Neutron Scattering for Condensed Matter Studies
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Week 9: Lecture 23B

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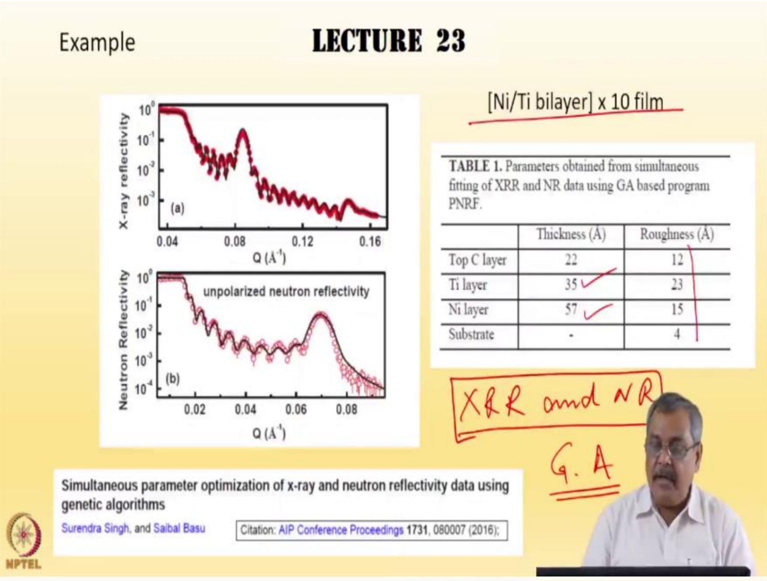

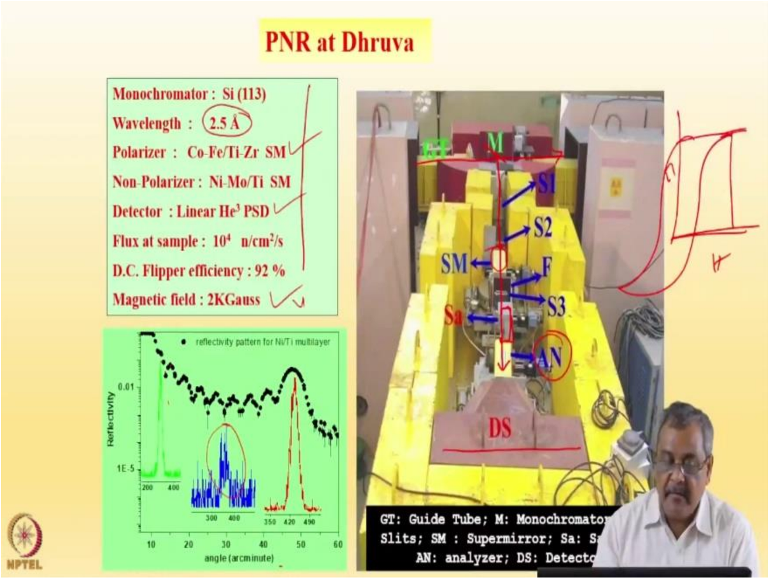

In the last lecture, I stopped with an example of simultaneous minimization of χ^2 for a Ni/Ti periodic multilayer film and I justified the use of genetic algorithm for χ^2 minimization for the data. I will now take you for a tour through the various instruments available for such (neutron reflectometry) experiments.

First, let me show you the instrument that we have at Dhruva in Trombay, India. This is the guide tube, where the neutrons are coming flowing down the guide tube, if I may say so, in this direction. I had shown you earlier that there are two small angle instruments on this guide, at this gap we have what is known as our PNR or polarized neutron reflectometer. The whole instrument is kept inside a shielding pit. Because as I told you earlier, that reflected intensity falls as $1/Q^4$ with angle which means it falls very rapidly and we need to minimize the background at the detector to the extent possible.

