Neutron Scattering for Condensed Matter Studies
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Week 7: Lecture 18 D

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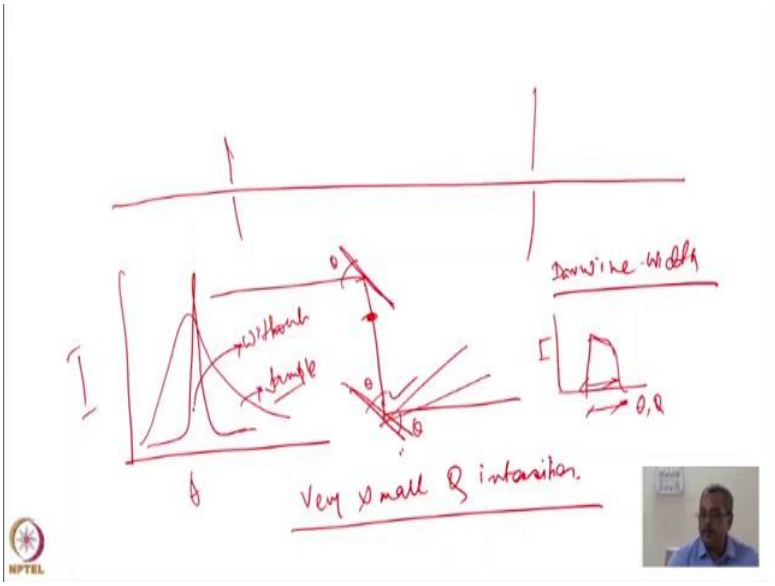

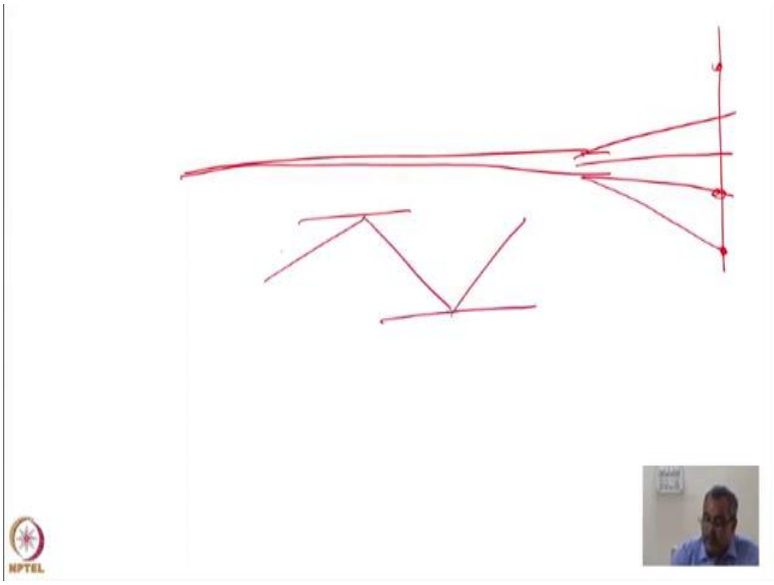

Let me come down to the instruments. I will primarily focus on the instruments that we have at Dhruva. I will also discuss some of the extremely interesting and tough experiments that have been done using SANS technique. We have two instruments, namely, SANS-I and SANS-II. Both of them are in Dhruva guide hall. SANS-I is based on velocity selector, earlier it was based on beryllium oxide filterer beam.

Most importantly, for most of the SANS experiments you are doing experiments at a small angle, so, large distances are involved. This is because you have to collimate the beam very tight. Same thing has been used by us for this. You can see the flight path, but this is not the largest that you can think. For example, if you go to advanced neutron source where you have a cold neutron source and you can afford to lose some neutrons, these flight paths are typically 15 to 30 meters long. And then, a sample comes here. This is an old photograph where the scattered beam falls on a one-dimensional detector.

There is another interesting instrument where we do not take recourse to a large flight path but there are two silicone single crystals having very small mosaic spread, close to what is known as Darwin width. For a single crystal, the reflected beam is very narrow, about few arc seconds for a Bragg diffracted beam. It also has some mosaic spread. Now, if I use two single crystals in parallel beam geometry at a certain angle, when Bragg angles are same, then if I rock the second crystal, keeping the first fixed then the rocking curve actually gives the width of the beam from the first crystal. So, you get a very very narrow beam.

