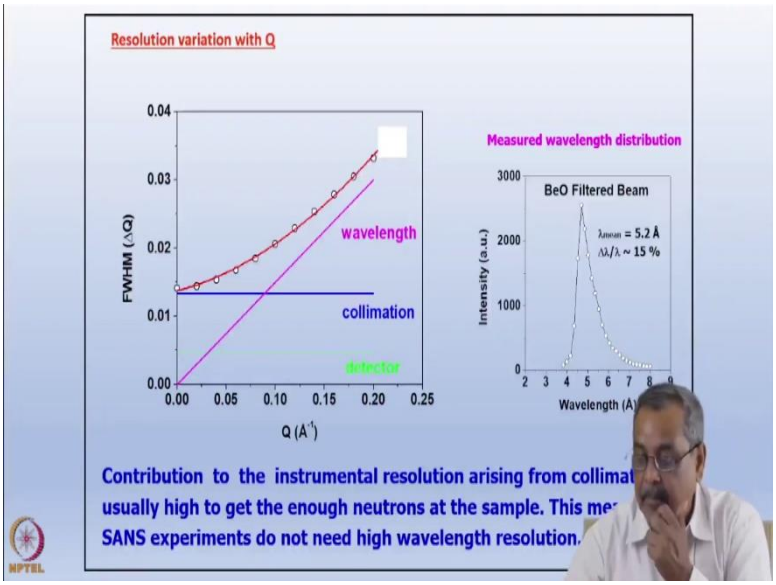

Briefly describing what we had earlier. It was a Beryllium Oxide filter-based monochromator. It is not really amonochromator, it is more of a filter, but it does have an interesting purpose. I can narrate to you, what is a combination of various things. A guide has a transmission curve which has this intensity versus λ curve with a cut-off wavelength which depends on the bending angle of the guide. This cut-off is because of the radius of curvature of the guide.

In the guide on which this instrument is put, the cutoff is at 2.2 Å. Beyond that I have a flat intensity with respect to λ. Now, that is what the guide transmits. In fact, the incoming beam in the guide is a Maxwelian and because of cut-off at 2.2 Å, guide blocks neutrons with wavlengths smaller than this and higher wavelengths are transmitted.

But now, there is a Berillium oxide filter which is a polycrystalline material. In a polycrystalline block, there are crystallites at all possible orientations inside the block. If I have a block satisfying $2d \sin\theta = \lambda$ that wavelength will be scattered out. Because in a polycrystalline block there are many many many crystallographic planes and we know $\sin\theta = \lambda/2d$, so, till $\lambda < 2d$, I will get $\theta < 90^o$ degree and some block or other will reflect out neutrons from the beam path and when $\lambda > 2d$, the wavelength is transmitted. So, there is one more cutoff wavelength for the polycrystal above which the beam passes through.

There is a filter which has got a cutoff at λ_{min} and for $\lambda < \lambda_{min}$ neutrons are not allowed to pass they are reflected out by Bragg reflection and for $\lambda > \lambda_{min}$ they will pass through it.

This is a transmission sectrum and there is this polycrystalline blockin an ideallistic case will that give a transmission like this with λ. But all these three things are multiplied together and what you get actually is a sharp rise and the fall because on the longer wavelength side of a Maxwellian. The intensity is falling and in the shorter wavelength side you have a cut off with this Berillium oxide and also on the guide.

This cutoff is not so sharp as we look at it ideally but it is more gradual. So, it will be somewhat like this.

So, this is Berillium oxide filtered beam, which is not a perfectly monochromatic beam. Actually $\Delta\lambda/\lambda$ is quite large 15% and the mean λ_{mean} is 5.2 Å. This is what we were using earlier.

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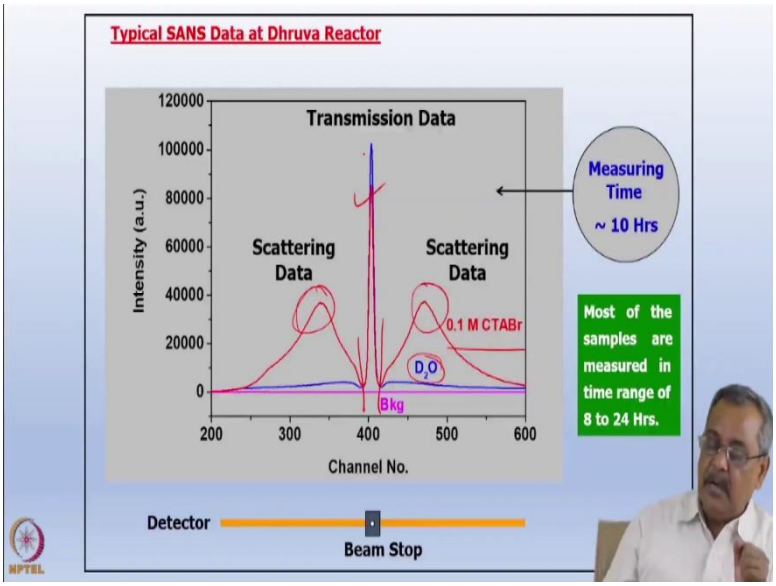