Here is the closer look of the PNR instrument at Dhruva. The guide neutrons are flowing down in this direction, we have got a silicon monochromator sitting in the guide (neutron) path which diverts the beam in this direction. S1 and S2 are two slits, which collimate the beam to few arc seconds because we need to have a narrow beam for reflectivity experiment. Then, there is a super mirror polarizer here. I will shortly narrate what is a (neutron) supermirror and how it works as a supermirror polarizer. This instrument is meant for vertical samples. There is a magnetic field here, these are the pole shoes for the permanent magnet and the sample is vertical. At the end, there is a position sensitive detector. We reach the detector after a (spin) analyzer. At the moment we do not analyze the beam reflected by the sample and this position sensitive detector arrest the reflected beam on it channel wise.

Experimental data look somewhat like this; I have just shown you each experimental point at *Q* space or it is as per angle, but each point is actually an integration over such an intensity profile on the position sensitive detector and so, each point is an integration over such values of reflected intensity after subtracting the background.

We also take the background, channel-wise on the PSD after covering the sample with a Cd strip. Cd is a strong neutron absorber so you do not have anything to come from the sample on the detector and then we do the background measurements. That tells me how many stray neutrons are there in the guide hall. It is much smaller than what we have in the reactor hall.

I have given the specifications here. At the moment, there is slight change in the wavelength and we have gone up in wavelength to 3 Å neutrons for the incident beam. We have Co-Fe/Ti-Zr polarizer supermirror. We also have a non-polarizing supermirror. The detector used is a linear He^3 detector. We have got a DC neutron beam flipper with an efficiency of 92% and we have a 2 K Gauss permanent magnetic field on the sample to magnetize it. Usually, thin films reach saturation magnetization at low fields (compared to bulks) in the hysteresis loops and generally for obtaining magnetic moment density we should saturate the thin film so that the magnetization align in one direction, and we can measure the magnetic moment density from a magnetized sample.

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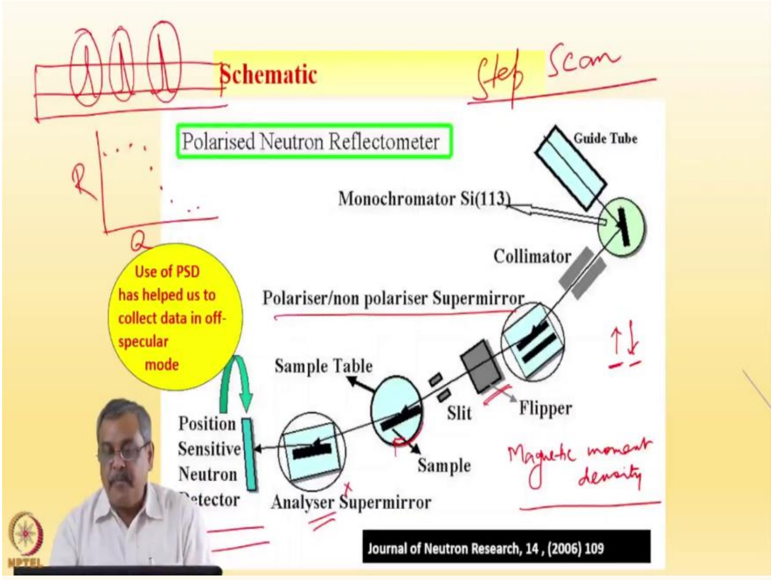