It is called a rocking curve. If you rock the second crystal, it gives very narrow beam, which will be a Darwin width ideally of the first crystal, because these are single crystals In this case

they are silicon single crystals, which gives me Darwin width. Now, after the first crystal, if I put my sample here then this sample causes small angle neutron scattering of the beam. And then when I rock the second crystal, I observe the widening of the beam.

Now, I have got a beam without sample and a beam with sample. Because this beam is very narrow. It is a rocking curve which captures the Darwin width of the first crystal. This being a single crystal, I can see very small-Q intensities. When I do a conventional small angle neutron diffraction, I get a very very narrow beam and I place the detector a distance so that the beam spreads out on the detector and I can measure them at very small angles.

This is the most commonly used technique, but the other technique using the rocking curve is also used. And we are using this rocking curve for one of the SANS machines. We have got two of these small angle neutron scattering machines, one using the rocking curve principle and the another one is a common machine in which the beam travels through a long path. There is a sample after that there is the detector shielding in which the beam travels and there is a one-meter position sensitive detector or PSD to capture the beam in this case.

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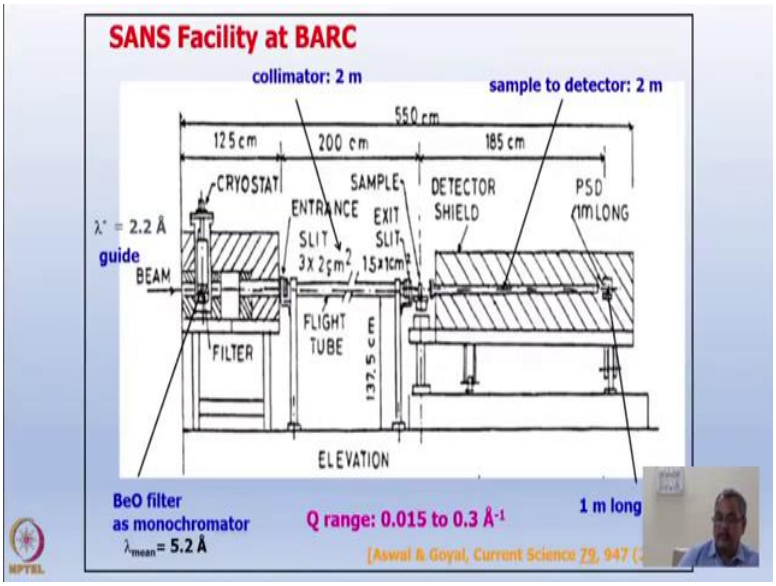

