Neutron Scattering Program at Dhruva Reactor: Applications to Advanced Materials Research

A. K. Bera, J. Bahadur, Amit Kumar, Debasis Sen and S. M. Yusuf*
Solid State Physics Division
Bhabha Atomic Research Centre
Mumbai-400 085, India

*Email: smyusuf@barc.gov.in

Abstract

Neutron scattering is an indispensable tool for probing structure and dynamics in condensed matter, encompassing a wide multidisciplinary research spectrum. The high penetrating power, simultaneous sensing capabilities of both light and heavy atoms, isotope sensitivity, comparable wavelength/energy to the atomic length scale/basic excitations in materials, capability of measuring atomic magnetic moment *etc*. have made thermal neutron a unique probe for characterization of materials. The current neutron scattering activity in the country is centred around the indigenously built Dhruva research reactor, at Bhabha Atomic Research Centre, Trombay, under the National Facility for Neutron Beam Research (NFNBR) program. These facilities are being used by a large number of research groups (including Physicists, Chemists, Material scientists, Engineers, and Biologists) from all over the country. A variety of materials including ceramics, biological, nuclear, pharmaceuticals, engineering, soft matter, *etc*. are being routinely investigated by these neutron scattering facilities, aiming to design novel materials for societal applications in the domains including electronics/spintronics, quantum technology, energy harvesting and storage, pharmaceuticals, drug delivery and cosmetics, water treatment and nuclear waste management. In this article, we provide a glimpse of basic concepts of the technique of neutron scattering as well as a few recent research highlights based on the neutron scattering facilities in Dhruva.

Key words: *Neutron scattering, magnetic moment, ceramics, soft matter*

1. Introduction

In 1930, Walther Bothe and Herbert Becker observed that if energetic alpha particles impinged on certain light elements, such as beryllium, an unusual penetrating radiation was produced. The observed radiation was not influenced by an electric field. Hence, it was thought to be gamma radiation. In 1932, Chadwick repeated this experiment. He targeted the produced radiation at paraffin wax, a hydrocarbon with large hydrogen content, hence offering a target dense with protons. He measured the range of the ejected protons and also analyzed how the new radiation affected the atoms of various gases. He concluded that the new radiation was not gamma rays, but neutral particles with about the same mass as the proton. These particles were neutrons [1,2]. Chadwick was awarded the Nobel Prize in Physics in 1935 for this discovery. Charge-neutrality of neutrons makes it impervious to the Coulomb interaction, allowing it to penetrate deeper into matter than other subatomic particles. When contemplating the application of this special attribute, one can imagine that using the scattering of neutrons by matter, crucial information can be obtained regarding the bulk characteristics of that matter. In fact, this opens a new dimension of possible physics knowledge that can be acquired using neutrons as probe for condensed matter.

Neutrons have a set of unique properties which make them an indispensable tool in a variety of investigations in Physics, Chemistry, Biology and Materials Science. The high penetrating power, sensing capabilities of both light as well as heavy atoms simultaneously, isotope sensitivity, comparable wavelength/energy to the atomic length scale/basic excitations in materials, capability of measuring atomic magnetic moment *etc*. have made thermal neutron a unique probe [3,4] for materials' characterization. Unlike x-ray photons with a similar wavelength, that interacts with the electron-cloud surrounding the nucleus, neutrons interact primarily with the nucleus. This makes neutron very sensitive to light atoms like hydrogen, which are difficult to detect using x-rays. Neutrons can also distinguish between the neighboring atoms of the periodic table like iron, cobalt, and nickel. Scattering and absorption cross sections of neutron vary widely among the isotopes of an element and thus neutron can distinguish between the isotopes. Neutrons have a magnetic moment, which allows them to interact with the atomic magnetic moments through dipole–dipole interaction. Therefore, neutrons can be used to investigate microscopic magnetic structures. It is worth mentioning that in 1994 the Nobel Prize in Physics was awarded [5] to Clifford G. Shull along with Bertram Brockhouse for their immense contribution in development of neutron scattering techniques. There are currently more than 30 neutron research facilities around the world that produce thermal neutrons for experimental research purpose. Neutrons are usually produced by two main routes (i) fission of Uranium-235 in a chain reaction (in research reactors) and (ii) by a process known as, "Spallation", in which bombarding heavy nuclei with high energy (~1 GeV) protons results in spalls of ~20-30 neutrons ejected from the heavy metal targets. In both the cases, a moderation process is required to slow them down to become so-called thermal neutrons.