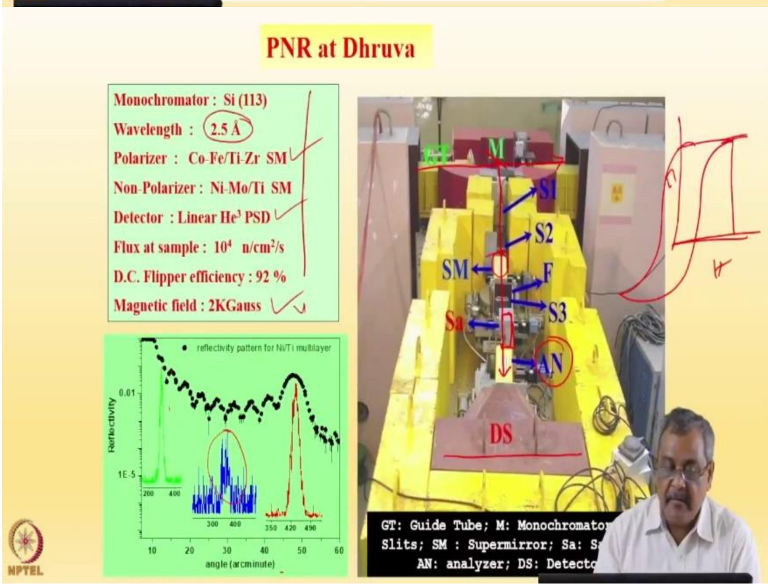

Now, this is a schematic of the same instrument. Si monochromator gives out a monochromatic beam reflected from the guide (beam). Then there is a polarizer/non-polarizer supermirror. Polarizer supermirror can polarize the (neutron) beam. Then there is a DC flipper. I can get one particular polarization after reflection from the super mirror and I can flip it by 180° using the flipper. So, on the sample, I can have either up or down polarization neutrons, which is either parallel or anti parallel to the magnetization in the sample. This is (the location for) analyzer supermirror. We generally do not use it because of the intensity restriction. Our source in Dhruva is a low intensity source. So, what we get from such experiments is magnetic moment density. If I do polarization analysis, after the sample, then we can get the magnetic structure (alignment directions). I will give an example later.

The position sensitive detector allows me to look at the specular reflectivity, it can also help me to collect data in off-specular mode. Have a look at the structure of the peaks that I showed you here, and then I can also get off specular intensity (on the detector). That is the advantage of using the position sensitive detector. But otherwise, this instrument is a step scan instrument. It is not like what I showed you earlier for the diffraction instruments that collect the data in one shot, here the sample is rotated around the beam in θ direction and if this is the position sensitive detector normal to the beam, the reflected intensity moves channel-wise on the detector and each integrated peak is one intensity (point) in the reflectivity profile. So, it is a step scan instrument, but the use of PSD helps us to collect off-specular data also; I will use some examples later.

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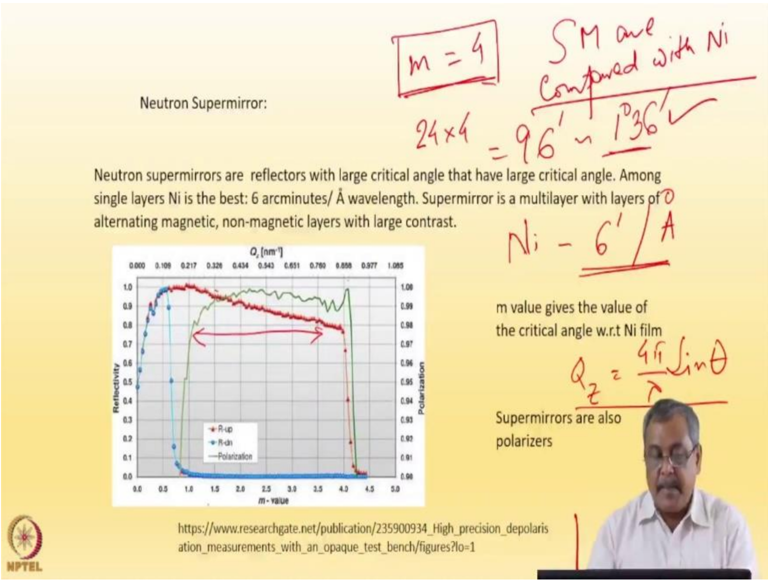