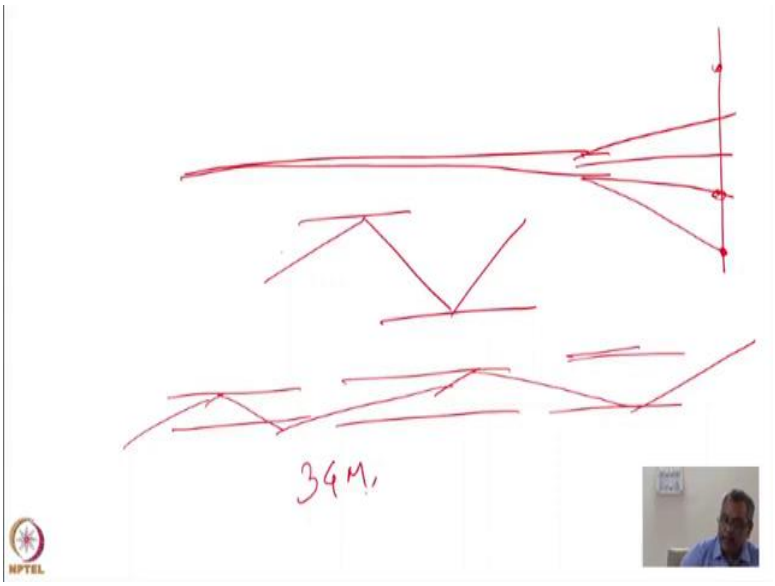

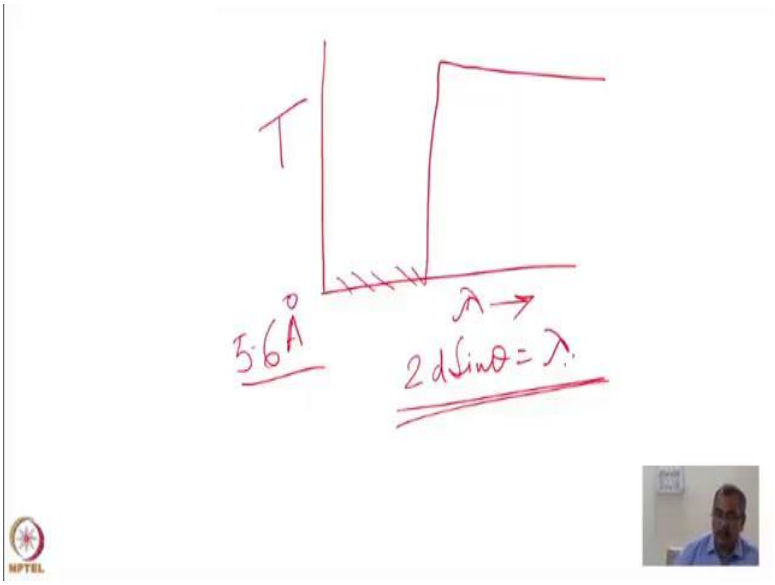

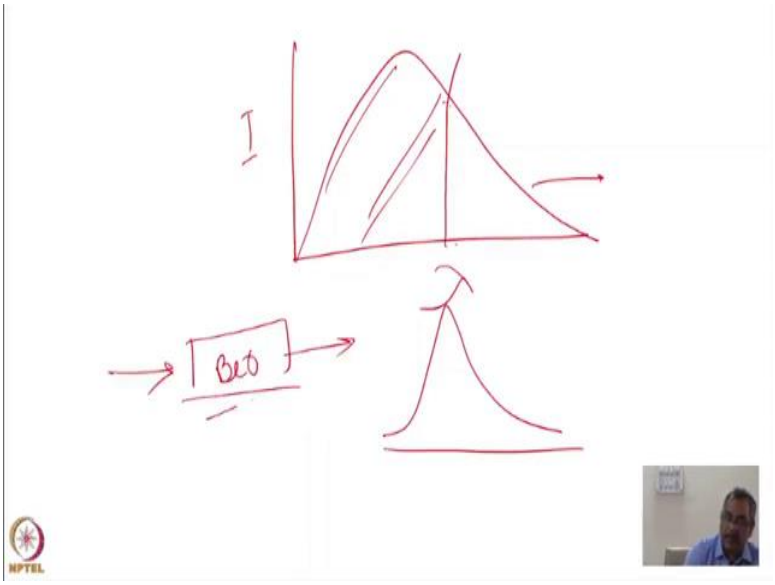

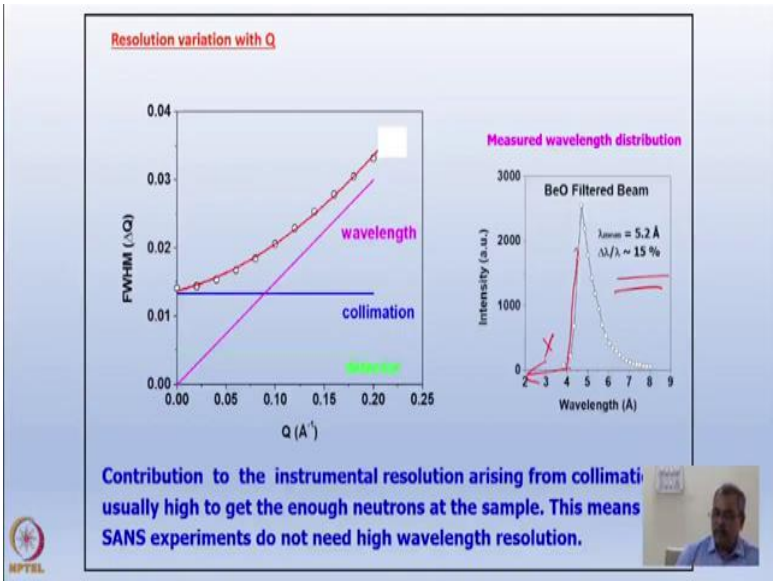

This is a schematic from this reference given. We have a guide which allows the neutron beam to travel. Earlier also, we talked about neutron guides which actualy acts as optical fibers for neutrons, where neutrons travel through total external reflection over large paths.

