Indian Institute of Technology
Kanpur

NP- TEL
National Programme
on
Technology Enhanced Learning

Course Title
Advanced Characterization Techniques

Lecture – 33

by…
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Hello everyone, welcome to the last class on Advanced Characterization Techniques. So in the course of this series you have gone through the basics of X-ray diffraction and scattering.

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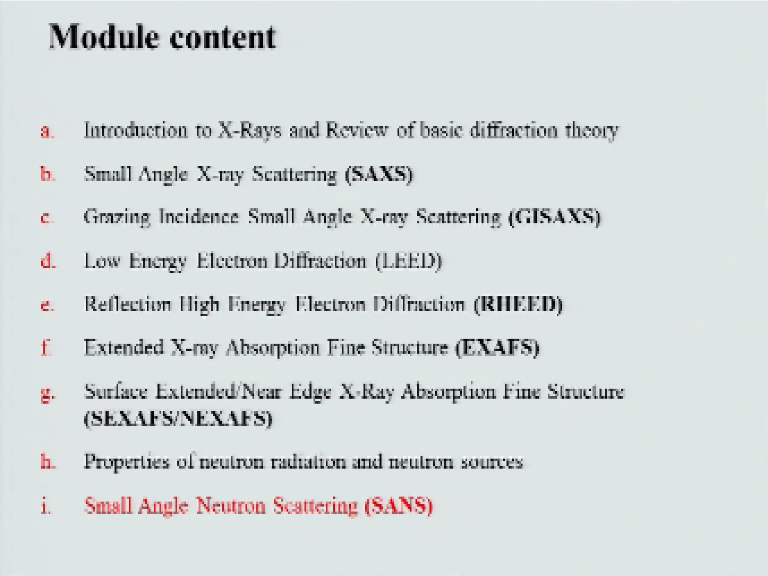

Module content

a. Introduction to X-Rays and Review of basic diffraction theory
b. Small Angle X-ray Scattering **(SAXS)**
c. Grazing Incidence Small Angle X-ray Scattering **(GISAXS)**
d. Low Energy Electron Diffraction (LEED)
e. Reflection High Energy Electron Diffraction **(RHEED)**
f. Extended X-ray Absorption Fine Structure **(EXAFS)**
g. Surface Extended/Near Edge X-Ray Absorption Fine Structure **(SEXAFS/NEXAFS)**
h. Properties of neutron radiation and neutron sources
i. Small Angle Neutron Scattering **(SANS)**

We have studied small angle X-ray scattering as well as gracing incidence small angle X-ray scattering. Then we also studied electron diffraction and finally we studied some spectroscopic technique using X-rays. In the last class we had studied a few things about neutrons and in the final lecture today we are going to extend what all we studied about neutrons to a technique which we had studied earlier, that is small angle scattering.

In the earlier classes, we had gone through small angle X-ray scattering and we had also realized what the advantages of using neutrons are and therefore in the present class, we are going to extend the same geometry or the same concept of small angle scattering to neutron scattering.

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Small Angle Neutron Scattering (SANS)

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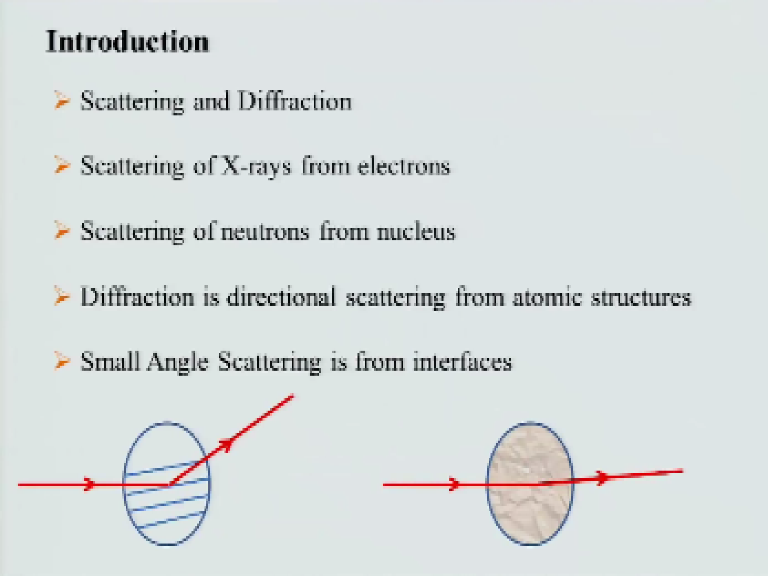

Introduction

- Scattering and Diffraction
- Scattering of X-rays from electrons
- Scattering of neutrons from nucleus
- Diffraction is directional scattering from atomic structures
- Small Angle Scattering is from interfaces

So as we had discussed earlier that interaction of photons with matter can lead to scattering or as well as diffraction. Now scattering of X-rays is essentially from electrons and we had seen earlier in the last class that scattering of neutrons is from the nucleus. We had also seen now this slide is almost borrowed from what I had shown you during small angle X-ray scattering that this is what is your diffraction where your incident X-rays or for that matter neutrons interact with the lattice planes which occurs at a very small level of the order of the wavelength of the incident X-rays or neutron while when we talk about scattering, scattering occurs at a higher lens scale and small angle scattering most of the times is from interfaces.

Now this is what we had seen earlier also ant the same concept remains valid even for neutron scattering. However I hope you remember that in the last class we had seen that depending on the energy, we can get neutrons of very different wavelengths and therefore we can see a variety of objects theoretically, I hope you appreciate that we can see a lot of wide range of objects using neutron diffraction compared to X-ray diffraction.

This is precisely because we have neutrons in a wide energy range which in turn gives us a wide wavelength regime. Having said that generally most of the neutron scattering or small angle neutron scattering also has the same size regime as that of small angle X-ray scattering.

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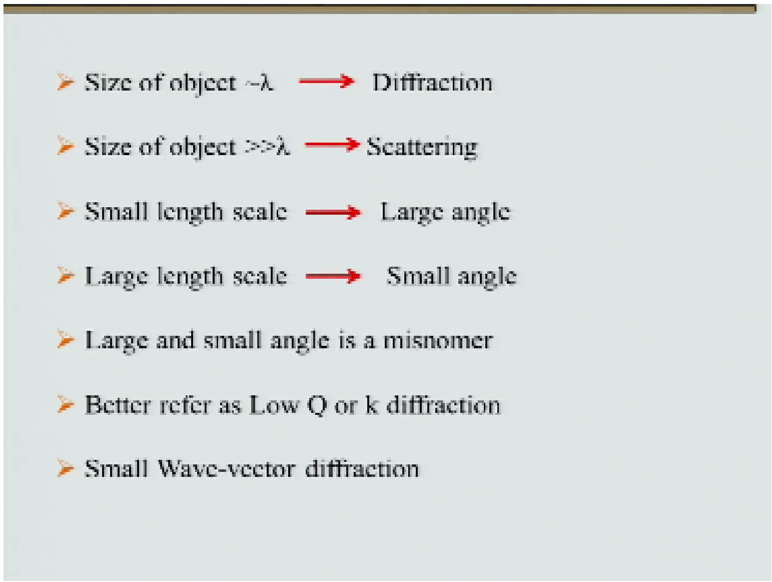

- Size of object ~λ ⟶ Diffraction
- Size of object >>λ ⟶ Scattering
- Small length scale ⟶ Large angle
- Large length scale ⟶ Small angle
- Large and small angle is a misnomer
- Better refer as Low Q or k diffraction
- Small Wave-vector diffraction

As we had seen earlier that at the very small lens scale we have very large angle and this constitutes the normal diffraction. As we have for X-ray we have similar condition for neutron diffraction also. However at large lens scales we get smaller angle. However I am now going to say something which may kind of surprise you. We are talking about small angle neutron scattering and in the last class or a couple of classes before we talked about small angle X-ray scattering.