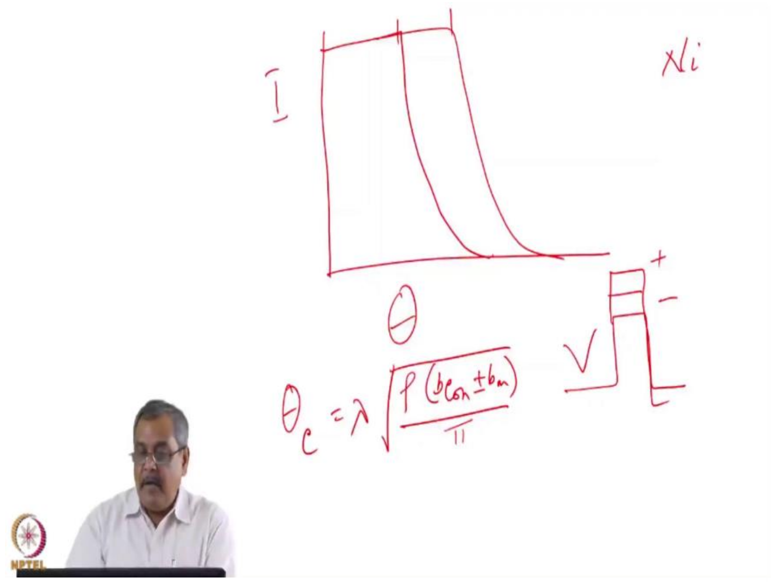
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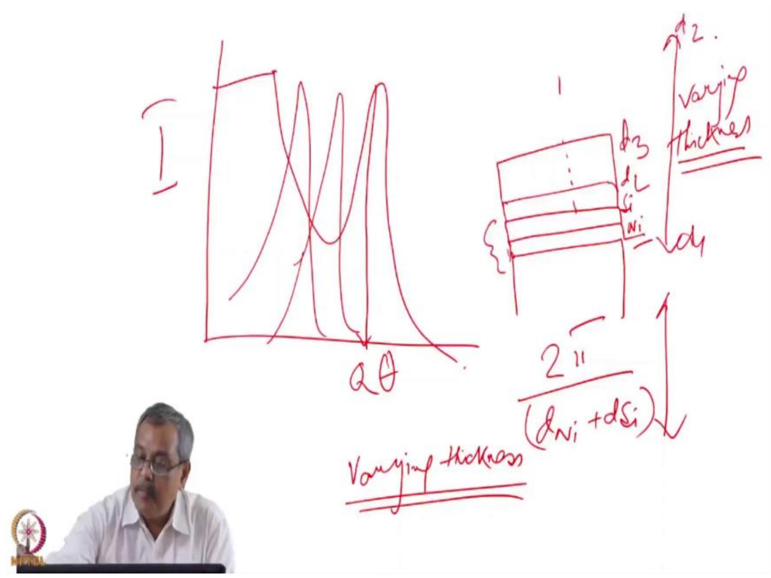
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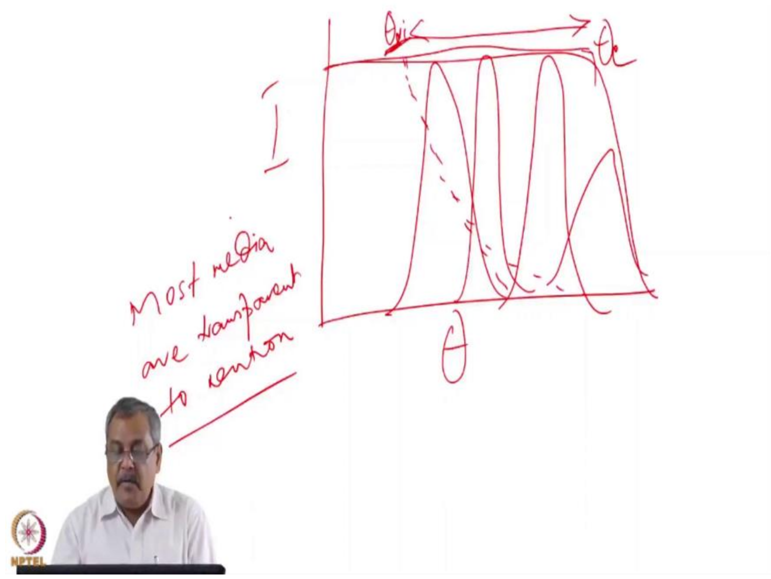
Varying thickness