We have a guide, which is 34 meter long. After 34 meters, we have the beam from the guide and there is a cryostat with a beryllium oxide filter and I will quickly tell you what it is. And then there is a detector shielding followed by a one-meter-long PSD.

Now, why Beryllium oxide? This is an interesting phenomenon we used earlier. If you put a powder crystal in the neutron beam path, there are lots of crystallites oriented in all possible direction, and if I can choose *d*-spacing judiciously, we know $2d \sin\theta = \lambda$. Now, when λ is larger then the largerest *d* spacing, that will be transmitted by the this polycrystal, I showed

transmission versus energy or lambda. So, long lambda will be transmitted, but when lambda is less than the largest 2d [$\lambda/2d < 1$], then you have some d spacing to scatter out the beam and you have a cut off for in this region. Beryllium oxide has a cutoff at 5.2 Å, if I remember correct.

Now, this is a Maxwellian which is coming from reactor when one looks at intensity plotted with respect to wavelength. If I put the Beryllium Oxide filter on beam, then it will cut out the lower side. This cut off is not so sharp, it looks somewhat like this, it is a broad monochromatic beam which is transmitted when I put the beam through a beryllium oxide polycrystalline filter.

So, this is a Beryllium Oxide filter acta as a monochromator and we get a lambda mean equal to 5.2 Å and we could cover the *Q* range point of 0.015 to 0.3 $Å^{-1}$.

This is a transmission cutoff. Here below this λ all the λ have been removed in the transmitted beam and this is a tail of the Maxwellian. So, this is what [a broad distribution] we could get. This is a variation of resolution with Q as measured.

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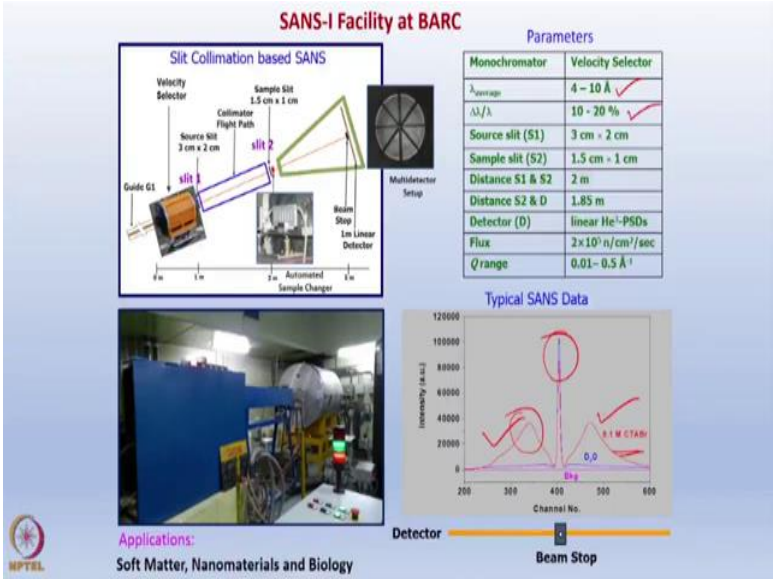

