

INTernational REMote Student Training at JINR

Introduction to Neutron Scattering Experiments at Large Scale Facilities

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CHAPTER 1

1:0 NEUTRONS

A neutral atom is composed of:

- Positively charged Protons
- Negatively charged Electrons
- Neutrally charged **Neutrons**

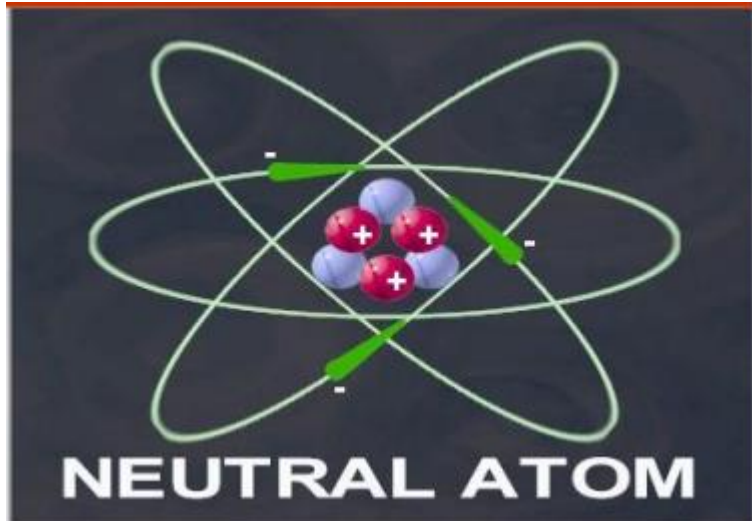


Fig1.1

1:1 THE NUCLEUS OF AN ATOM

The nucleus of the atom is the inner core of the atom. It is made up of protons and neutrons together. The nucleus determines the identity of the element and its atomic mass. This atomic mass number is the sum of the total number of protons and neutrons in the nucleus. Proton and neutrons have essentially the same mass but only the proton is charged while the neutron has no charge

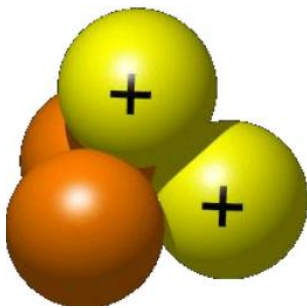


Fig1.2: The nucleus of an atom

1:2 THE NEUTRON

Neutrons are the other particle found in the nucleus of an atom. Unlike protons and electrons, however, neutrons carry no electrical charge and are thus "neutral." Atoms of a given element do not always contain the same number of neutrons. This phenomenon is referred to as Isotopy.

1:2.1 NEUTRON PROPERTIES

There are basic properties of the neutron that must be presented in order to understand how and why neutrons are unique relative to the other components of the radiation environment in space. Some of them include:

- a. **The lifetime of the neutron:** A free neutron has a half life of approximately 15 minutes (the neutron decays via beta decay to a proton and an antineutrino). What this effectively means is that there are no neutrons in the primary Galactic Cosmic Ray (GCR) environment. By the time GCR enters our solar system, any neutron created at the source of the GCR has decayed away. There are some neutrons emitted from the sun that do live long enough to reach the vicinity of Earth, but those are very few in number compared to the other radiations emitted from the sun.
- b. **High Penetration Power:** However, compared to charged particles, neutrons are much more highly penetrating and can go through shielding without interacting because they have no charge. As a result, neutrons can become more significant in terms of their contribution to the total dose and effective dose behind thick shielding in space.
- c. **Neutron Dosage:** The dose and subsequent biological damage delivered by neutrons depends greatly on the energy of the neutron. On Earth, radioactive sources of neutrons generate neutrons between 0 and 10 MeV. In space, the energy spectrum of neutrons is much different, with neutron energies going well beyond 10 MeV, up to several TeV. The presence of high energy neutrons poses problems not only for dosimetry and monitoring, but also for radiobiologists seeking to understand the biological effects from high-energy neutrons.

1:2.2 CLASSIFICATION OF NEUTRONS BASED ON ENERGY

This classification of neutrons was done primarily to distinguish how neutrons interact with materials. They include:

- a. **Cold neutrons:** These have energies below thermal energies), typically corresponding to meV and sub meV energies, i.e., from 0 to 0.025 eV. Cold neutrons are used in a variety of applications, including studies on the structure of bio molecules. Both scattering and

absorption reactions can occur at these energies (scattering will be discussed as we progress).

- b. **Thermal neutrons:** The energy of neutrons that are in equilibrium with the motion of the atoms that make up the medium in which the neutron is found. Neutrons colliding with atomic nuclei either pick up energy if they are moving slower than the colliding nucleus, or lose energy if they are moving faster. This constant slowing down and picking up of energy by free neutrons leads to a distribution of neutron energies centered about the most likely energy (thermal energy). The distribution is called a Maxwell – Boltzmann distribution, which is shown in the plot below. For biological materials, scattering is the dominant reaction, although absorption of thermal neutrons by hydrogen and nitrogen is also present and can deliver dose to the biological material.