I
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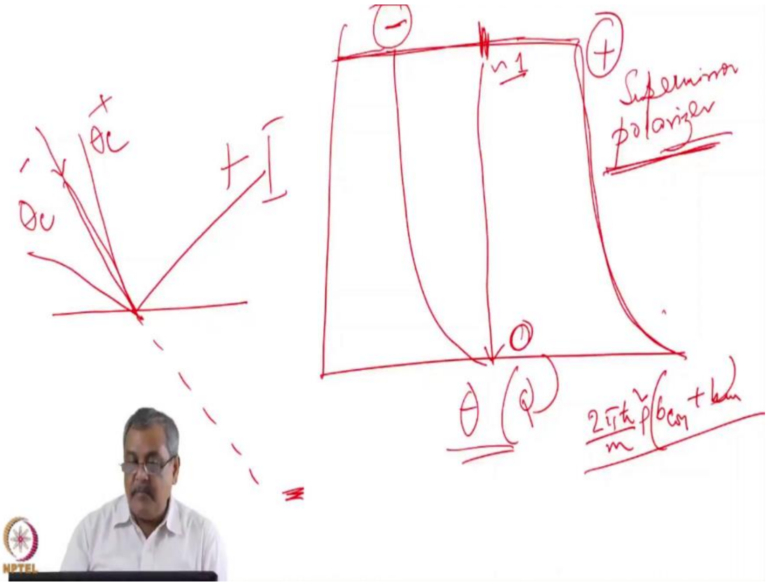
Most media are transparent to neutron



Now, I will explain to you what is a neutron supermirror?

Let us consider a magnetized nickel film. As I told you earlier that for positive and negative direction of neutron beam, it sees different potentials and hence different critical angles which are given by, $\theta_c = \lambda\sqrt{\frac{\rho(b_{coh} \pm b_m)}{\pi}}$. So, I have two critical for a single magnetic film.

Now, let us take the case of a magnetic/non-magnetic periodic bilayer, say Ni/Si periodic bilayer. Then apart from the critical angle for nickel, I will also have a Bragg peak depending on the periodicity, which is observed at a Q value commensurate with the d-spacing and $\frac{2\pi}{(d_{Ni}+d_{Si})}$ will tell us where this Bragg peak will be present.

This is a (case of a) periodic bilayer with one periodicity. Now, the interesting thing is that if I can keep changing the periodicity means while depositing this multilayer film, I keep changing the thickness of nickel and silicon layers so that bilayer thickness keeps changing in the multilayer film then in such a case the Q value or the angle for the Bragg peak keeps shifting. And if I keep on going to thicker and thicker film, this Bragg peak will go to lower and lower theta.

So, for a multilayer with varying thickness of the periodic bilayer, I have got a number of Bragg peaks and as I keep on increasing the thickness from a certain value to some other value both for nickel and silicon the Bragg peak position keeps varying. Ultimately, due to the overlapping Bragg peaks, I get an extended critical angle in the reflected intensity vs theta plot and that is why it is called a supermirror. So, the total external reflection is effectively extended to larger critical angles by using a periodic bilayer with varying thickness.

This is possible because most media are transparent to neutrons. So, neutrons can penetrate deeply and I can get an (intensity) structure like this. This is about a neutron supermirror. Now, the next step is about a neutron supermirror consisting of magnetic and non-magnetic materials. I can magnetize the mirror and then in the intensity vs theta profile critical angle will be different for up and down neutrons. Say it is higher for up neutrons while smaller for down neutrons because here the up neutrons see a larger potential.

Now, if I use an angle to reflect the neutron beam, which is between θ_c^+ and θ_c^- then the down neutron does not get reflected as reflectivity is 0 for this (polarization). While for the up neutron the reflectivity almost one here. That means, by reflection of unpolarized beam at an angle between these two (angles), it will give me a reflected beam with a particular polarization while the transmitted beam will have other polarization. So, this is not only reflecting the neutron beam, but it is also polarizing the neutron beam and hence this is called a supermirror polarizer.