However this term “small angle” is actually a miss moment because if at all we are having a change in wavelength, the angle at which we are getting scattering is going to change and this is particularly valid for neutrons where we can get a wide variety of wavelengths. Therefore the correct term should be actually low Q or low K diffraction for what we term as small angle scattering and normal wide angle scattering can be referred to as I q diffraction or scattering.

So this is just the kind of terminology that has kind of stuck with the technique but I hope you appreciate that in small angle scattering, we are mostly dealing with what is happening at very low Q values while at higher Q values or K values, we deal with wide angle X-ray scattering which is a conventional intensity vs 2 theta plot.

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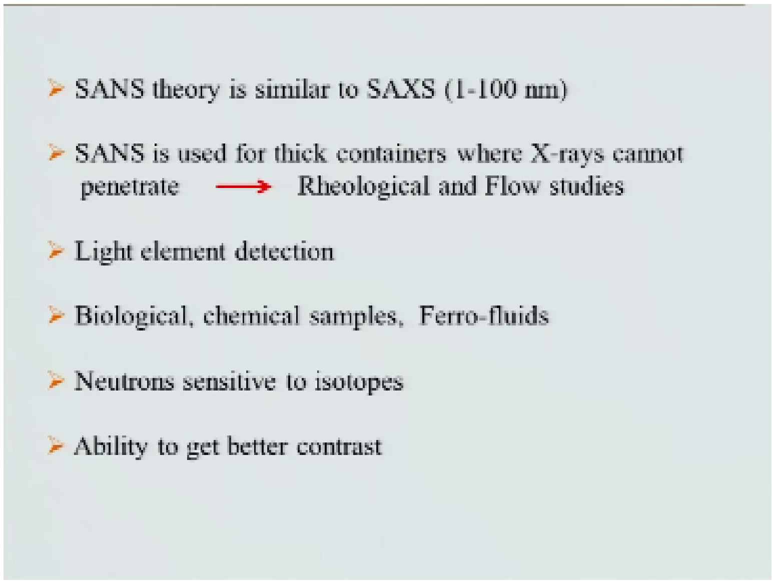

- SANS theory is similar to SAXS (1-100 nm)
- SANS is used for thick containers where X-rays cannot penetrate ⟶ Rheological and Flow studies
- Light element detection
- Biological, chemical samples, Ferro-fluids
- Neutrons sensitive to isotopes
- Ability to get better contrast

So let us move over from there and I would just like to remind you again that the small angle neutron scattering theory is exactly similar to that of small angle X-ray scattering theory and for most practical applications we are having the same size regime as we have for small angle X-ray scattering. But the big advantage that small angle neutron scattering offers because of the use of neutrons is that we can penetrate deeper within containers which are made of metallic materials and containing various soft matter like your polymers and what all things are happening to it as a function of flow or applied stress and therefore we can study the realogical as well as flow behaviour of the soft matter using small angle neutron scattering.

At the same time a big advantage that neutrons offer over X-rays is their ability to detect light elements. So particularly this is very, very important when it comes to biological materials and therefore small angle neutron scattering has extensive application in biological, chemical as well as Ferro-fluid materials. Here again for Ferro-fluids you know that neutrons are sensitive to the magnetic moment and therefore can give us information about the magnetic structure. So all this information is obtained using small angle neutron scattering which is generally not obtained in small angle X-ray scattering.

One more important thing that we had stressed in the last class was about the ability of neutrons to separate between isotopes. Now this ability of neutrons can be used to get better contrast. We will talk about this in the couple of slides but I hope you appreciate that the advantages of neutrons over X-rays can be exploited in this technique to get relevant information from over experiment.

So just to revise and put things in perspective, I hope you remember that we had this sample on which whether neutrons or electrons are getting kind of they are incident.

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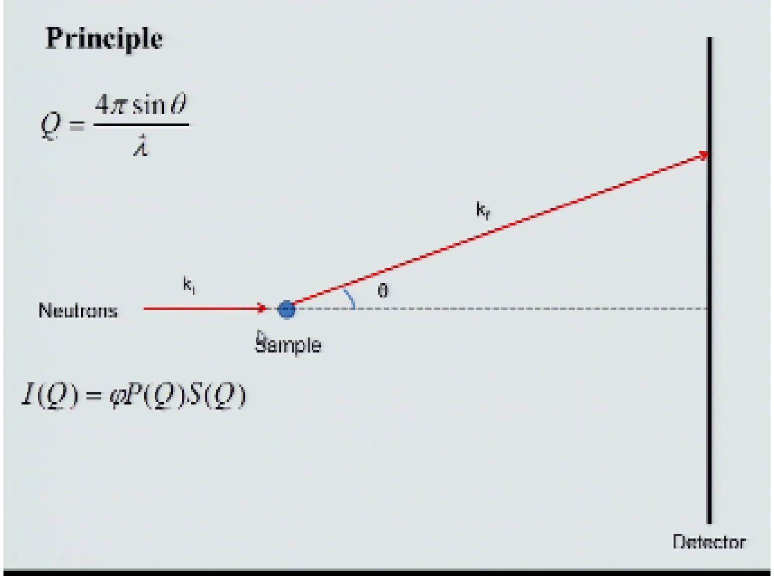

Principle

$$Q = \frac{4\pi \sin\theta}{\lambda}$$

$$I(Q) = \varphi P(Q) S(Q)$$

And we see that it scatters the incident beam and you see if KI is your incident beam wave vector and KF is your scattered wave vector. We have defined our scattered wave vector Q as $q = 4 \pi \sin \theta / \lambda$ right and the overall intensity that we got on the screen or our detector is nothing but φ, is nothing but the distribution of particles and PQ is nothing but the form factor. If you remember like this contribution comes from all the atoms that constitute our particle and the SQ is nothing but the structure factor and the structure factor comes because of the arrangement of these particles. So this theory is exactly similar to that of small angle X-ray scattering.

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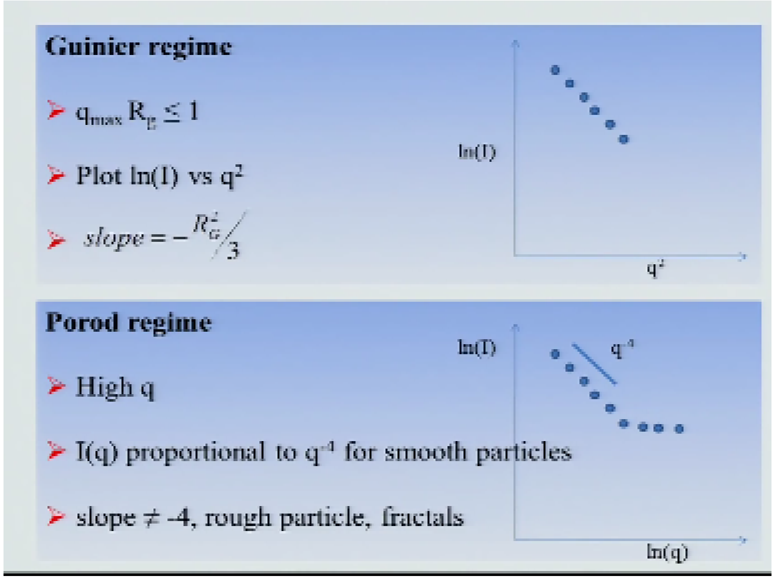

Guinier regime

- $q_{max} R_g \le 1$
- Plot ln(I) vs q^2
- $slope = -R_G^2/3$

Porod regime

- High q
- I(q) proportional to q^{-4} for smooth particles
- slope ≠ -4, rough particle, fractals

So again if you remember we had two regimes, we had the Guinier regime and the porod regime. The Guinier regime was valid at Q max rg<=1, this product Q max is nothing but your wave vector into rg, where rg is the radius of gyration which was for a spherical sphere as √ π / 3 x the radius of the sphere. So it gives you the slope of this line, -rg / 3 if you plot lot of intensity vs Q squared. This is our quinier regime.