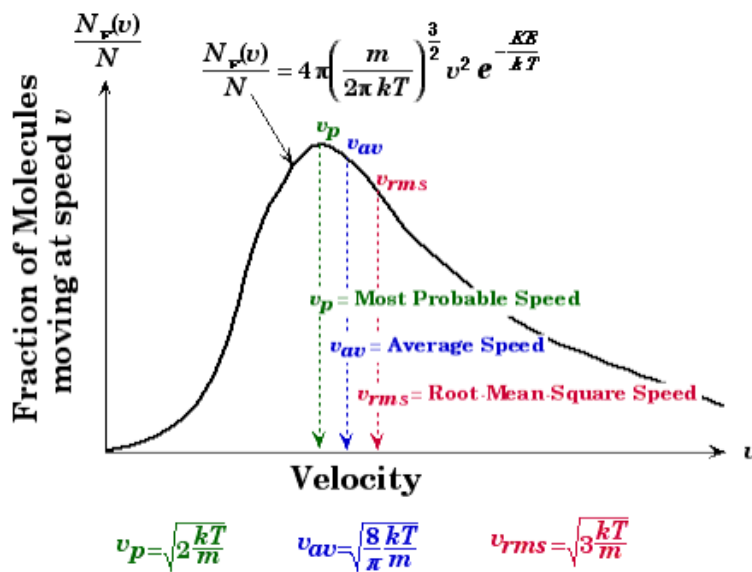


Fig1.3: Maxwell –Boltzmann distribution plot

- c. **Epithermal neutrons:** These have energies that are between thermal (0.025 eV) to a few hundred eV. This represents the transition region between thermal and slow neutrons where “resonances” present in the nuclei that interact with the neutron begin to present themselves.
- d. **Slow neutrons:** These neutrons generally have energies between 100 eV to 0.5 or 1 MeV. In this energy range, many of the nuclei that the neutron interacts with have nuclear structure within the combination of neutrons and protons that make up the nucleus, and that structure leads to an enhanced probability of interaction between the neutron and nucleus.
- e. **Fast neutrons:** These have generally between 0.5 and 20 MeV of energy. These are the energies of neutrons emitted by fission sources. This represents the upper limit of where most of the research on neutron radiobiology and fundamental neutron interaction cross sections has been done. This is not to say that there has been no

research above these energies, it's just that the amount of information and data at energies above 20 MeV is not as well established as the data below.

- f. **High energy neutrons:** These have energies above 20 MeV. A significant fraction of the dose and effective dose from neutrons in space is delivered by high energy neutrons and represents the region of greatest uncertainty in the biological effects from neutrons in space.

1:3 X-RAYS

X-rays are electromagnetic radiation of exactly the same nature as light but of very much shorter wavelength. X-ray wavelengths are in the range 0.5 – 2.5 Å while that of visible light is ~ 6000 Å.

X-rays are emitted when high-energy electrons or any other charged particles bombard a metal target. The x-ray spectrum typically consists of a broad continuous band containing a series of sharp lines. X-rays are the result of the slowing down of high-energy electrons as they strike the target. It may take several interactions with the atoms of the target before the electron gives up all its kinetic energy. The amount of kinetic energy given up in any interaction can vary from zero up to the entire kinetic energy of the electron. Therefore, the wavelength of radiation from these interactions lies in a continuous range from some minimum value up to infinity.

1:3.1 X-RAY SPECTRUM

X-rays are produced when accelerated electrons collide with the target. The loss of energy of the electrons due to impact is manifested as x-rays. X-ray radiation is produced in an x-ray tube. Most of the kinetic energy of the electrons striking the target is converted into heat, less than 1% being transformed into x-rays.

The Kinetic Energy of the accelerated X-rays is given by:

$$E_k = eV = \frac{1}{2}mv^2$$

Where e = electron charge (1.6×10^{-16} C)

E_k = kinetic energy,

V = applied voltage,

m = mass of the electron (9.1×10^{-31} kg),

v = electron velocity (m/s)

1:3.2 CONTINUOUS X-RAY SPECTRUM

Continuous spectrum arises due to the deceleration of the electrons hitting the target. This type of radiation is known as the bremsstrahlung x-rays, German for “braking radiation”. It is also called polychromatic, continuous or white radiation. Some electrons lose all the energy in a single collision with a target atom.

1:3.2.1 Properties

1. It has a smooth and monotonic function of intensity vs wavelength when plotted.
2. The intensity is zero up to a certain wavelength, the short wavelength limit (λ_{SWL}). The electrons transfer all their energy into photon energy:

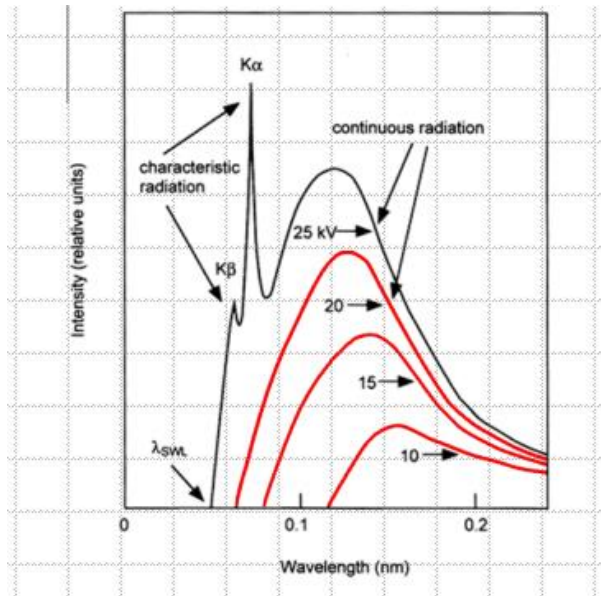


Fig1.4: Intensity vs wavelength plot

Using $eV = hv_{max}$

$$\lambda_{SWL} = \frac{c}{v_{max}} = \frac{hc}{eV}$$

$$\lambda_{SWL} = \frac{12.398 \times 10^3}{V}$$

Where λ is in \AA and V is in volts.

v_{max} = maximum velocity

3. The total x-ray energy emitted per second depends on the atomic number, Z of the target material and on the x-ray tube current. This total x-ray intensity is given by:

$$I_{cont} = AiZV^m$$

Where A = proportionality constant

i = tube current (measure of the number of electrons per second striking the target)

and m = constant ≈ 2

1:3.3 CHARACTERISTICS X-RAY SPECTRUM

Characteristics X-rays were discovered by W.H Bragg and became systemized by H.G. Moseley.

The characteristic peak is created in when a hole in the inner shell, created by a collision event, is filled by an electron from higher energy shell. Let a K-shell electron be knocked out, the vacancy can be filled by an electron from the L-shell or the M-shell.