What are the resources available to researchers in India for using neutron scattering ?

It is nearly 65 years since study of materials began with facilities at the then Atomic Energy Establishment Trombay (later renamed as Bhabha Atomic Research Centre or BARC), using India's first nuclear reactor, Apsara. Over the following decades, CIRUS and Dhruva research reactors became operational at BARC. Further, after decommissioning of Apsara reactor in 2009, its upgraded version Apsara-U reactor became operational in 2018.Today, the neutron scattering activity in India is mainly centered around the indigenously built Dhruva reactor (a medium flux, natural Uranium, heavy water moderated, heavy water-cooled reactor; max

thermal power 100 MW, max central thermal neutron flux $\sim 1.8 \times 10^{14}$ neutrons/cm^2/s) and it serves as the National Facility for Neutron Beam Research (NFNBR).

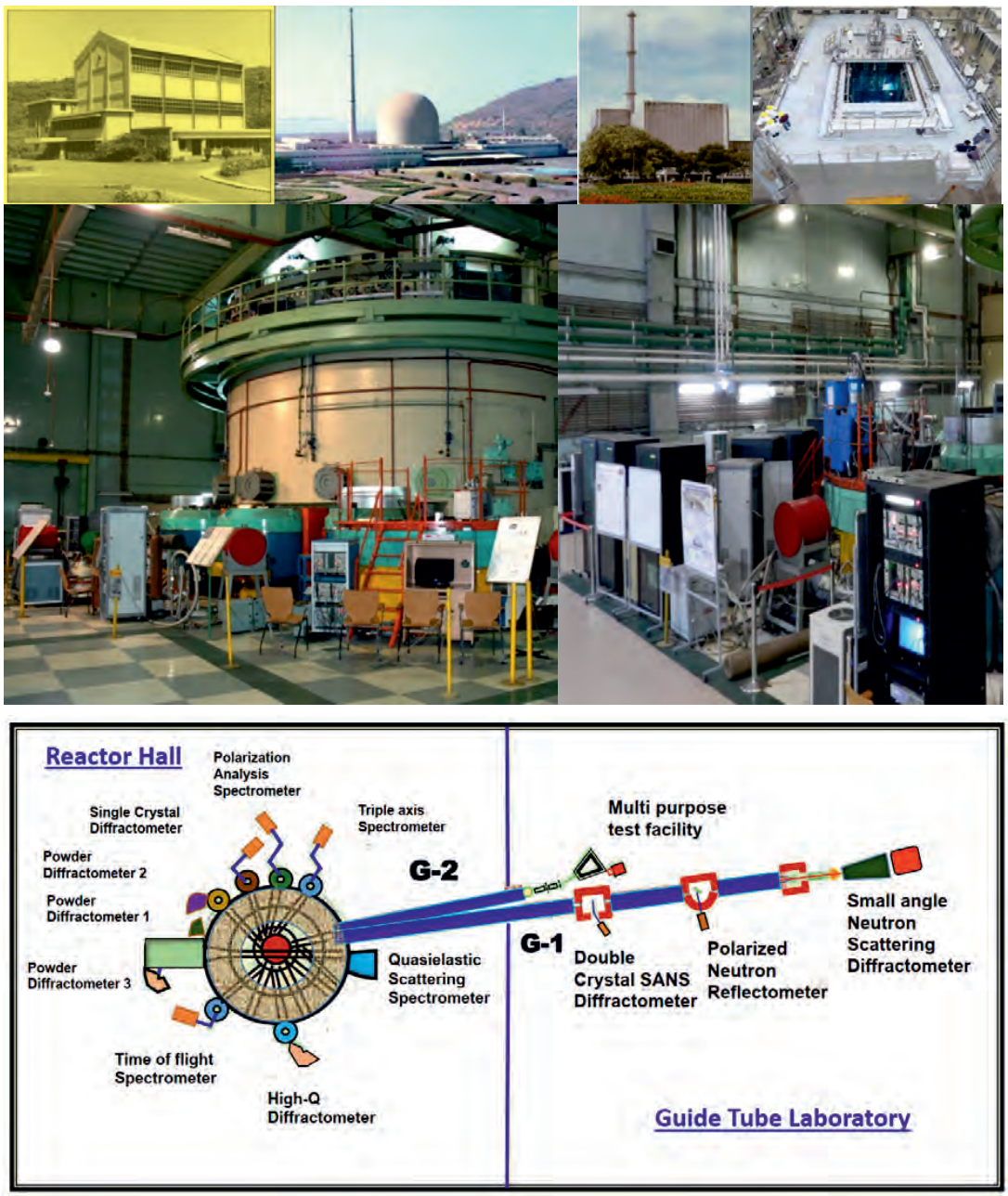

Figure 1: Top panel (Left to right): Apsara, CIRUS, Dhruva, and Apsara-U reactors at Trombay. Middle panel: Various neutron scattering facilities at Dhruva reactor. Bottom panel: Schematic diagram showing the location of the various neutron scattering instruments in Dhruva reactor hall and at the adjacent guide tube laboratory.

Where the atoms are and what they do ?

It is worth mentioning that the length-scale that can be differentiated by human eye is typically 0.1 mm and the dynamics that can be perceived is typical 30 to 60 frames per second. However, structure of materials exists at various length scales and similarly the characteristic dynamics in materials also occur at different time scales. Neutron scattering offers probing such structure (typically in the range of 1- 1000 Å length scale) as well as the dynamics (typically in the range of picosecond to microsecond time scale).