However at high Q, now mind you this higher Q is not the high Q regime which corresponds to the wide angle scattering but at high Q regime we see that lot of i is proportional to Q^{-4} and we see that from this information about what all slope we get, we can get information about the shape of the particle. This is what we did in small angle X-ray scattering. Now the same concept can be borrowed and applied directly to small angle neutron scattering.

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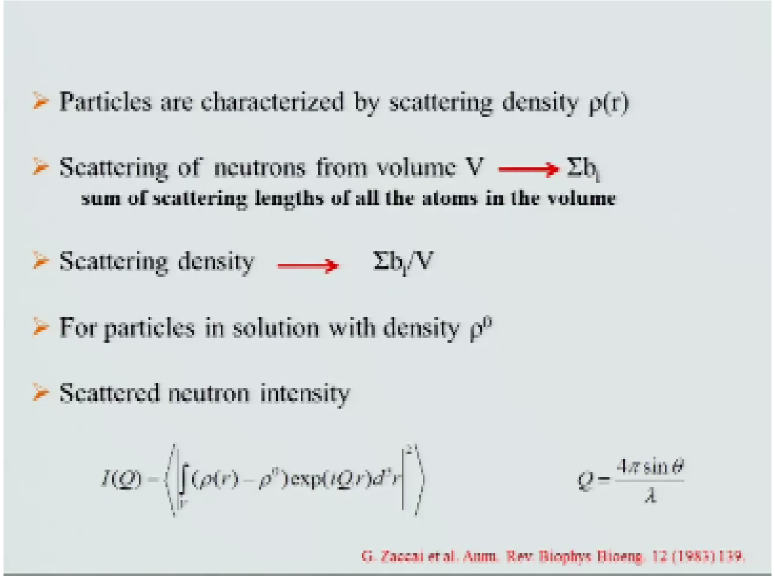

- Particles are characterized by scattering density ρ(r)
- Scattering of neutrons from volume V ⟶ Σb_i
  sum of scattering lengths of all the atoms in the volume
- Scattering density ⟶ $\Sigma b_i/V$
- For particles in solution with density ρ^0
- Scattered neutron intensity

$$I(Q) = \left\langle \left| \int_V (\rho(r) - \rho^0) \exp(iQr) d^3r \right|^2 \right\rangle \qquad Q = \frac{4\pi \sin\theta}{\lambda}$$

G. Zaccai et al. Ann. Rev Biophys Bioeng. 12 (1983) 139.

Having said that, let us try to see one particular thing or other one particular concept that is not seen or not quite prominent in X-rays and that is the ability to separate between the isotopes. But before we go there, let us kind of rebuild and revise how actually scattering occurs. So all of us are aware that any particles if we take the particles are characterized by a scattering density which is ρ far. No this ρ far or the density of scattering, depends on what all atoms are there.

So the particle which comprises of atoms. So this scattering tendency of the particle is nothing but the summation of scattering tendency or scattering ability of all these atoms. So therefore you see that your scattering density ρ far is nothing but summation di. Now it has to be averaged over the volume of the particle and therefore the scattering density is nothing but summation pi over ro. Now these particles are not standing alone. Instead most of the cases these particles are there in a solution.

So we also have to assume like what is the density of the solvent under consideration. So let it be say ρ0. So if at all our neutrons were to pass through a solution comprising of a solvent of scattering density ρ 0 and particle with scattering density ρ r where r is a function of the location of the particle. The intensity that we get can be given by this formula. Here mind you, look what we are having over here. So this is the difference in the scattering density of the particle and the solvent and this is what gives us the contrast. This is the resultant scattering density.

Here again the value of Q which we had seen the wave vector is nothing but 4π sin θ/ λ right, and you see this exponential term and you see it is proportional to d ^{3} r and the whole thing squared. So this is again very similar to our structure factor and this also accounts for the size part.

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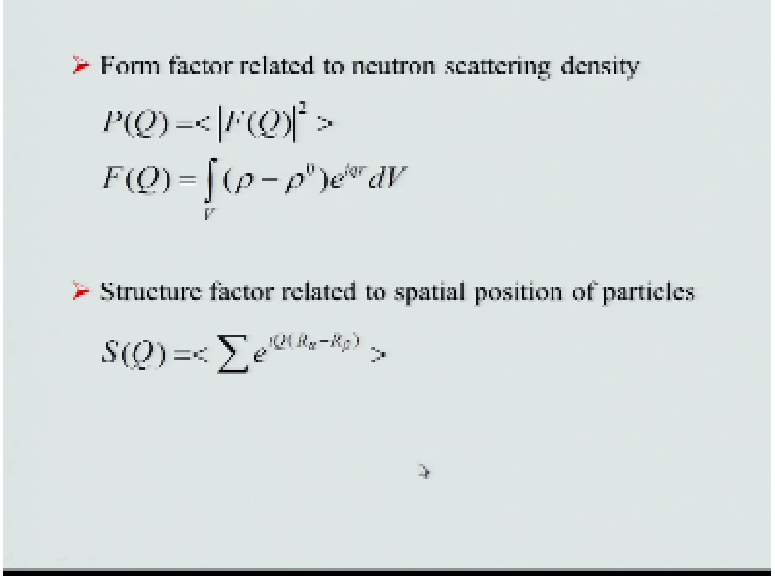

- Form factor related to neutron scattering density

$$P(Q) = <|F(Q)|^2>$$

$$F(Q) = \int_V (\rho - \rho^0) e^{iqr} dV$$

- Structure factor related to spatial position of particles

$$S(Q) = <\sum e^{iQ(R_\alpha - R_\beta)}>$$

So moving on, the form factor is related as we had seen in case of small angle X-ray scattering also to the scattering density. Therefore our P of Q is nothing but F of Q ^{2}. Now this is very similar to what we got in case of diffraction. The intensity that we get is directly proportional to the square of the amplitude. The same over here and this F of Q is nothing but the difference in the scattering density of the particle and the solution or the solvent. So this is the F of Q term.

Similarly if you remember the structure factor which we had calculated in small angle X-ray scattering remains the same when it comes to small angle small angle neutron scattering and it has to do with the spatial position of the particles. So see, we have a form factor which depends on the size and shape of the particle while the structure factor decides the or rather is dependent on the spatial position of the particles and here you can see that r alpha and r beta are nothing but the spatial coordinates of the two particles.

So there is a big summation sign and everything is averaged over the particle under consideration. So now let us move ahead.