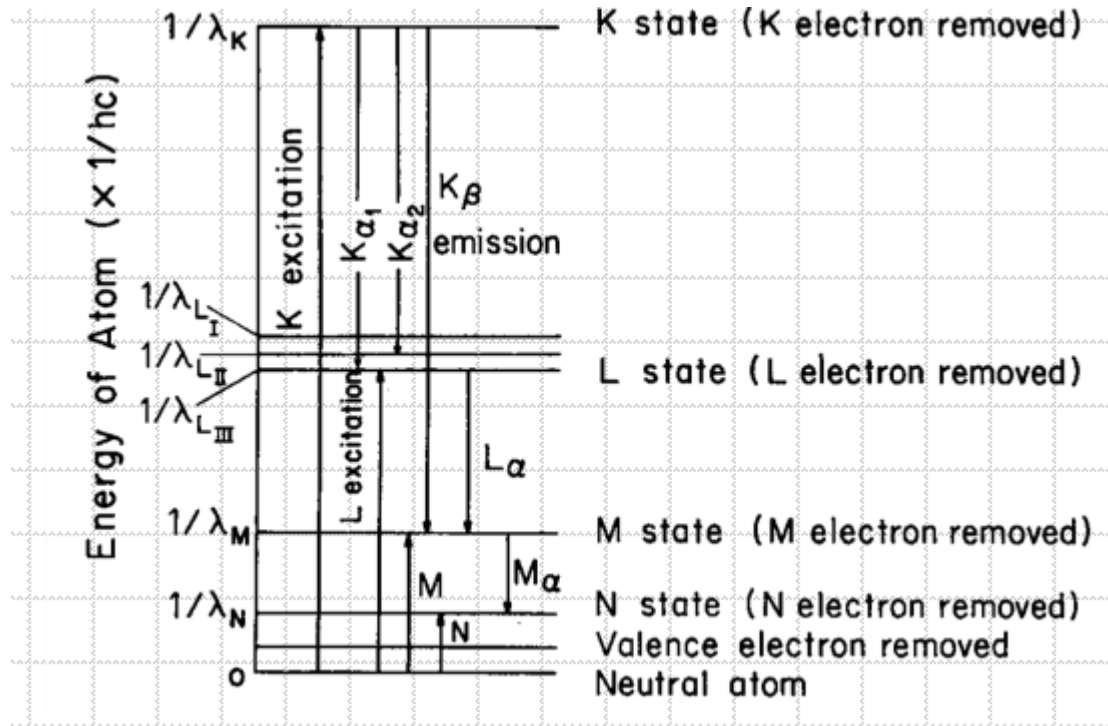


Fig1.5: Movement of electrons between shells

1:3.3.1 PROPERTIES

1. Only the K-lines are usually useful in x-ray diffraction.
2. There are several lines in the K-set. The strongest are $k_{\alpha_1}, k_{\alpha_2}, k_{\beta_1}$
3. α_1 and α_2 components are not always resolved.
4. k_{α_1} is always about twice as strong as k_{α_2} while the ratio of k_{α_1} to k_{β_1} averages about 5:1

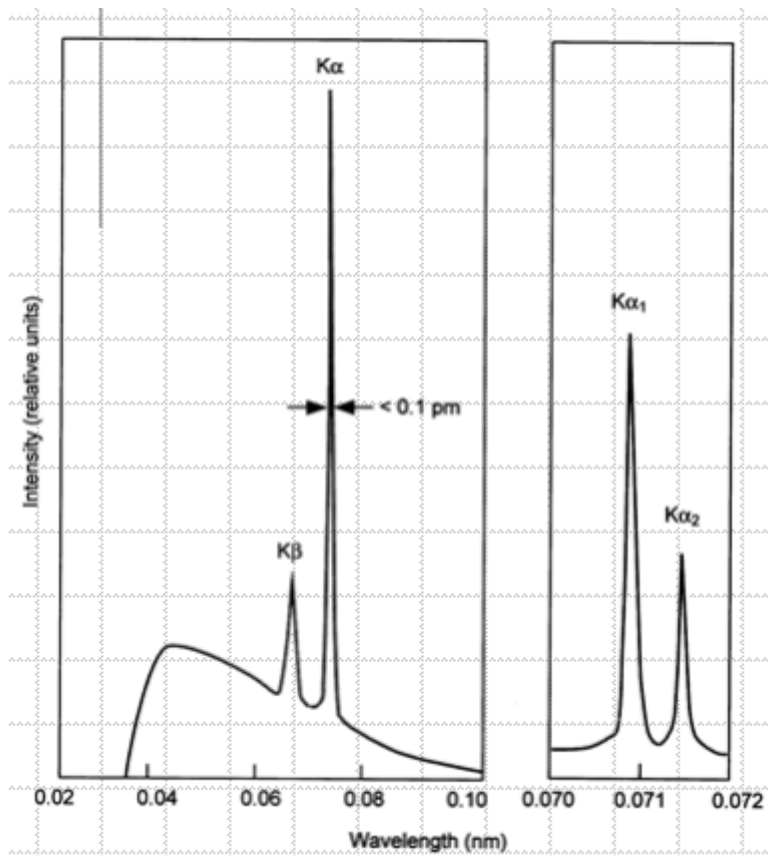


Fig1.6: Intensity vs wavelength plot for the different k-sets

- The intensity of any characteristic line depends both on the tube current, i and the amount by which the applied voltage V exceeds the critical excitation voltage for that line. For a K-line:

$$I_{k\text{-line}} = Bi(V - V_k)^n$$

Where B = proportionality constant

V_k = the K excitation voltage

$n \approx 1.5$

- Characteristic lines are also very narrow, most of them less than 0.001 \AA wide.
- High intensity and narrow K-lines makes x-ray diffraction possible, since it generally requires the use of monochromatic radiation.

1:3.4 X-RAY ABSORPTION

When x-rays encounter any form of matter, they are partly transmitted and partly absorbed. It was found experimentally that:

$$I \propto x$$

In differential form, we write:

$$-\frac{dI}{I} = \mu dx$$

Where I = Intensity

x= distance

μ = linear absorption coefficient

1:3.5 SOURCES OF X-RAYS

X-rays are produced in x-ray tubes. The tubes must have source of electrons, high accelerating voltage and a metal target.

X-ray tubes are of two types: The Gas Tube (which is obsolete now) and the filament tube.