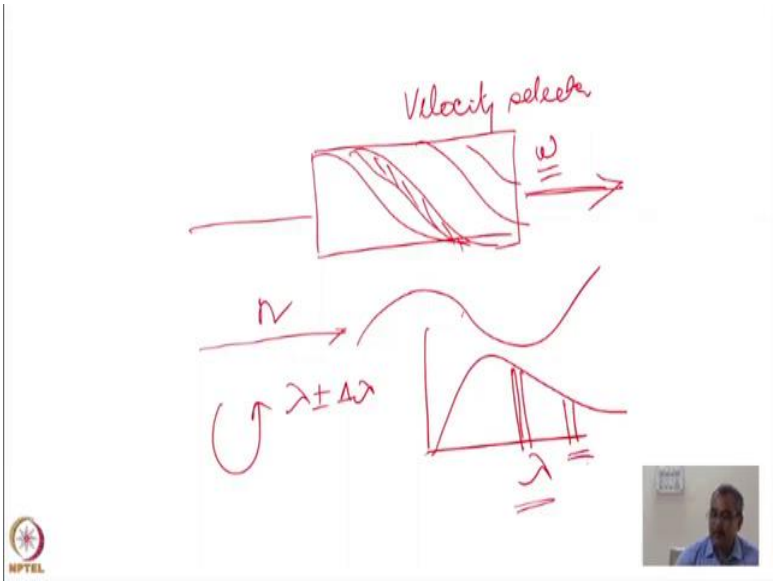

This instrument has been modified and what we have now actually, which is very commonly used in almost all the neutron sources, known as velocity selector. The physics of velocity selector I had discussed with you earlier when I discussed monochromators. Let me remind you once again, this is a cylindrical material on whose body we have cut down these helical slits and you rotate it around the cylindrical axis. These helical slots. When a neutron is passing in the laboratory frame, it is moving in a straight line. But in this rotating frame of this velocity selector, the straight line becomes a helix. And if these slots on the body of the velocity selector matches the velocity, then you have a wavelength and a velocity band so, you have a $\lambda \pm \Delta\lambda$ that is allowed to pass through the velocity selector. The beauty of this technique is that earlier we made a Barium Oxide filtered beam, where we could not play with the λ, now, depending on the rotational speed of the velocity selector, I can choose my λ because the helix for the neutron changes. Now from a Maxwellian and depending on the rotation speed of this velocity selector, I can choose a certain band with different λ_{mean} because, depending on the rotational velocity, this helix changes, and then the Helix gives me which lambda I am choosing.

That is what I have mentioned here, it can be 4 to 10 Å. And, in case of a small angle neutron scattering or SANS, we can do with a large $\Delta\lambda/\lambda$ because, this I will come to later, my resolution actually is dictated by the $\Delta\theta/\theta$ and I can use a large $\Delta\lambda/\lambda$.

So, beryllium oxide based instrument which I had earlier described has been changed using the velocity selector and the 1 D detector has been replaced by an array of detector. At any instant we can collect more data.

I show a typical data. Here you see this central part is the direct beam and this is the scattered beam which will be used for obtaining various parameters for a sample. Here it is a 0.1 molar CTAB, which is a surfactant. As I was telling to you earlier this fall on a 1-dimensional detector. But now, instead of 1 detector, I have got many, so, this scattered beam which forms as cone, I can detect the cone on these detectors. It should be centered on this, this is the direct beam and I can collect the data on large number of detectors, improving intensity. Most of the applications are of soft matter, nanomaterials and biological samples in these experiments.

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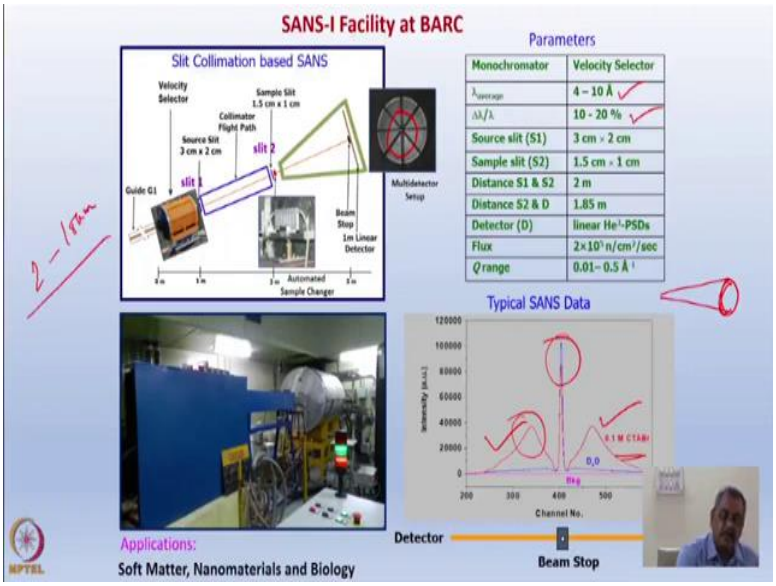