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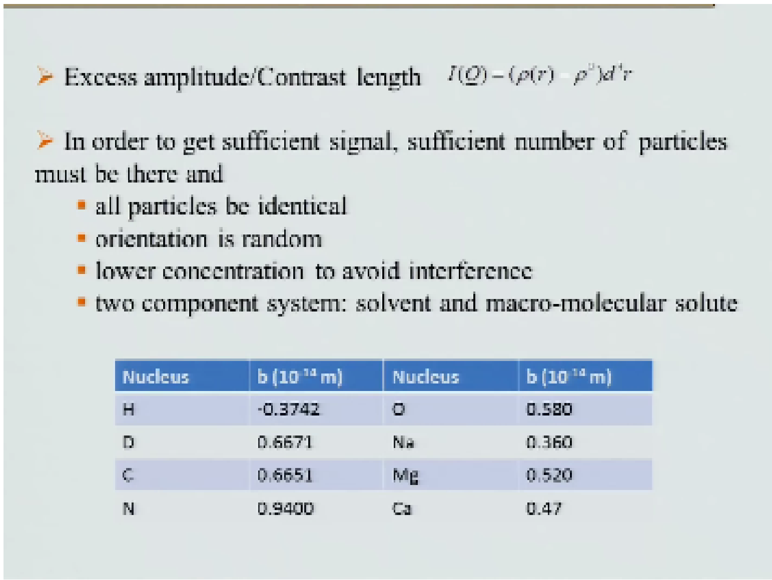

- Excess amplitude/Contrast length $I(Q) = (\rho(r) - \rho^{0})d^{3}r$
- In order to get sufficient signal, sufficient number of particles must be there and
  - all particles be identical
  - orientation is random
  - lower concentration to avoid interference
  - two component system: solvent and macro-molecular solute

Nucleus	b (10^{-14} m)	Nucleus	b (10^{-14} m)
H	-0.3742	O	0.580
D	0.6671	Na	0.360
C	0.6651	Mg	0.520
N	0.9400	Ca	0.47

So you see that we do have a excess amplitude or contrast lens. So this is very important. So this is what we want to maximize. In small angle X-ray scattering, in order to get a estimate of the shape as well as size of the particle in the solution, we need to maximize the contrast and this is what is the contrast. So I would like to remind you that this is your scattering ability was nothing but summation Vi over V. Isn't it, ρ vi = v x dq and r.

So what we get over here is actually a contrast length and this is the term, the term on your right hand side which you want to maximize. Now in order to get sufficient signal, sufficient number of particles must be there. If the particle size is very small, you should have sufficient number of these particles, very small particles in your solution so that you get good enough signal and from this signal you can decipher the size, shape as well as distribution of these particles in the solvent and in order to do this, you have to make certain approximations and we assume that all particles should be identical.

Their orientation is random. We have lower concentration so that there is no interference of scattering caused with one particle with that of caused by the other particle. Another important thing is we ideally like a two component system. So you have a solvent and your particle which may be a macro molecule. You do not like any interaction taking between these two and having the presence of third phase. So in this case, the situation is very clean and you can do very easy analysis of your sample and get information about the size, shape as well distribution of the particle under consideration.

So before I proceed, you see here the contrast term. The contrast length that I have shown over here. We have just noted down for different nucleus. Mind you, this contrast term is for nucleus. I had mentioned that it is for different atoms but mind you, our neutrons we should remember that neutrons do not interact with the means they do interact with the atom but they do not interact with the electron cloud of the atom but with the nucleus of the atom and you see for light elements, we do have different values and very good values, light as well as heavy elements.

Another important thing is, look what we are having here, Hydrogen and Deuterium and as I had mentioned that they have different scattering cross section. Here again we see that, they have different scattering length. Not only the magnitude is different but also their sign is different and this is where the biggest advantage of neutron diffraction lies. You know that most of the biological systems comprises of Hydrogen.

So many a times we can play and replace Hydrogen with Deuterium and try to get or improve the contrast. Now this is something that is very difficult to do using small angle or rather it should be not possible, it is not possible in small angle X-ray scattering.

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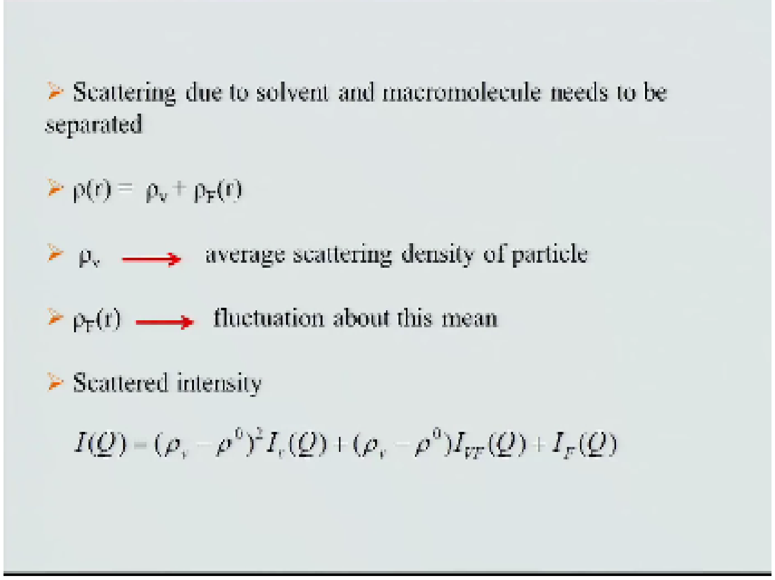

- Scattering due to solvent and macromolecule needs to be separated
- $\rho(r) = \rho_v + \rho_F(r)$
- $\rho_v \longrightarrow$ average scattering density of particle
- $\rho_F(r) \longrightarrow$ fluctuation about this mean
- Scattered intensity

$$I(Q) = (\rho_v - \rho^0)^2 I_v(Q) + (\rho_v - \rho^0) I_{VF}(Q) + I_F(Q)$$

So again let us go back and try to work on this, macro molecules on one stuff. So scattering due to solvent and macro molecule needs to be separated. At the end of the day, we are getting signal from both this solvent as well as the macro molecule that we are studying. So the overall scattering is essentially ρ of r is ρ v+ ρ f r, where ρ v is your average scattering density of the particle. Now this comes from the particle. While ρ f of r comes from the fluctuation about this mean. Now this is caused essentially because of the interaction of the particle with the solvent and therefore the scattered intensity, I hope you appreciate can be given as nothing but ρ v which you have over here - ρ 02 iv q + ρ v - ρ 0 I_{vf} Q and + if q term.

So now this fluctuation term also plays a very important role in deciding the intensity and this is where it comes, the overall intensity. So if at all we have to see contrast, we have to change this fluctuation and this will happen, how this will happen? Again this will happen while changing the interaction between the solvent and the macro molecule.

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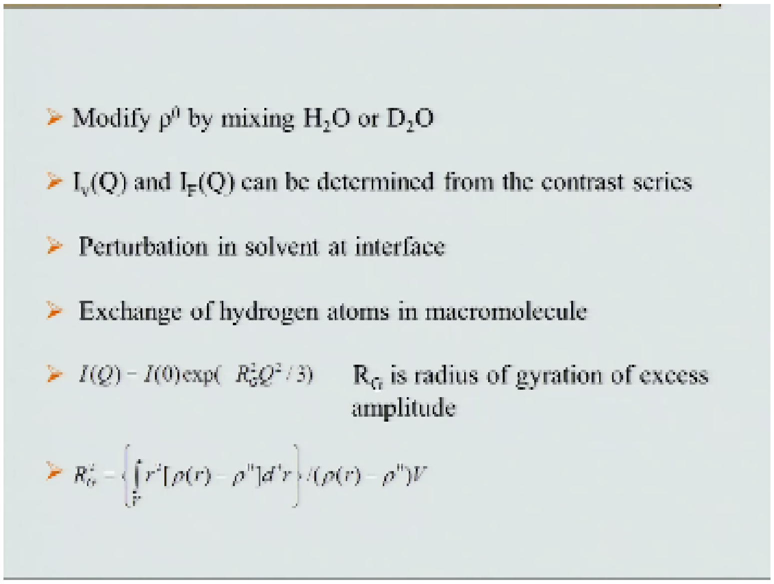

- Modify ρ^0 by mixing H_2O or D_2O
- $I_V(Q)$ and $I_F(Q)$ can be determined from the contrast series
- Perturbation in solvent at interface
- Exchange of hydrogen atoms in macromolecule
- $I(Q) = I(0)\exp(-R_G^2Q^2/3)$ R_G is radius of gyration of excess amplitude
- $R_G^2 = \left\{\int_V r^2[\rho(r) - \rho^0]d^3r\right\}/(\rho(r) - \rho^0)V$

So that is what I mean, that we can modify ρ0 by mixing H2O or D2O, deuterium oxide. So if you have a particular solvent, you can add water in it or you add heavy water that is D2O in it. Now this will ensure that our ivq as well as ifq can be determined from the contrast series. Like in one case, you have only your solvent and macro molecule. In the other case, you add water and in the other case you add D2O and then you compare.