THE FILAMENT TUBE

It was Invented by Coolidge in 1913. It is the most widely-used laboratory X-ray source. Its Major components are a water-cooled target (anode) and a tungsten filament (cathode) that emits electrons, A high potential (up to 60 kV) is maintained between the filament and the anode, accelerating the electrons into the the anode and generating X-rays.

The interior of the tube is evacuated for the electron beam; thin beryllium windows transmit the X-rays and cooling water is circulated through the anode to keep it from melting (>99% of input power generates heat).

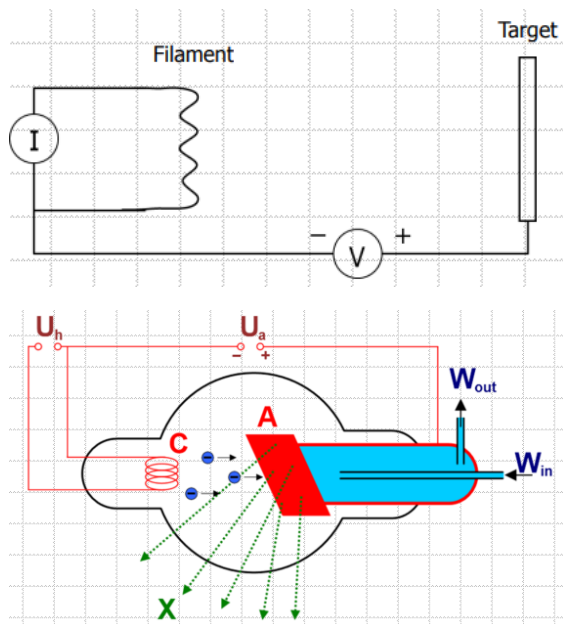


Fig1.7: The X-ray tube

1:4 NEUTRON SCATTERING

Neutron scattering is done to determine the positions and motions of atoms in condensed matter. It is the technique of choice for condensed matter investigations in general.

1:4.1 CHARACTERISTICS OF NEUTRON SCATTERING

1. Neutron scattering lengths vary "wildly" with atomic number and are independent of momentum transfer.
2. Neutrons interact through nuclear interactions. X-rays interact with matter through electromagnetic interactions with the electron cloud of atoms. Electron beams interact through electrostatic interactions. Light interacts with matter through the polarizability and is sensitive to fluctuations in the index of refraction. For this, neutrons have high penetration (low absorption).
3. In neutron scattering, scattering nuclei are point particles neutron scattering does not contain scattering from the electron cloud.
4. Neutron sources are very expensive to build and to maintain.
5. Neutron sources are characterized by relatively low fluxes and have limited use in investigations of rapid time dependent processes.

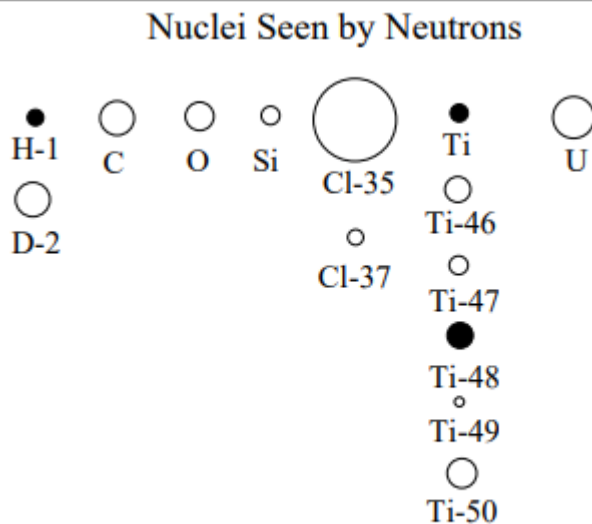


Fig1.8: Neutrons interacting with the Nuclei

1:4.2 TYPES OF NEUTRON SCATTERING

1. **Measurement of the sample transmission:** This measurement requires a monochromatic beam (or the time-of-flight method), some collimation and a simple neutron detector (end-window counter). Transmission measurements contain information about the sample content and the relative fractions of the various elements.

- 2. Elastic neutron scattering:** This consists in measuring the scattered intensity with varying scattering angle. This is a way of resolving the scattering variable,

$$Q = \left(\frac{4\pi}{\lambda}\right)\sin\left(\frac{\theta}{2}\right)$$

Where, λ = neutron wavelength

θ = scattering angle

Elastic scattering is performed by either step-scanning or using a position-sensitive detector. The instruments that can be used include diffractometers, reflectometers and SANS instruments. These instruments investigate sample structures either in crystalline or amorphous systems.

- 3. Quasielastic/inelastic neutron scattering:** This consists of monochromation, collimation, scattering from a sample, analysis of the neutron energies then detection. The extra step uses a crystal analyzer (or the time-of flight method) in order to resolve the energy transfer during scattering. In this case both

$Q = k_s - k_i$ and $E = E_s - E_i$ are resolved. Quasielastic scattering corresponds to energy transfers around zero, whereas inelastic scattering corresponds to finite energy transfers. The main types of quasielastic/inelastic spectrometers are the triple axis, the time-of-flight, and the backscattering spectrometers. They investigate sample dynamics and structure. Inelastic instruments are used to investigate phonon, optic and other types of normal modes. Quasielastic instruments are used to investigate diffusive modes mostly.

1:5 NEUTRON DIFFRACTION

Neutron diffraction is used to measure the differential cross section, $\frac{d\sigma}{d\Omega}$ and hence the static structure of materials.

Depending on the scattering angle, structure on different length scales, d , is measured using

$$\frac{2\pi}{Q} = d = \frac{\lambda}{2\sin\theta}$$

For crystalline solids & liquids, we use a wide angle diffraction. For large structures, e.g. polymers, colloids, micelles, etc. we use small-angle neutron scattering.