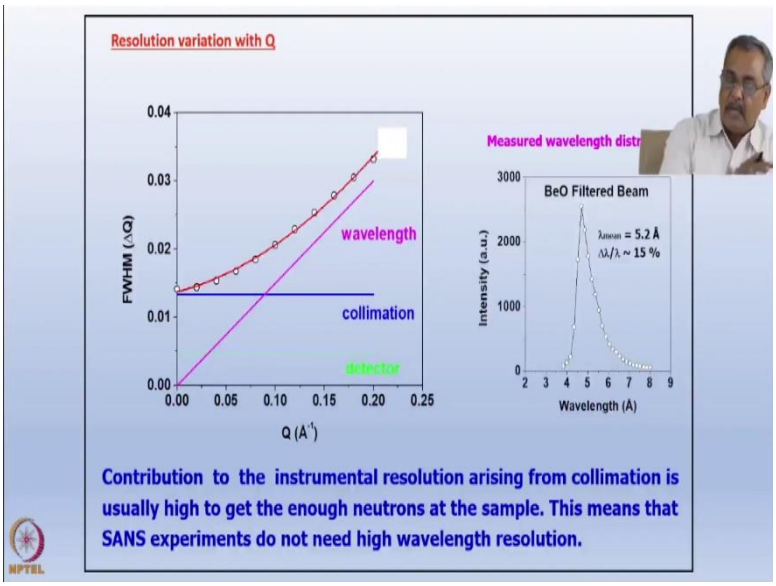

When I impinge this beam on a sample, I have got two parts, one is the transmission data which you see here, that is the direct beam which has passed through the sample and put its footprint on the one-dimensional detector and you have scattering data on both the sides of the detector.

On a 1-dimensional detector, for a 0.1 molar solution of CTAB you can see this is the small angle data we collected. We have also shown the data from the D_2O. The CTABs are basically large inhomogeneities in a solution and D_2O has nothing like that structure. It is just a solvent and you get a flat background with a direct beam profile from pure D_2O.

That means, in these experiments, we could not go below these angles, because at the smaller angle side, this will cut the direct beam and we have got data only up to a certain angle small

angle. This is coming as intensity vs channel number over here in a 1-dimensional detector, which will dictate the Q value. And I have just taken the trouble to plot the resolution with changes, you can see with the *Q* or the angle or the channel number in the 1-dimensional detector and this is what the resolution function is.

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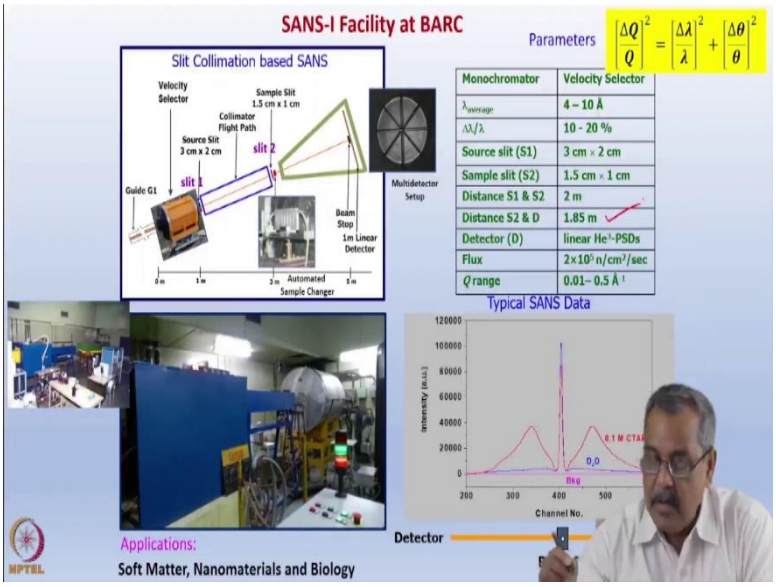