So let us go back and see what is your iv? IV is the scattering density of the intensity due to scattering by the particle while is actually due to the fluctuation part and the fluctuation is going to change as we change the solvent. So it is a bit mathematically involved but I hope you appreciate by changing different solvents, we can get some information. Having said that, there are however two problems.

The first problem is that there is perturbation in the solvent at the interface. Like if you change the solvent, that is almost always some perturbation. So the perturbation level may change. At the same time, what may happen, like if you are changing something with say hydrogen, in a solvent if you are putting water, what may happen is the hydrogen iron which is in your macro molecule may get exchanged with the water? The hydrogen iron in the water that you are adding.

Things will get even more problematic if your hydrogen is replaced with deuterium. But having said that, if still the affirmation technique of what changing the contrast by adding hydrogen in water and D2O is quite valid and as we move ahead, we again come across this intensity vs intensity as function of Q term, which is nothing but I0+, we see here your radius of gyration Q^2 / θ.

Now this is again similar to what we had got using small angle X-ray scattering. The only difference that we have over here is that in this case again your scattering ability or scattering density is a function of you see or your particle as well as but you have to subtract it from that of the solvent. I am invariably using particle and macro molecule as meaning the same, though you should keep in mind that these macro molecules are pretty large and they can elaborate can form a small particle.

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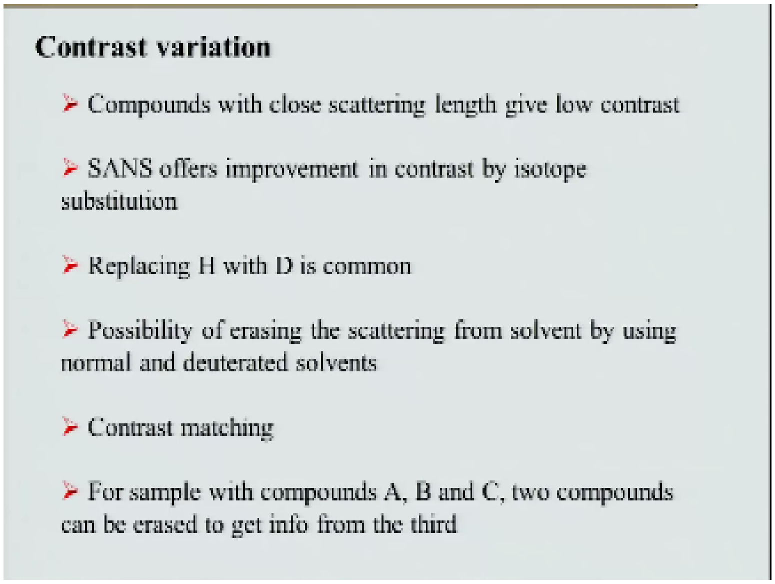

Contrast variation

- Compounds with close scattering length give low contrast
- SANS offers improvement in contrast by isotope substitution
- Replacing H with D is common
- Possibility of erasing the scattering from solvent by using normal and deuterated solvents
- Contrast matching
- For sample with compounds A, B and C, two compounds can be erased to get info from the third

So this is what we talked about was actually what is known as contrast variation. So we know that compounds with close scattering length give low contrast, that is because if the two atoms are having the same two elements are having the same scattering ability you cannot differentiate between the scattering due to the individual element.

However small angle neutrons scattering offers improvement in contrast by isotope substitution so instead of, if you are having hydrogen, why do not you put deuterium? If you want to look for say a particular isotope of nitrogen instead of N14, we substitute it with N15. So this gives us the possibility of erasing the scattering from solvent by using normal and deuterated solvent.

This means you take a solvent which is normal and in one which contains what and in the other case you put heavy water and see try to match how the contrast is changing and if you do it that way, what we can do is, see the whole idea is if we have more than one type of particle or more than one type of macro molecule, what we can do is we can play with the elements and we can glow.

So it is just like if the entire wall is painted in white, you paint the region of interest in black. So there is a lot of contrast and if you want to see the outer part, what you do is, the black region is painted in white and the rest of the region covers into black. So this is what has been shown over here.

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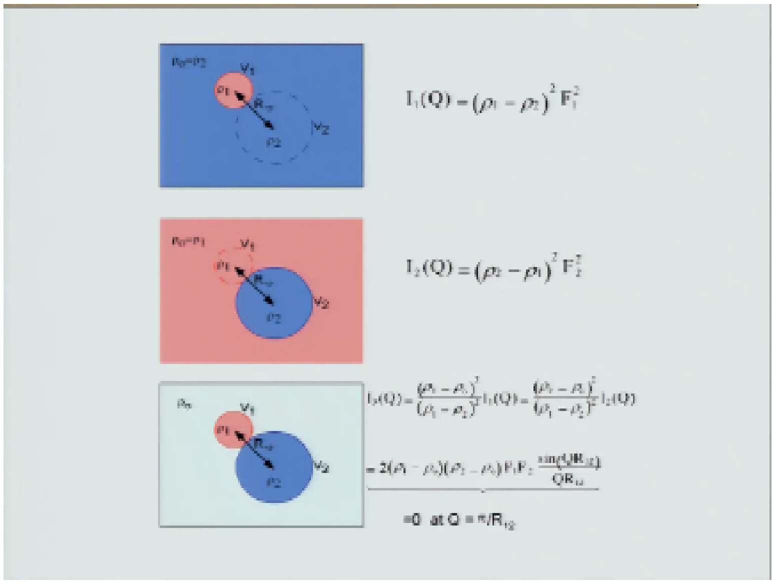

$$I_1(Q)=(\rho_1-\rho_2)^2F_1^2$$

$$I_2(Q)=(\rho_2-\rho_1)^2F_2^2$$

$$I_3(Q)=\frac{(\rho_1-\rho_s)^2}{(\rho_1-\rho_2)^2}I_1(Q)+\frac{(\rho_2-\rho_s)^2}{(\rho_1-\rho_2)^2}I_2(Q)$$

$$+2(\rho_1-\rho_s)(\rho_2-\rho_s)F_1F_2\frac{\sin(QR_{12})}{QR_{12}}$$

$=0$ at $Q=\pi/R_{12}$

So you see here what we do is when you have two entities, you have ρ1 and ρ 2. So these are two particles. So now what we do is, we do contrast match. So in contrast matching what actually happens is we look at ρ 2, the particle 2 and make its contrast similar with that of solvent. So what actually happens, we are not able to differentiate between particle 2 and the solvent. But this gives us the maximum contrast for particle 1.