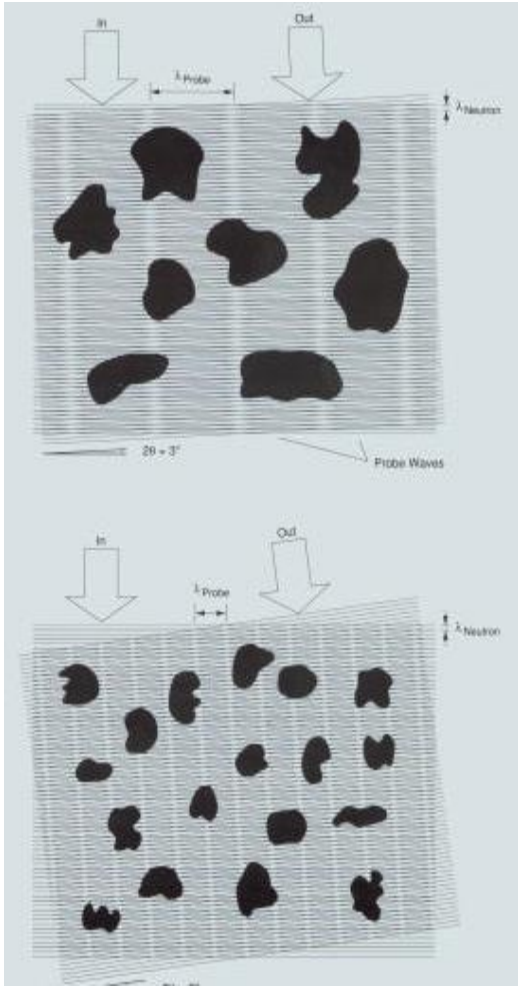


Fig1.9: Neutron diffraction in crystals

CHAPTER TWO

1.0 USING SASVIEW

SasView is a Software Package used for Small Angle Scattering Analysis. It was originally developed as part of the NSF DANSE project and it was formerly referred to as SansView. It is a free-to-use, open source project. SasView is presently managed by an international collaboration of facilities.



Fig2.1 Front page of the SasView software

1.1 USING SASVIEW FOR POLARIZATION/MAGNETIC SCATTERING

The scattering length density ($SLD = \beta$) for magnetic systems is a combination of the nuclear and magnetic Scattering Length Density (SLD). The resulting effective SLD for polarised neutrons depends on the state of spin of the neutron before and after being scattered in the sample.

We evaluate models in Sasview which define a Scattering Length Density parameter as magnetic models introducing the magnetisation (vector) $\mathbf{M} = M(\cos\theta_M, \sin\theta_M \sin\phi_M, \sin\theta_M \cos\phi_M)$ and the associated magnetic SLD given by the simple relation:

$$\beta_m = b_H M$$

Where $b_H = \frac{\gamma r_0}{2\mu_B}$

M= magnetization magnitude

$\gamma = \text{gyromagnetic ratio} = -1.913$

$\mu_B = \text{Bohr Magnetron}$

$r_0 = \text{classical radius of electron}$

It should also be noted that the external field coincides with the polarization axis. This polarisation axis determines the scattering geometry. The polarisation turns adiabatically to the guide field of the instrument, before and after the field at the sample position. The observed spin-resolved scattering at the detector is not changed by this operation. The magnetisation vector and the magnetic field (defining a symmetry axis) will be oriented symmetrically around the field. We couple the magnetisation with the field axis via a constrained fit For AC oscillating/rotation field varying in space and with time, this will allow to easily categorize a phase shift of the magnetisation lagging behind a magnetic field varying from time frame to time frame.

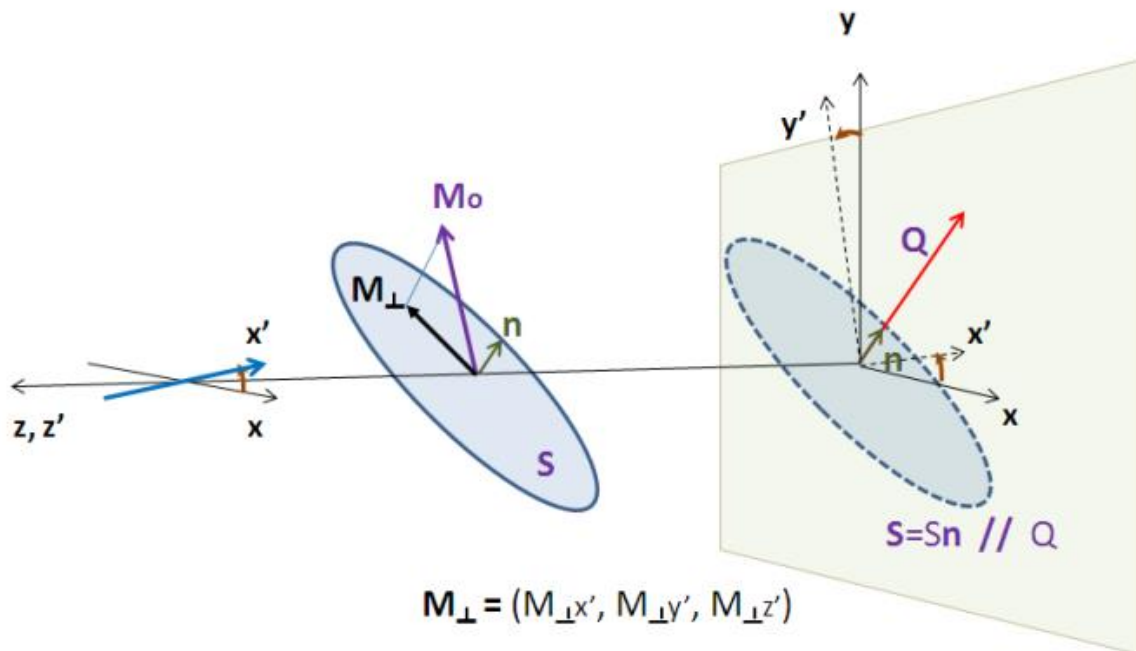


Fig2.2 Magnetic scattering and polarization

The user input entered into the data explorer panel:

sld_M - the magnetic scattering length density :math:`b_H M`
sld_m θ - the polar angle :math:`\theta_M` of the magnetisation vector
sld_m ϕ - the azimuthal angle :math:`\phi_M` of the magnetisation vector
up_frac_i - polarisation efficiency :math:`u_i` *before* the sample
up_frac_f - polarisation efficiency :math:`u_f` *after* the sample
p_theta - the inclination :math:`\theta_P` of the polarisation from the beam axis
p_phi - the rotation angle :math:`\phi_P` around the incoming beam axis

CHAPTER THREE

3.0 WORKING WITH SASVIEW

We used the Data Operation tool in SasView to subtract an underlying function from experimental data. Below were the various steps taken to achieve this:

3.1 RUNNING SASVIEW ON WINDOWS

Select SasView from the Start bar after which we select the software itself from the 'All Programs'. The software windows appears on the screen.