I started describing to you how neutron reflection takes place and now, I have shown you a device based on the principle of reflection, which can polarize a neutron beam. These supermirror polarizers are commercially available and are used heavily for various neutron instrumentation.

Let me just show you the reflectivity profile obtained from this Fe/Si supermirror with *m* = 4. Now, what is *m* here? Usually, supermirrors are compared with Ni critical angle and Ni has a critical angle 6'/Å. What does it mean?

As I showed you that critical angle is directly proportional to the wavelength of the incident beam. Ni has a critical angle 6'/Å. This means, if I have 1 Å neutron the critical angle will be 6 arcminutes and if I have a 4 Å neutron, the critical angle will be 24'.

Now, the supermirror, discussed here, has *m* = 4, that means, for all wavelengths this has a critical angle of 4 times that of nickel. That means, if I use a 4 Å neutron on this super mirror, the critical angle will be 24×4 = 96'= 1° 36'.

Here is the reflectivity curve with respect to m value; you can see that for *m* = 4, the reflected intensity for one polarization is almost 80% not exactly 100%. That is good enough (for instruments). But, because there are down neutrons and their reflectivity has fallen over here, any angle in this range for reflecting neutrons will give a polarized beam. Polarization efficiencies are also plotted here and it is almost 99%.

I will get a 99% polarized neutron beam of up neutrons if I use a reflection angle, which has a Q_z value between these two, $Q_z = \frac{4\pi \sin\theta}{\lambda}$ and I can calculate the theta for the (desired polarization). As I showed you, this can go up to 1.5 degrees and that is why it is called a supermirror. We use neutron super mirrors heavily for all our instruments.

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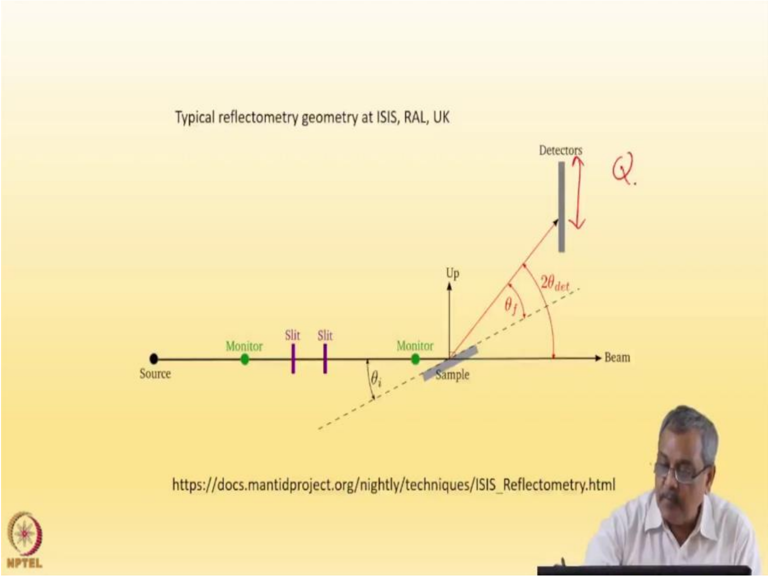

Now, I will show you the general reflectometer designs at ISIS (spallation) neutron source. You have a (pulsed) source and the slits collimate the beam. There is a (neutron) monitor and again there is a position sensitive detector arresting the beam. Because this is a polychromatic incident beam, so, in a single setting, I can get a large range of Q. I would like to mention to you, that the reflectivity profile if I measure it as a function of time-of-flight (TOF), say for a single layer film, then it will look somewhat like this. Kiessing oscillations are here at smaller values of TOF because large TOF means larger wavelength and hence smaller Q. This is like the mirror image of the reflectivity profile obtained from a reactor source.