Often, in a typical neutron scattering experiment, neutrons with a particular energy are incident on the material under investigation. The neutrons interact with the material and the intensity of scattered neutrons is analyzed with respect to the wave vector transfer and/or the energy transfer. The mathematical analysis of such dependence of scattering intensity unveils the structure and dynamics prevailing in materials.

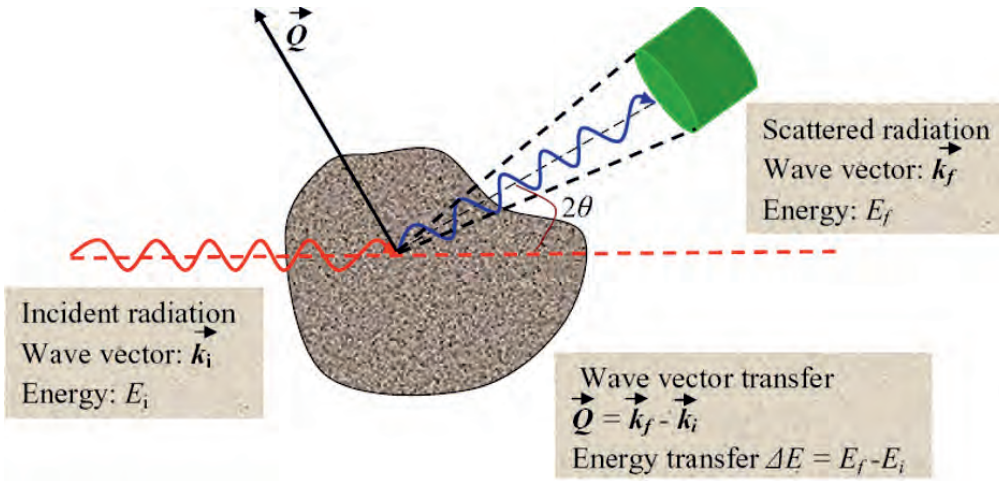

Figure 2: The schematic diagram illustrating neutron scattering experiments with monochromatic radiation.

Of course, accessing the wide range of length and time scales by a single instrument for characterizing all kinds of materials is utopian. In reality, every neutron scattering instrument has its own window as far as the probing of the structure and dynamics is concerned.

At the Dhruva reactor, there are 14 indigenously developed neutron scattering facilities under the NFNBR. Among them, 12 facilities are operated and maintained by Solid State Physics Division of BARC. One facility is operated and maintained by Technical Physics Division, BARC and one by DAE-UGC-CSR, Mumbai centre. Apart from in-house research, more than 50 user groups (including Physicists, Chemists, Material scientists, Engineers, and Biologists) from various universities, national/international institutes as well as DAE institutes, use these facilities every year.