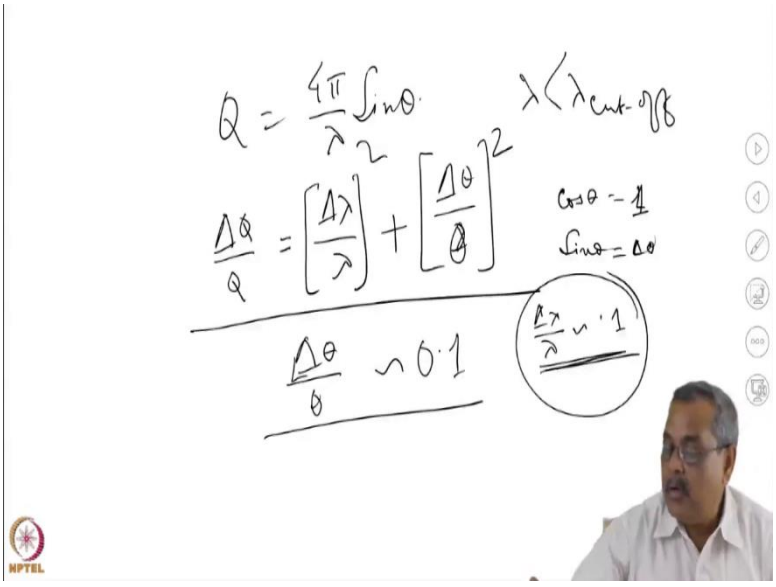

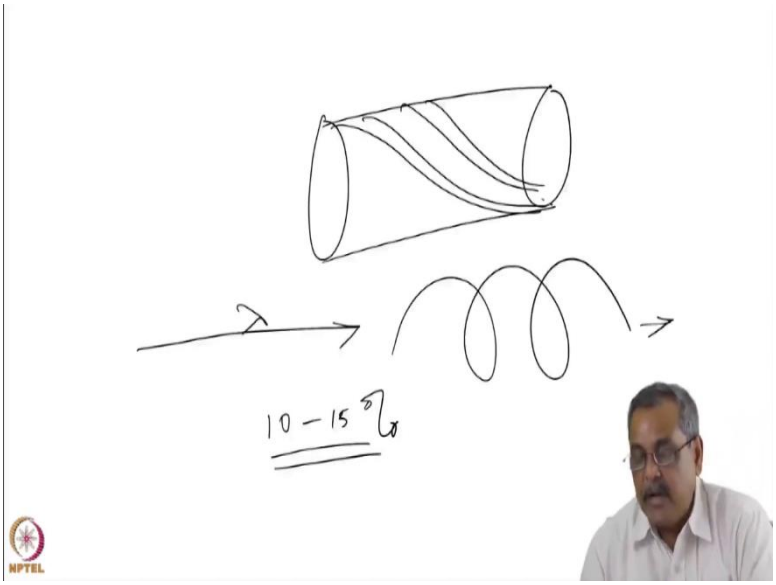

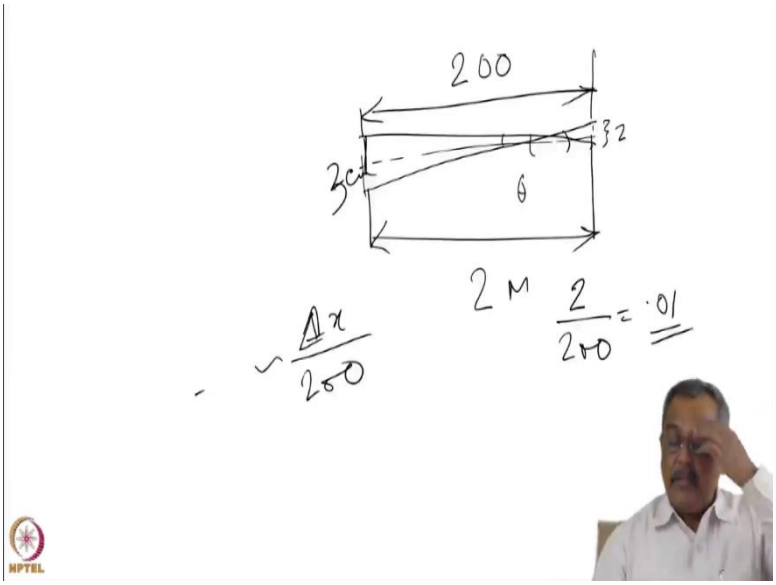

This has changed now, with the velocity selector. What does the velocity selector do? Earlier I had a Beryllium oxide filter. The beryllium oxide filter was cooled, because if I do not cool the polycrystalline block which is acting as a filter, it will cause thermal scattering of the neutron which is undesirable as I want a sharp cutoff and I want neutrons of wavelength $\lambda < \lambda_{cut-off}$ to be completely stopped from going. That is why we used to cool the Beryllium oxide polycrystalline block. Now we have a velocity selector in this small angle neutron scattering instrument.

We know that $Q = \frac{4\pi \sin\theta}{\lambda}$, with this we get, $\left[\frac{\Delta Q}{Q}\right]^2 = \left[\frac{\Delta\lambda}{\lambda}\right]^2 + \left[\frac{\Delta\theta}{\theta}\right]^2$ considering $\sin\theta = \Delta\theta$ and $\cos\theta = 1$ for small angles.

For small angles, $\frac{\Delta\theta}{\theta}$ will be of the order of 10% or 0.1. But if this is acceptable, then from the expression you can see from this expression that $\frac{\Delta\lambda}{\lambda} = 0.1$ should also be acceptable to us. So, one takeaway from this expression is that, if I am working in the small angle neutron scattering zone, I can relax my wavelength resolution. This I can not do when I am working at a larger angle in a crystallographic diffraction. So, I can accept $\frac{\Delta\lambda}{\lambda} = 0.1$ in small angle zone and how to do it? We can do it mechanically.