So we can now very clearly see the particle 1 over here. Now what about particle 2? Well, we play the same trick. Now instead of matching the scattering ability of the solvent with that of particle 2, we match it with that of particle 1. So now particle 1 is invisible and we see the contrast only of particle 2. That is great because using these two techniques, one after the other we have got information not only about particle 1 but also about particle 2, which means we have been able to differentiate between two species which have very very close scattering length or scattering ability.

So you see how by intelligently doing contrast matching, we can get information and separate out two species which are having very close scattering tendency. Let means remind you that this can be done only in small angle neutron scattering because of the sensitivity of neutrons to differentiate between the isotopes of the same elements. So this is one of the biggest advantages of going for small angle neutron scattering.

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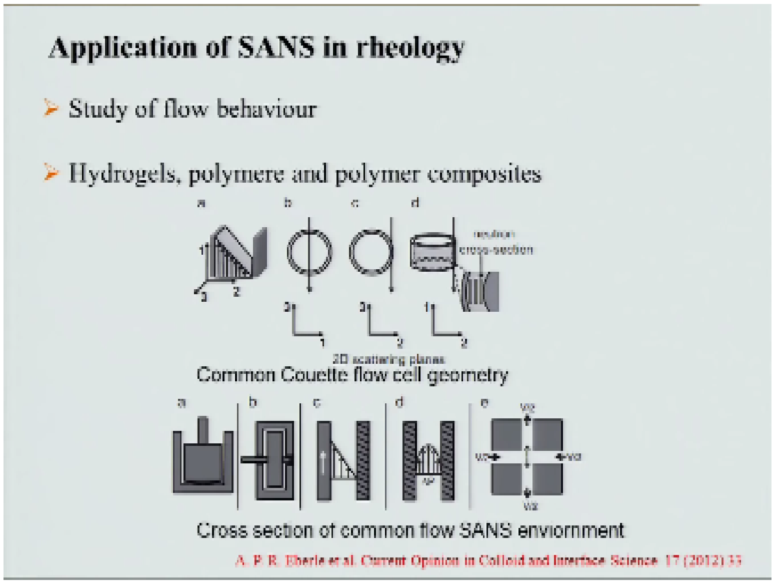

Application of SANS in rheology

- Study of flow behaviour
- Hydrogels, polymere and polymer composites

Common Couette flow cell geometry

Cross section of common flow SANS enviornment

A. P. R. Eberle et al. Current Opinion in Colloid and Interface Science 17 (2012) 33

Just to give you some kind of feel about where all we can use small angle neutron scattering, I have cited a few examples. I will not go back and kind of repeat the same things that we did using small angle X-ray scattering because with small angle neutron scattering, we can do all the things that we can do using small angle X-ray scattering and therefore in the next couple of slides I am going to show you certain things which you can do only with small angle neutron scattering.

So if you remember small angle X-ray scattering or for that matter any X-ray scattering or diffraction experiments are always limited by the penetration depth of X-rays while if you want to study flow behavior of polymeric hydro gels, polymers or polymer composites, polymer panoply composites, well there is no way like we have to apply stresses on it or to make the polymer flow.

Now how do we do that? Well we need containers and that is what has been shown over here. The common quite a flow jell geometry as well as the cross section of common flow which are used in small angle neutron scattering environment. Now keep this thing in mind like all these containers contain our polymer or hydro gel of interest and we are going to apply stresses on it, shear stress to see how their flow, we are going to actually squeezing the material into these Now how do we get signal and how do we see how the structure is evolving as a function of stress or flow?

Remember in order to actually get some information, our probe has to have the ability to pass through these containers and gather important information from the polymer or hydro gel under consideration. No this is absolutely impossible with small angle X-ray scattering. Even if you are using a synchrotron, the synchrotron radiation will not penetrate through these containers and we will not get good signal.

However, small angle neutron scattering due to its high penetration depth can give us some signal and we can really study in c2 the flow behavior, how the rearrangement of the particles, how the particles is taking place or the nano play polymer composite, how their flow behaviour as well as the reorientation can be easily studied using small angle neutron scattering. This is what has been shown over here and you see here.

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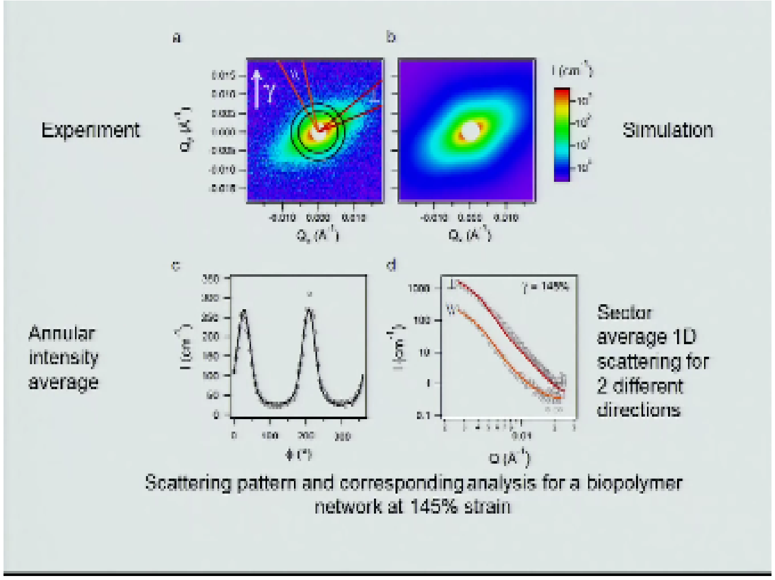

Scattering pattern and corresponding analysis for a biopolymer network at 145% strain

We have a biopolymer that was subjected to a shear strain of 145% and this is how the pattern goes, you see we do see some kind of elongation over here right and what all information we have, this is the experimental part, we can incorporate again go back and kind of simulate it get his value which has been shown over here. Now this is done using again commercially available software or which are available at the various neutron sources in the world.

And are very similar to the software that we use for small angle X-ray scattering but having said that, the input that we are taking one is your structure factor and the other one and most importantly is our form factor, so you see we can get very nice information from the simulations and we can predict how the particles have sheared as a function of strain. Not only that, we can also get the annular intensity average and this is what been plotted over here.

And also this sector average 1d scattering which clearly shows that there is a difference in the scattering tendency of the particles perpendicular and parallel to the direction of straining and here again we see what is very important that there is indeed some reorientation of phases or particles in the bio polymer. So you see like how we can use this and get information during in situ experiments a function of stress or flow behaviour in polymers using small angle neutron scattering.