As neutron scattering provides quantitative information about the atomic positions and its motion, along with the magnetic properties, a wide range of materials are being investigated using these facilities. These include crystalline and amorphous materials, various magnets, liquid crystals, superconducting ceramics, proteins, polymers, *etc*. Neutron scattering provides fundamental understanding of structure and dynamics of materials and thus helps to design novel materials that aim to improve our daily lives. The in-depth experiments and quantitative analysis

help to design materials for societal applications in the domain including electronics/spintronics, quantum technology, energy harvesting and storage, pharmaceuticals, drug delivery and cosmetics, water treatment and nuclear waste management.

The research under the NFNBR can be broadly classified into three categories: i) Long-range and short-range structural and magnetic ordering (in the atomic length scale (Å)) in advanced materials; ii) Large-scale structures in soft matter and nano-structured materials, where one is interested in a length scale of typically 10 to 1000 Å and iii) Pico to micro second dynamics in functional materials. The neutron research leads to high quality publications (~150 /year) in international peer reviewed journals, PhD theses(more than 10/year), summer project/internship. In addition, a few neutron instruments and associated components are built there and exported to foreign countries under various MoUs.

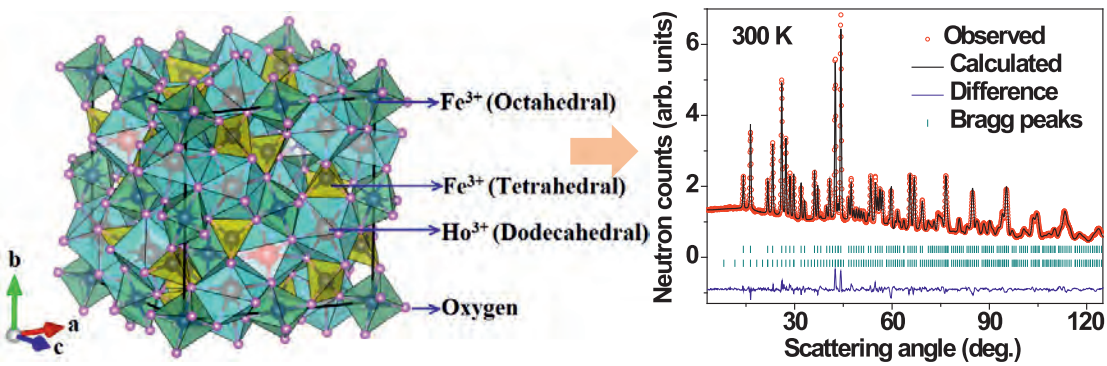

Figure 3: The long-range ordering in crystalline material, $Ho_3Fe_5O_{12}$[6] is manifested as the Bragg peaks in a neutron diffraction experiment (measured using the PD-2 diffractometer, Dhruva). Mathematical analysis of such pattern provides atomic and magnetic structures of materials.

The nature of atomic arrangement in a material often governs its various physicochemical properties and thus it is required to understand such atomic arrangement in materials. As mentioned above, neutrons can give the nature of structural ordering in crystals as well as magnetic ordering in magnetic materials. It is worth mentioning that long-range ordering exists in a crystalline material while only short-range ordering is observed in glassy and non-crystalline materials. Neutron diffraction is a powerful and nondestructive technique to study ordered structures. Diffraction methods can be categorized on the basis of different interactions: i) nuclear diffraction: here diffraction takes place due to the interaction between neutrons and atomic nuclei, and ii) magnetic diffraction: diffraction due to the interaction between the magnetic moments of neutrons and magnetic moments of atoms. The neutron diffraction technique can be applied to study long-range (crystalline solids) as well as short-range (amorphous) ordered materials. The ordering in real space is manifested as peak like feature in the scattering data (Fig. 3).

As previously mentioned, one of the unique features about neutron diffraction is the neutron's magnetic moment that can interact with the magnetic moment of the material under examination. Therefore, neutron diffraction can provide the microscopic atomic level information of the spin arrangement in a given material and hence, the most powerful technique for studying ground state magnetic properties [7,8].