I told you this earlier and again I will quickly tell you that a velocity selector is nothing but a cylindrical block, which has got helical slots cut on the body and the neutron of a certain wavelength which is traveling in a straight-line path in this rotational frame of reference, when I convert this, it becomes a helix. And if that helix which matches the helicity of the neutron, it will pass through the velocity selector. So, though it is called velocity selector, basically it is a wavelength selector and in this mechanical assembly, I can get 10 to 15 percent velocity width which will add to the intensity and at the same time we will not disturb the experiment because I know my $\frac{\Delta\theta}{\theta}$ is of the order of 10%.

Here you can see that we can calculate this. The source slit is 3 cm × 2 cm and the sample slit is 1.5 cm × 1 cm. Before the sample a 2 m distance have been kept. There is a 3 cm slit and there is a 2 cm slit, so, this is the angular resolution $\Delta\theta$ for this distance of 2 m. So, this is 2 cm, this is 3 cm, this separation is 200 cm. I can do it trigonometrically, a very simple task. But without doing it, if we assume that they were equal it will be approximately $\Delta x/200$. Here it is slightly different, it will be $\Delta x/200$. This is $\Delta\theta$ assuming $\tan\theta = \Delta\theta$. if I consider 2 by 200, so, it is around 0.01 as you can see. In this case, this is the angular width which I can tolerate here and distance from S2 to the detector is 1.5 m. And here, the detector is a multidetector setup. I have given the expression for the resolution. And I have also given you the detector setup, which is 1.5 m away. It is an improved version and this is being used till few years back.

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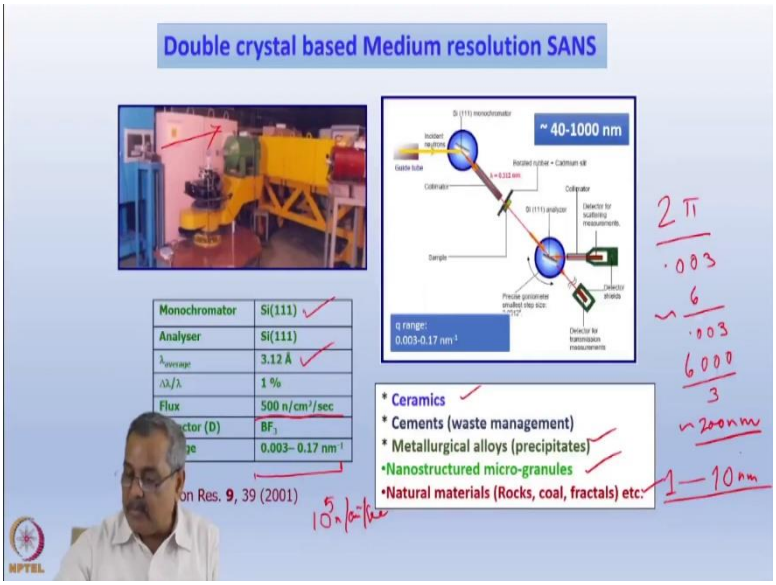