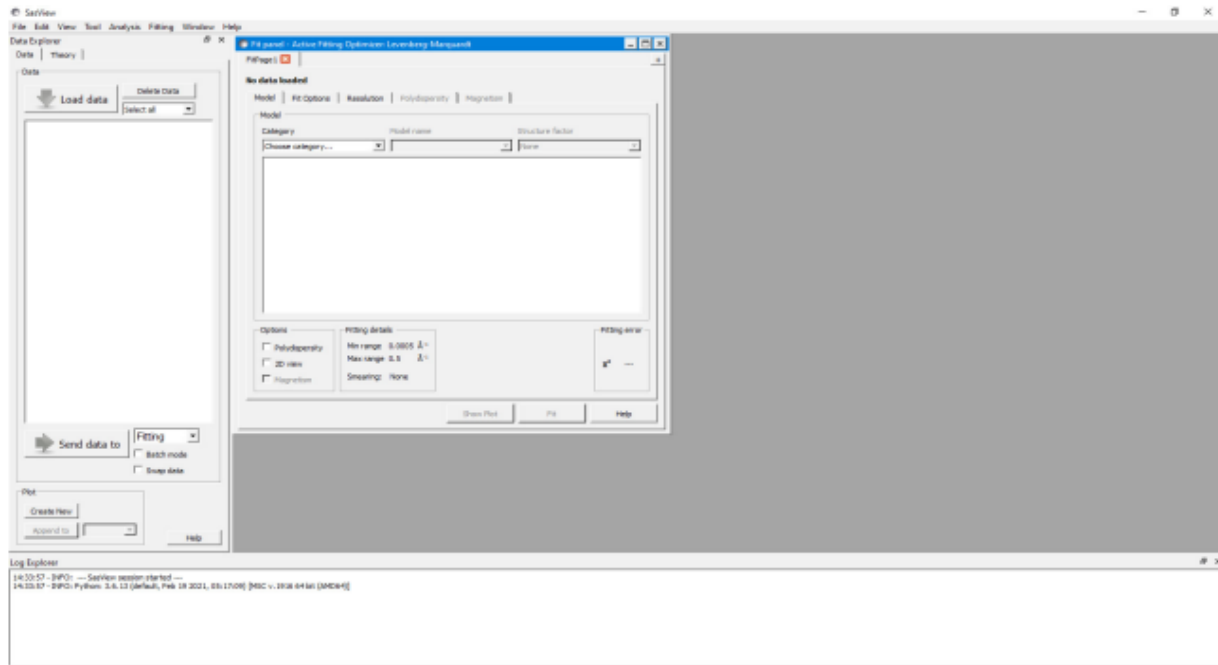


Fig3.1

3.2 EXAMPLE

The quasi-lamellar scattering from within a bundle of nylon-6 (polyamide-6) fibres is superimposed on top of the scattering from the surfaces of the fibres themselves. Subtracting this surface scattering highlights the scattering from the underlying morphology.

3.3 WORKING WITH THE DATA ON SASVIEW

In the Data Explorer panel, click the Load Data button, and navigate to the \test\1d_data folder in the SasView installation directory. Select the ISIS_83404.txt dataset and click the Open button. At the bottom of the Data Explorer panel, ensure the drop-down says Fitting. Then click the Send To button. To see the data, click Create New