Figure 4: Photographs of the various neutron diffraction instruments at Dhruva reactor, BARC, Trombay, to study crystal and magnetic materials.

Under the NFNBR program, there are five neutron diffractometers (Fig. 4); a four- circle single-crystal diffractometer three powder diffractometers (PD-1, PD-2, PD-3), and a high-Q diffractometer. Additionally, a polarized neutron spectrometer [9,10] is available to study long/short range magnetic ordering in materials. Besides, dedicated sample environments, including (i) a wide sample temperature range of 1.5-1300 K (by using He-based closed cycle refrigerators and high temperature furnace), (ii) magnetic field range over 0-7 Tesla, and (iii) pressure up to 2.5 GPa are available to investigate materials under different thermodynamic conditions. Huge variety of materials including ceramic, alloys, molecular magnets, inter-metallic, hydrogen-based compounds, and glasses *etc*. are being routinely investigated by these neutron diffraction instruments. Some of the recent significant research highlights using these facilities are described below.

The neutron diffractometers are extensively used to achieve microscopic understanding of static magnetic ordering in the several classes of advanced magnetic materials [11-21], such as low-dimensional materials, spintronic materials, multi-ferroic materials, molecular magnetic materials, magneto-caloric materials, and permanent magnets. Fundamentally, magnetic ordering appears as a second ordered phase in addition to the phase corresponding to the crystalline ordering. If the magnetic and crystallographic unit cells are identical, as in case of ferro and ferrimagnetic ordering, enhancement of intensities of low angle nuclear Bragg peaks is observed below the magnetic ordering temperature (TC), as depicted in Fig. 5. In case of antiferromagnetic ordering with magnetic cell doubling, new and purely magnetic Bragg peaks appear below the antiferromagnetic ordering temperature, TN. This is depicted in Fig. 6 in case of a low dimensional quantum magneticsystem, $Na_2Co_2TeO_6$[11].

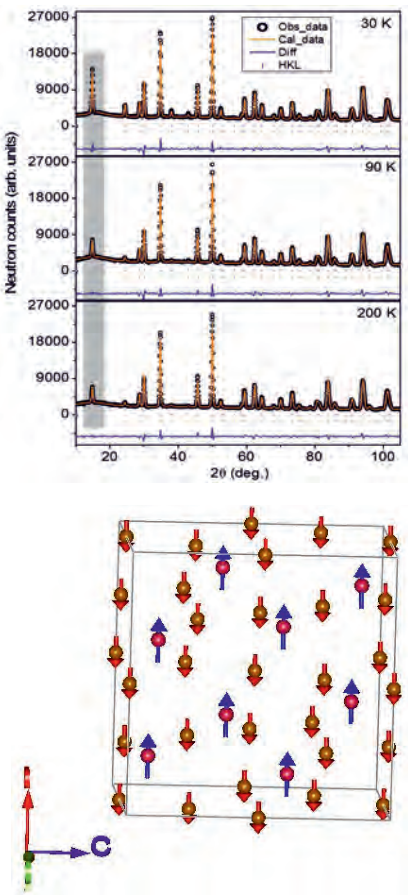

Figure 5: (Top) The Rietveld refined neutron diffraction patterns (measured using the PD-2 diffractometer, Dhruva) for $CoCr_{2-x}FexO_4$ (x = 0.15) showing evolution of ferrimagnetic ordering below 200 K [21]. (Bottom) The ferrimagnetic arrangement of the tetrahedral and octahedral moments along the crystallographic a direction.