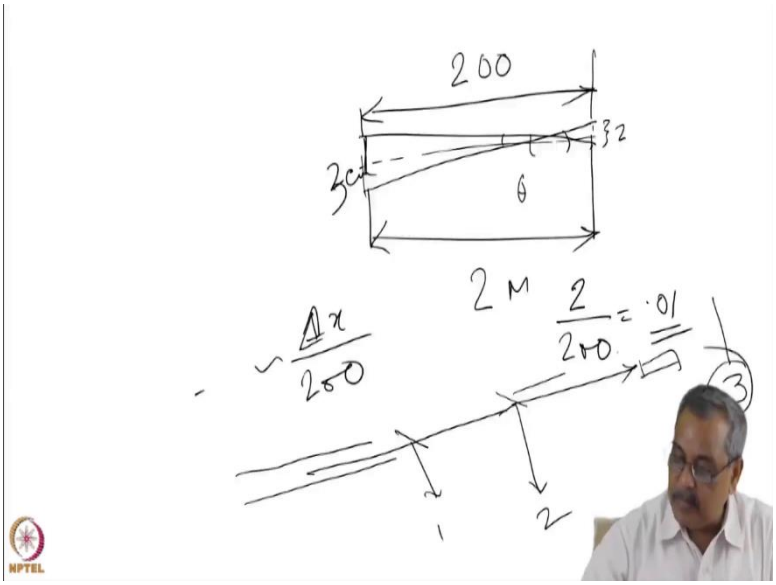

This is a Double crystal based medium resolution SANS, which I explained to you just now. Both of these crystals are Si single crystals in (111) reflection geometry with respect to incoming guide beam.

From the guide neutron beam comes out to this instrument and the guided neutron beam goes on farther and then the beam comes out to the reflectometer, and then this guide goes to the SANS. Then there is a velocity selector and the SANS machine. So, there are 3 instruments on the same guide. Here I am talking about the first outlet in this neutron guide, and that is the advantage of neutron guides. On a single guide, you can put number of instruments and here we have put 3.

Medium resolution SANS is one of them. In this instrument, we have Si(111) as the first monochromator and also Si(111) as the analyzer which rocks with in the $\theta - 2\theta$ mode to obtain the broadening caused by the sample, the λ_{avg} is 3.12 Å and here $\Delta\lambda/\lambda$ is much better. It is 1% but the flux is low. In the previous SANS machine that I showed you the flux was around 10^5 n/cm^2/sec while here the flux is much smaller that is 500 n/cm^2/sec.

These numbers are actually quite small, if you compare them with a standard x-ray machine, but we can work with it. There is a BF_3 detector and the Q range is actually something which you need to see, it is 0.003 to 0.17 nm^{-1}. Here Q_{min} is 0.003 nm^{-1} and if we use, $2\pi/Q_{min}$ we can se much larger objects that is around 200 nm. The other instrument which I described to you, typically around 1 nm to 10 nm inhomogeneities will be its range. It can be slightly less or slightly more. But here I can go to much larger objects and the studies done on MSANS include ceramics, metallurgical alloys, micro granules with fractal geometry. I can not give example from all of these but I will try to choose some of the examples to explain to you the utility of this particular SANS machine.

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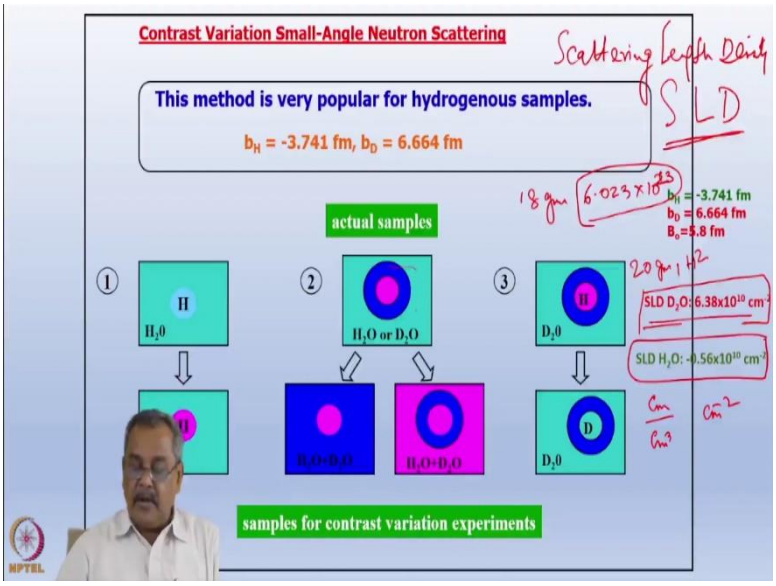