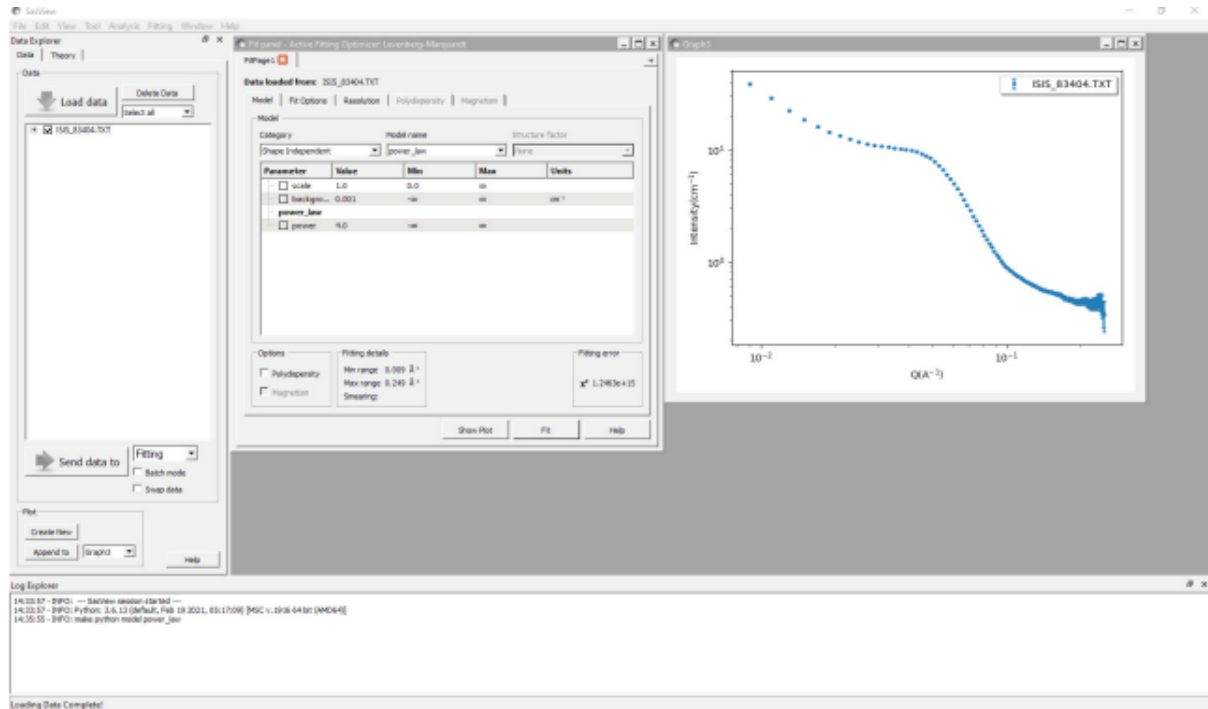


Fig3.2 Viewing the data

We now need to choose the form factor to subtract. Surface scattering can often be approximated by a power law dependence, so here we shall try to subtract a power law with background model from the data. In the FitPage, go to the Category drop-down and select 'Shape Independent'. And in the Model name drop-down select the model 'power_law'.

Now adjust the model parameters until you are satisfied with the shape of the fit and the model parameters. In the screenshot below the parameters are:

Scale	0.007
Background	0.25
Power	1.8

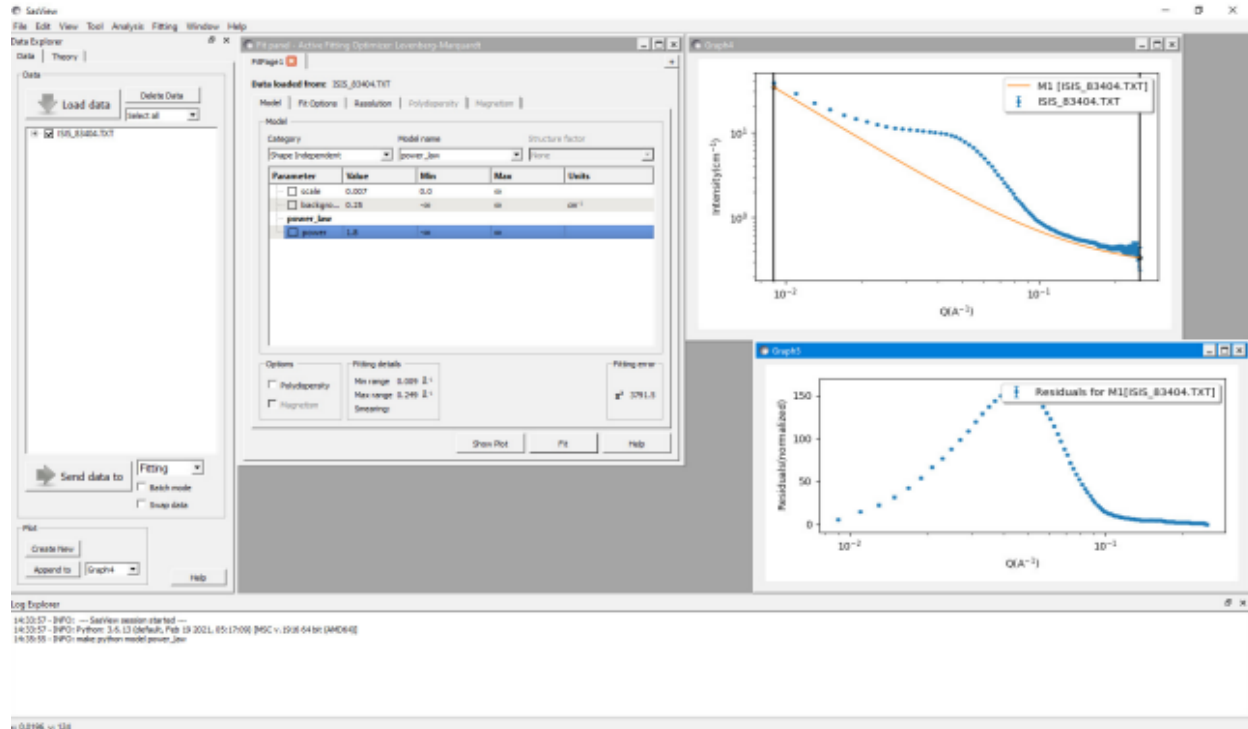


Fig3.3 Viewing the fit and model parameters

Then go to the Tool option on the Menu Bar and select Data Operation. The following window will appear:

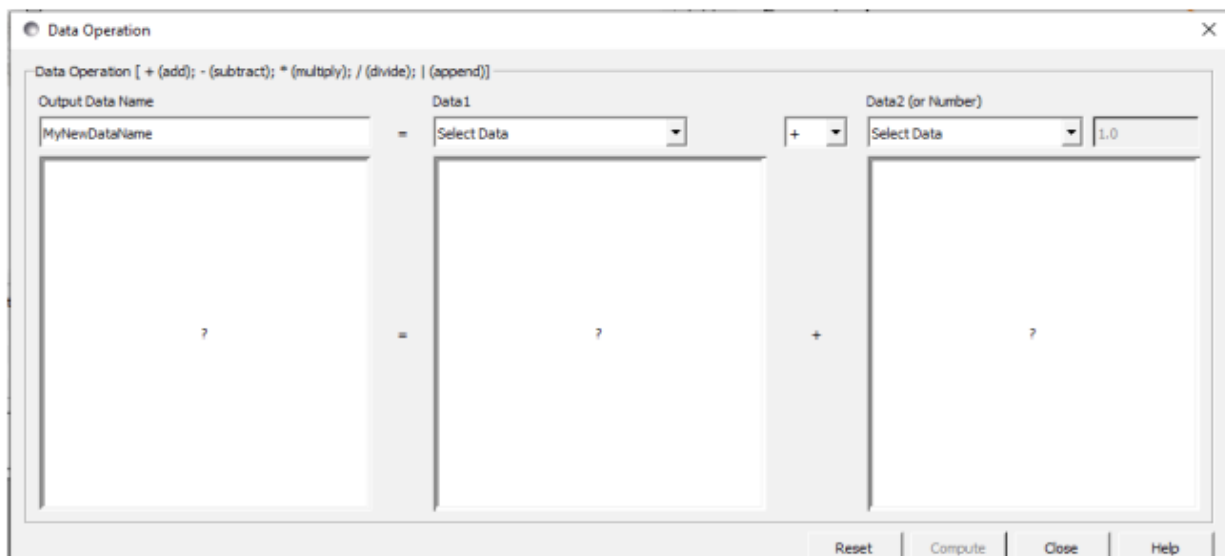


Fig3.4 Selecting Date Operation

Set the 'Output Data Name' to whatever you you want the subtracted dataset to be called; here 83404_pl_sub. Then, use the 'Data1' drop-down to select the experimental dataset ISIS_83404.txt, set the operator to '-' (minus), and use the 'Data2' drop-down to select the

computed model (generically called M# [dataset name], where # is a program-generated reference number); here M1 [ISIS_83404.TXT].

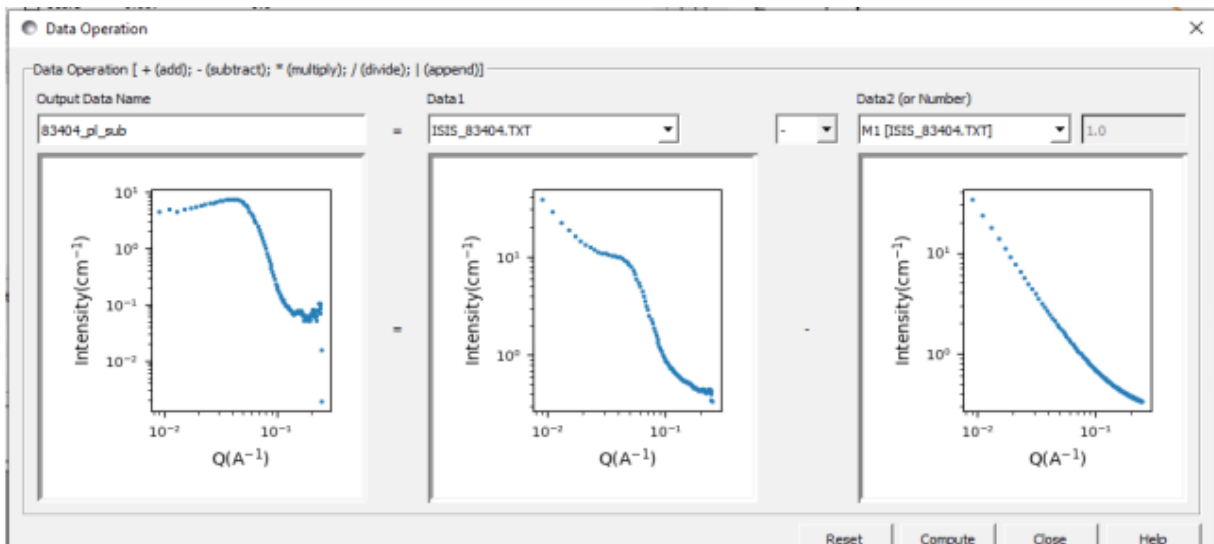


Fig3.5 Generating a Program

Finally, click Apply and Close. The subtracted dataset will appear in the Data Explorer.

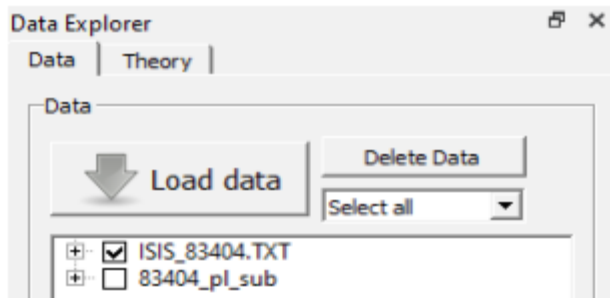


Fig3.6 The Subtracted dataset

...from where it can be plotted, fitted, exported, or further manipulated.

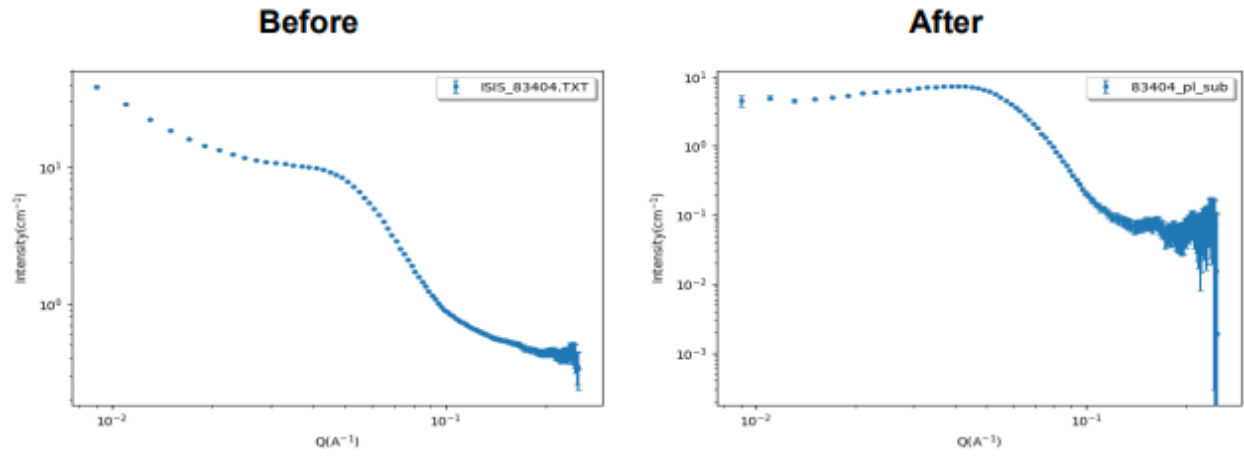


Fig3.7 The plotted dataset

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