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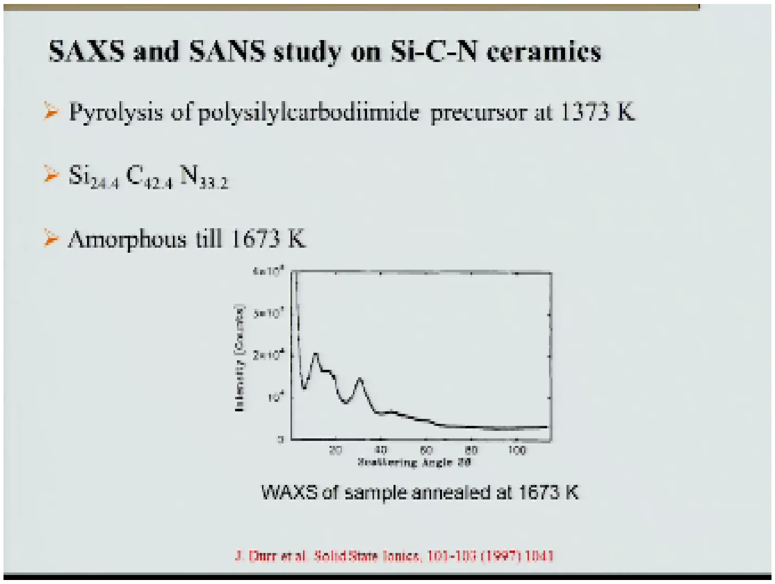

SAXS and SANS study on Si-C-N ceramics

- Pyrolysis of polysilylcarbodiimide precursor at 1373 K
- $Si_{24.4}\,C_{42.4}\,N_{33.2}$
- Amorphous till 1673 K

WAXS of sample annealed at 1673 K

J. Durr et al. Solid State Ionics, 101-103 (1997) 1041

Now as I had mentioned and as we also appreciated in the last class that X-ray is scattering and neutron scattering or for that matter, X-ray and neutron can be used as complimentary sources. So here I am going to present a small example from a very old paper which is on Silicon-Carbon-Nitrogen ceramics and here both small angles X-ray scattering and small angle neutron scattering were used to actually characterize the material.

So these composites were prepared using pyrolysis of polyselil carbodemide precursor at 1373K to obtain silicon 24.4, carbon 42.4, and nitrogen 33.2 ceramic. Now these ceramics were amorphous upto 1673 that is 1400 ^{0} centigrade and here you see after at 1400 ^{0} centigrade, we do get some peaks indicating the crystalline nature. But below this temperature it is absolutely amorphous but this is about wide angle X-ray scattering.

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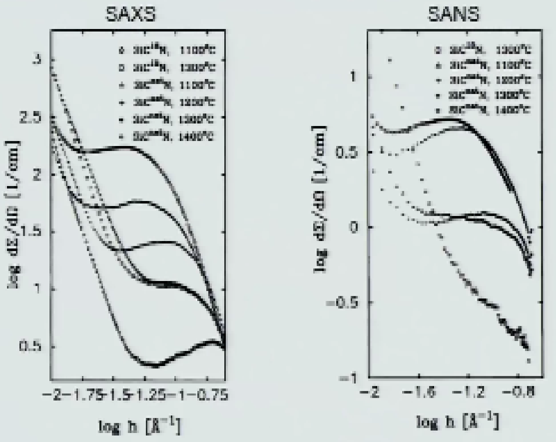

Small Angle Scattering at different annealing temperatures

Now let us go and look at what happens in small angle scattering. So on my left hand side I have the small angle X-ray scattering data and on my right hand side I have my small angle neutron scattering data. Here again you see what we have, you see here 15 end and here we have substituted the nitrogen with its isotope. While here we have the native nitrogen. Now let us compare the 1101 sample with 15 ends and the native end.

Now you see the 1101 comprises of this style over here and you see where it goes. So it goes over here somewhere. So it is here and the 1100 with the native nitrogen is a triangle and you see where it goes. It goes over here so we do have a difference. Theoretically we are not supposed to have this difference isn't it because as we know X-ray scattering is not sensitive to isotopes? However this may be an exception in this ten be attributed to some problem during the processing.

However if you go back and look at the silicon carbide SiC with n native centered at or rather unhealed at 1300 degree centigrade, you see that it goes somewhere over here. So we see that here in this region there is almost a bit of math. So let us assume that there is not much of a difference and here again one point that I would like to emphasize is that, though we talked about the difference between 1100, 15 end and native end, we see their almost behaviour. This region is almost parallel.

So you remember at very low K if you are getting a parallel region, this corresponds to this region well let us not talk about it but it corresponds to the Porod region mind you this is in negative. So now if you go to the small angle neutron scattering, so let us look at 1300 ^{0} centigrade curve. So you have these squares which are over here and here once you go, you see with the native nitrogen.

So you see there is a huge bit of contrast. In fact it is characteristically different. Once you have native nitrogen and here there you have the isotope. So you see how much contrast we get using small angle neutron scattering.

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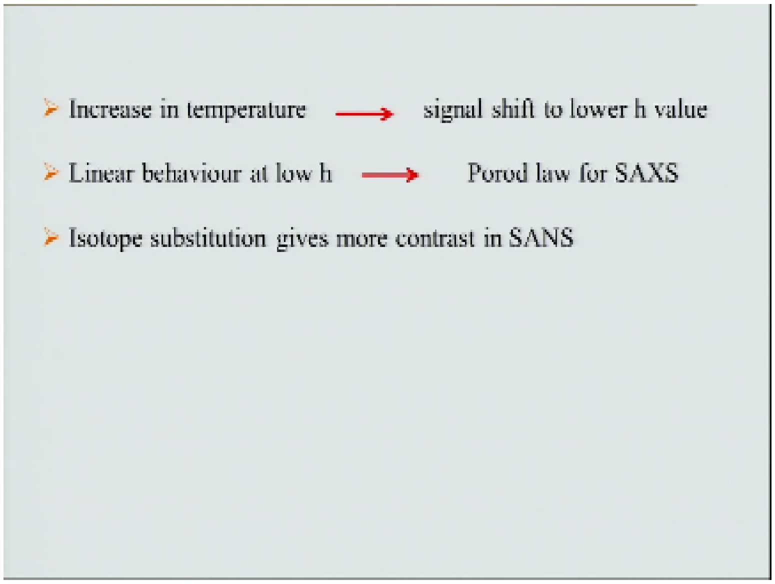

- Increase in temperature → signal shift to lower h value
- Linear behaviour at low h → Porod law for SAXS
- Isotope substitution gives more contrast in SANS

We saw that increase in temperature lead to the signal shift at lower edge values. On this edge is similar to Q or K that we had used. Now even then at low K or low edge we saw a linear behaviour which is similar to the Porod region. Now here you see here we do not see that. So you see the contrast. We are getting different information from these two scans though the range of the Q is same.

So see with small angle X-ray scattering, this is more of a linear, log, log, straight line this is the slope will be about -2 $^{-4}$, so around -4. So actually this indicates the Porod region. So here you see we see a nice Porod region while here no Porod region is valid. So we see depending on the wavelength and the character whether you are using X-ray or neutron, we can get different regime.

So I just wanted to emphasize that we cannot just blindly take the entire theory of small angle X-ray scattering and extend it to neutron scattering and there indeed are differences and here again we saw that isotope substitution gave quite a bit of contrast of characteristic difference in small angle neutron scattering. So this is how our study should aim. Like you start with normal wide angle X-ray diffraction and then you can do small angle X-ray as well as small angle neutron scattering. I am not going to go in details of this particular observation but let us go ahead and look at one of the best advantage of neutrons.