Another important tool in case of neutron is using the contrast matching. This is interesting, we are talking in terms of scattering length density (SLD), and not in terms of atomic scattering strength.

We are talking in terms of macroscopic parameters like density, and not in terms of microscopic atomic structure, because here, I have intentionally reduced the resolution. Reducing the

resolution is making the sensitivity broader in terms of size. So, in that size scale, I do not talk about atomic structure of the medium, but we will talk in terms of density.

I have a sample, [what the sample will be I will come to it later], and suppose, it has got a scattering length density contrast, in a medium, most commonly, in a liquid medium. Such samples are basically soft materials, sometimes proteins, sometimes surfactants, micelles and polymers etc. Such materials mostly studied by small angle neutron scattering, in the liquid medium. Please check, I worked it out for you last time. The scattering length density for D_2O is 6.38×10^{10} cm^{-2}. The way I did it, if you remember, I worked out the scattering length for one H_2O and D_2O molecule as $2b_H+b_O$ and $2b_D+b_O$, respectively. In 18 gm of H_2O you have 6.023×10^{23} molecules and in 1 cc of H_2O we have 1/18 gm. Similarly, you can do the same for D_2O by taking its molar mass as 20 gm. Now it is we know how much is in 1 cc and finally we get the scattering length density in units of per centimeter square. Scattering length density of D_2O is positive while that of H_2O is negative which is quite interesting for contrast variation.

Now, as you can see, I can lighten up different parts of my scatterer. If it is a core-shell structure I can light the core, I can make the contrast strong and I can match the contrast of the shell with the surrounding solvent by judiciously choosing D_2O, H_2O mixture. And I can only see this part or this part, either the core part or the shell part.

And you can see by color showing it shown that when I match the scattering length density of the solvent with that of the shell, then what I see is this or when I match the scattering density of the core with the solvent, what I see is the shell and I can play with the contrast and it is very routinely done for neutron small angle scattering.

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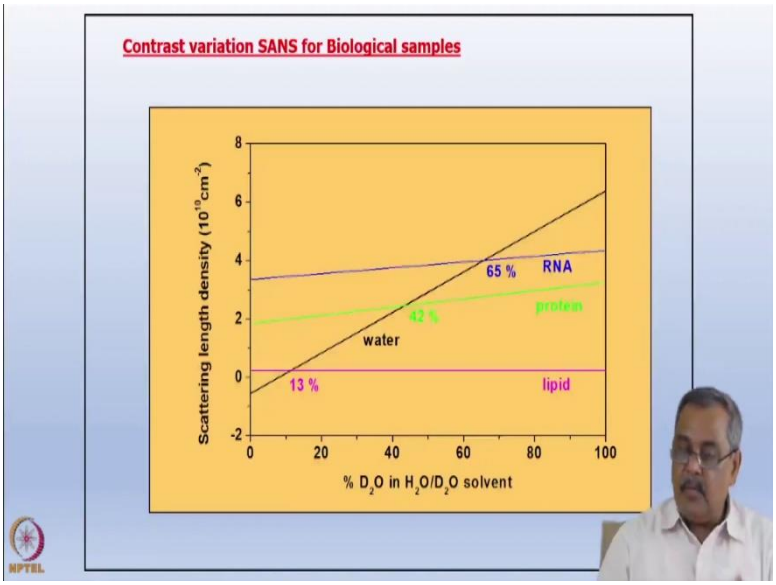

This is a plot of that the contrast., You can see some of the routine samples which are used regularly in small angles scattering viz. lipid, RNA, protein. You can see their scattering length densities and as you can also see that percentage of D_2O and H_2O/ D_2O mixture. If it is 100 percent it goes to 6.0 something for D_2O and this is -0.57 and as you keep mixing D_2O and H_2O, we can go across the whole band of scattering intensities, where you can match with the solvent scattering density for various parts of a scatterer as I showed you.