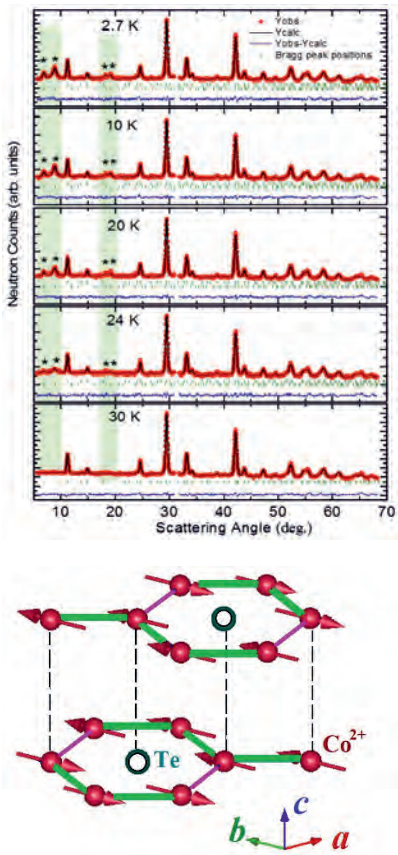

Figure 6: (Top) The temperature dependent neutron diffraction patterns (measured using the PD-1 diffractometer, Dhruva) of 2-dimensional honeycomb lattice magnet $Na_2Co_2TeO_6$. (Bottom) The determined zigzag antiferromagnetic structure at low temperature. The green lines represent the zigzag arrangement of the ferromagnetic chains of Co^{2+} spins. Such spin chains are antiferromagnetically (represented by magenta lines) coupled within the honeycomb planes.

In addition to the ground state magnetic properties, neutron diffraction technique has been widely used at Trombay to study structure property correlations, such magneto-structural, magneto-electric, and magneto-caloric properties in functional materials [12,15,16,19,20]. Neutron diffraction and depolarization studies [6,13,14,21] have been carried out to get microscopic and mesoscopic (domain level) understanding of magnetization reversal phenomenon [22]. Neutron diffraction studies on the atomic site distribution, evolution of structural parameters across the T_C, and microscopic details of magnetic ordering in magneto-caloric materials [7] have been greatly improved the understanding of the role of magneto-structural coupling in achieving high magneto-caloric effect. The structural phase diagrams of pure sodium niobate and its derivatives, a possible highly efficient dielectric capacitor, has been derived using neutron diffraction as a function of composition, temperature, pressure, and particle size [23]. Besides, the neutron diffraction study has revealed the role of site disorder on spin polarization in quaternary Heusler alloy, NiFeMnSn [12], and the presence of ion conduction pathways through polyhedral networks in garnets and layered oxides, leading to their utility in solid oxide fuel cell (SOFC) and battery materials [17-18].

Figure 7: SANS, MSANS and reflectometer (Top panel) instruments at the guide tube laboratory (Bottom panel) of Dhruva reactor. The neutron guides at the guide hall.

Single crystal neutron diffraction is used to understand the molecular conformation, intermolecular interactions, hydrogen bond interactions and hydrogen atom stereochemistry in hydrogen bonded systems as a function of temperatures. Especially, the crystal structures of the potential organic based proton conductors, $(K_{1-x}(NH_4)_x)3H(SO_4)_2$ for SOFC energy conversion devices have been determined using single crystal neutron diffraction. The role of hydrogen bonds (connecting two SO_4^{2-} ions) in facilitating a faster kinetics of super-ionic phase transition in $(K_{1-x}(NH_4)_x)3H(SO_4)_2$ as compared to $K_3H(SO_4)_2$ has been brought out [24]. Single crystal neutron diffraction studies on nickel sulfate hexahydrate crystals grown under different conditions have brought out role of growth conditions on the thermal stability and fragility of the crystals [25].

The local atomic arrangements in amorphous materials, glasses, and disordered-crystals have been investigated using the high-Q-diffraction technique, specially the structural characterization of lead phosphate glasses that are good candidates for radioactive waste loading in nuclear technology. Neutron scattering study reveals that doping of Fe helps in cross linking the poly phosphate chains in these glasses. Thus, increasing Fe content makes 3D connected networks thereby increasing the structural rigidity and improving the leaching properties. Recently, using various experiments including high-Q diffraction on highly irradiated graphite samples from the CIRUS reactor at Trombay and *ab initio* simulations, various 2-, 3-, and 4-coordinated topological structures in defected graphite have been identified, and a microscopic mechanism of defect annihilation on heating and release of the Wigner energy has been provided [26].

Apart from the structural ordering in atomic length scale, the morphology of the mesoscopic length scale (1 to 100 nm) structures and their correlations are also often crucial for many of the physicochemical properties of materials. In fact, structures in mesoscopic length scales are ubiquitous not only among naturally available materials but also for various synthetic materials, including micelles, polymer, precipitates, nanoparticles, porous materials *etc*.