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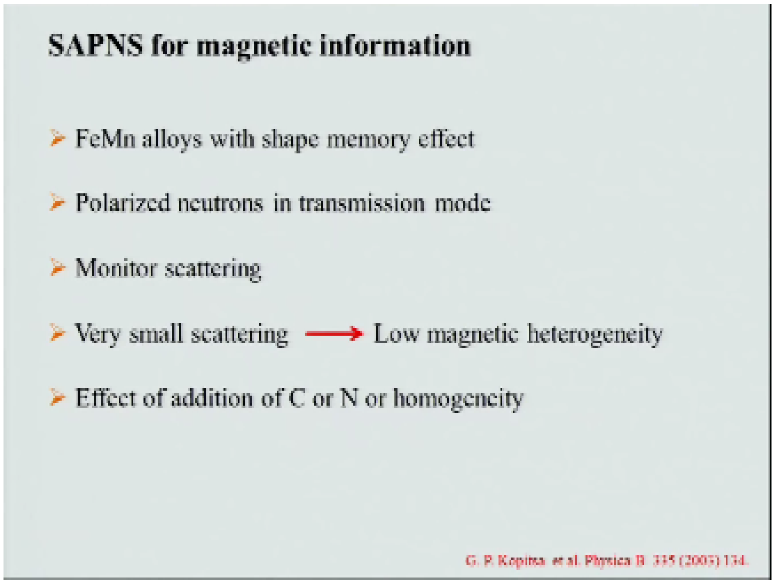

SAPNS for magnetic information

- FeMn alloys with shape memory effect
- Polarized neutrons in transmission mode
- Monitor scattering
- Very small scattering → Low magnetic heterogeneity
- Effect of addition of C or N or homogeneity

G. P. Kopitsa et al. Physica B 335 (2003) 134.

That is its ability to get information about the magnetic structure. So this example from a paper by what they have done is there is iron manganese alloy which has shape number effect. So that they remember once you change, it has a function of stress and go from assonate one phase to another phase, they remember its shape. So we use polarized neutrons in transmission mode and I have not given the details but in this case, these are nice metallic materials.

So instead of having a suspension, these were rolled plates of about 3mm thickness. Now you cannot imagine even synchrotron light to pass through 3mm of iron manganese alloys. A sheet of iron manganese alloys which is about 3mm thin even if the synchrotron radiation can penetrate, the signal will be very very weak. However, neutrons can easily penetrate and by monitoring the transmission signal, they were able to see that there was very small scattering which essentially indicated that there is low magnetic heterogeneity.

Not only that, they also added carbon and nitrogen. Again you can add directly or you can add their respective isotopes. So you can add carbon 12 or carbon 14 or nitrogen 14 and nitrogen 15. However, this study they have not done but you always have this option to maximize the contrast.

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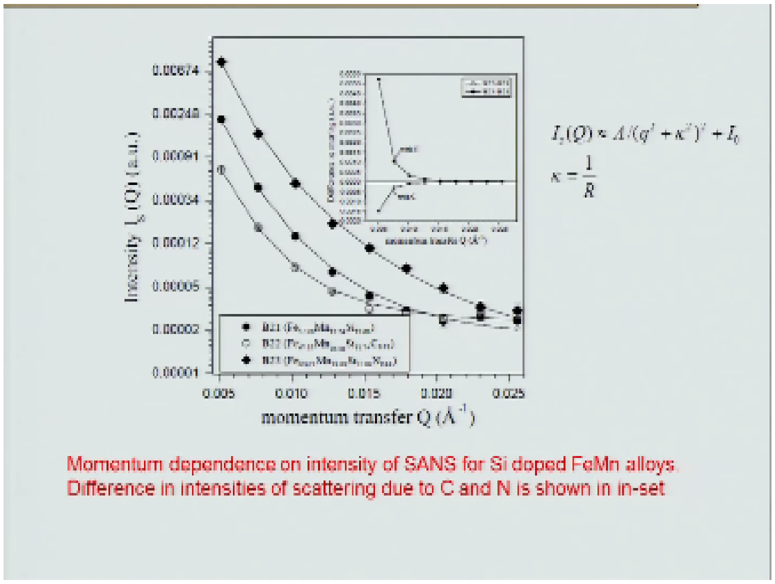

$$I_s(Q) \approx A/(q^2+\kappa^2)^2 + I_0$$

$$\kappa = \frac{1}{R}$$

Momentum dependence on intensity of SANS for Si doped FeMn alloys.
Difference in intensities of scattering due to C and N is shown in in-set

And this is what has been shown over here. So you see that the momentum dependence on intensity. So this is our momentum transfer or Q of sans for silicon doped apron manganese alloys. You see that the difference in intensities of scattering is because of carbonate nitrogen. So this way we can study what happens due to addition of trace elements or interstitial elements on the overall shape memory effect which has tremendous metallurgical as well as mechanical applications so again using only and this can be done only using small angle neutron scattering because we cannot get any information about the magnetic structure using X-ray scattering.

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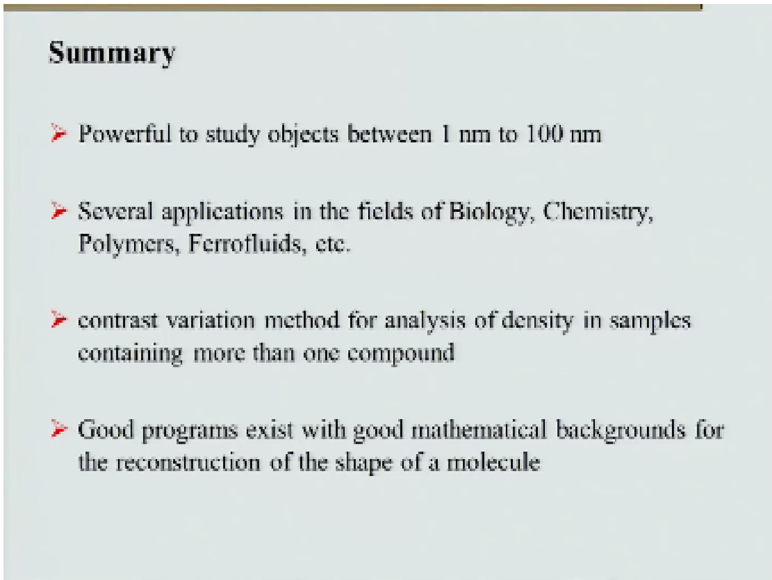

Summary

- Powerful to study objects between 1 nm to 100 nm
- Several applications in the fields of Biology, Chemistry, Polymers, Ferrofluids, etc.
- contrast variation method for analysis of density in samples containing more than one compound
- Good programs exist with good mathematical backgrounds for the reconstruction of the shape of a molecule

So to summarize I hope you appreciate that small angle neutron scattering offers us a very powerful tool to study objects between 1200 nm. It can be used or rather it is used widely for several applications in the field of Biology, Chemistry, Polymers as well as Ferro fluids. The most important advantage is the contrast variation method for analysis of density in samples containing more than one compound.

So we can get different materials, different kind of thesis which is present in the same material. We are able to separate them in a much better way using small angle neutron scattering and this aspect which I have not touched about but as we already know there are very good software's which are available for analyzing small angle X-ray scattering as well as small angle neutron scattering data and we can reconstruct the size, shape as well as distribution of the particle under consideration.

With small angle neutron scattering, we can also use to get information about the size, shape and distribution of the macro molecule which are of the order of which fall in the same size regime. So with this we come to the end of this module. Having said that I hope I have been able to show you or rather give you a glimpse of the entire spectrum of advance characterization technique that use diffraction scattering as well as spectroscopy principles.

The major aim of this module was to just expose you to all these techniques so that you become aware. I have not really gone into greater details and there are plenty of texts available on these techniques. I therefore request you to go through the text which is on the website where you can see what all textbooks can be offered. Having said that, let me remind you that all these techniques what we have used are very much dependent on the instruments that we are having.

And therefore the best information can be obtained at the facilities which offer all these techniques. So the main point is if at all you are going to carry out such synchrotron diffraction or scattering experiment or neutron diffraction or neutron scattering experiments, it is always suggested to spend some time at that particular facility and carry out your experiments. With this I will wish you all the best for your advance characterization techniques which you will probably find use at some time in your academic career thank you.

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Acknowledgement
Ministry of Human Resources & Development

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Co-ordinator, NPTEL IIT Kanpur

Prof. Satyaki Roy

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