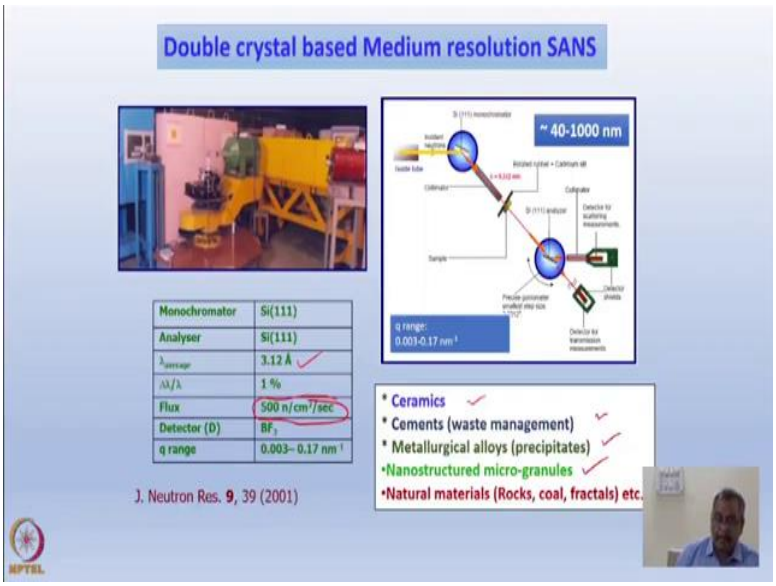

This is the instrument, which I can call SANS 1, and this instrument is SANS 2. As I explained to you earlier here you have got two Si(1, 1, 1) monochromators and there is a collimator between the two Si and you can see that the second Si is rocked around the first one. You capture the rocking curve of this one by rocking the second crystal and here the *q*-range is 0.003 – 0.17 nm^{-1}. Ceramics, cements and metallurgical alloys and precipitators have been studied heavily on what we call MSANS. This one can measure typically say 2 - 10 nm, the range of particle size in samples with SANS 1. Whereas here, in SANS 2 you can see at 40 - 1000 nm particles. So, they are complementary to with each other, and many times the data from both of them have been merged to get a larger *q*-range of data.

This is a a Si(111) monochromator and the same is the analyzer. Here the λ is 3.12 Å and the $\Delta\lambda/\lambda$ is extremely small just 1% and I must mention to you that in a reactor like Dhruva the flux on this 500 n/cm^2/sec. So do not get disappointed since this is a very, very small number compared to if you consider a beam in a synchrotron source or any other advanced neutron source. But with this kind of flux also you can study many interesting problems.

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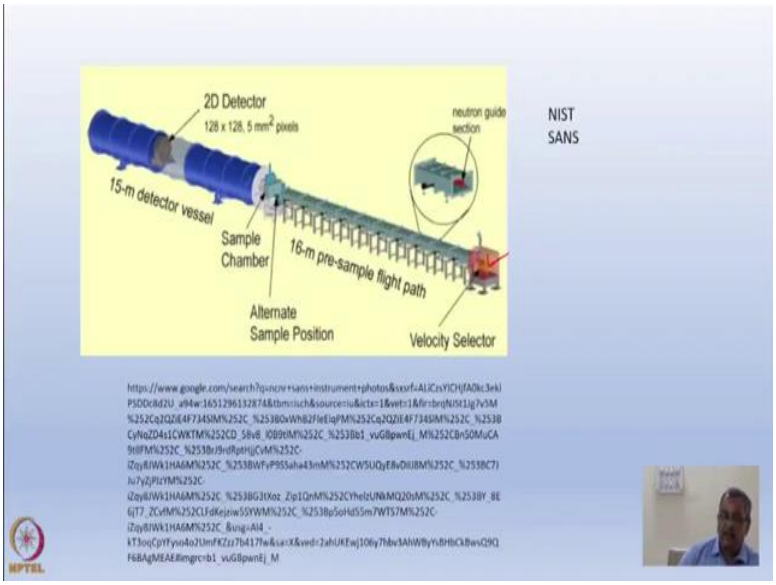

I have just shown you one international example, the NIST SANS detector. It has the same principle but you have large number of neutrons falling on the sample. You can see the pre sample flight path is 16 meters, you have got a detector vessel which is further 15 meters down in 30 meter SANS and here also you have a velocity selector that gives you the required or desired range of lambda for your experiments. With this stop and I will continue with this in the next lecture.