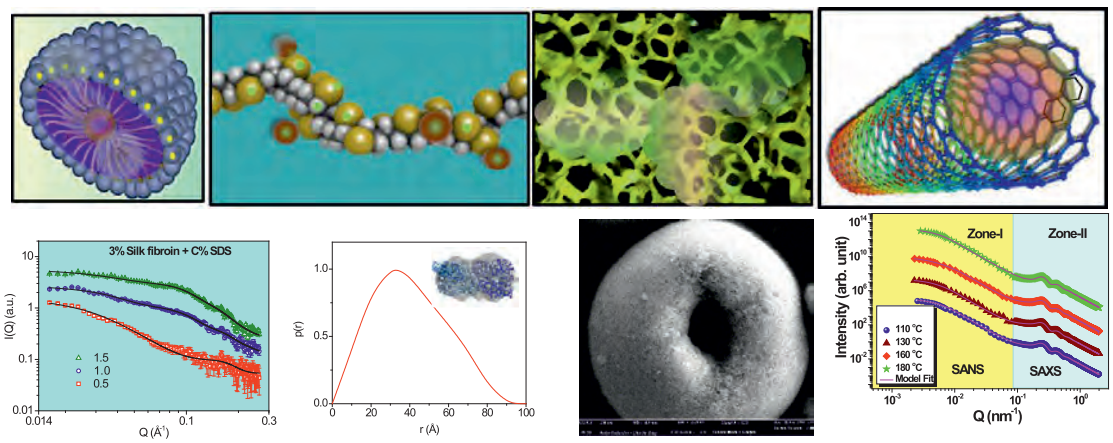

Figure 8: Top Panel: Typical examples showing the mesoscopic length scale structures in materials, such as micelles, polymer, porous materials, nanotube *etc*. Bottom panel (Left to right): SANS-1 data from gelation of Silk Fibroin by Sodium Dodecyl Sulfate (SDS) and model fit for the hydrogel [30].The shape analysis of mosquito-larvicidal BinAB toxin with the receptor (Cqm1) protein [32]. Nano-structured spray dried microgranules and their small-angle scattering (SANS-2) data [33]. Scattering experiments determine the morphology of the granular structure with varying drying temperature. Such granules are found as potential candidate for filtration, bio-remediation and electro-responsive applications.

Probing such large-scale structure demands the measurement of scattering intensity at small angular regimes (typically 0.01 - 5°). This is because of the fact that in a scattering experiment one works in an inverse or reciprocal space rather than the normally perceivable space (known as 'real space'). Accessing scattering intensity at very small scattering angle domain indeed remains a challenge due to the effect of the angular divergence of the direct beam itself. The technique that is used to probe the mesoscopic structures is known as small-angle neutron scattering (SANS). This technique provides the information about the size, distribution and correlation among the various inhomogeneities in materials. The shape of the particles can also be inferred from the functionality of SANS profile. When it comes to the measurement of such mesoscopic length

scale at the surface of the thin film/multilayer materials, the neutron reflectivity technique is used. Such reflectivity measurements provide the surface information of the materials, such as multi-layers and coatings. At Dhruva guide tube laboratory, two SANS (SANS and MSANS) and one reflectometer [Fig. 7] instruments have been operating.

The SANS-I facility [27] is suitable to study materials in the length scale from 1 to 30 nm, covering a variety of soft matter (e.g. polymers, amphiphiles, nanomaterials *etc*.) and biological systems. The technique is particularly useful to study hydrogenous structures as well as nanoscale multi-component systems [28-30] due to the unique advantage of easy possibility of the contrast variation. This advantage arises from the fact that the scattering length is negative for hydrogen and positive for deuterium, which enables the variation of contrast simply by partial deuteration. SANS is widely used for solution structure studies of bio-macromolecular complexes. SANS-I facility has been used to study the interaction of mosquito-larvicidal BinAB toxin with the receptor (Cqm1) protein [31]. SANS-2 (MSANS) facility [32] can probe the size ranging 20 nm to 500 nm and is primarily used for studying microgranular materials, ceramics, cements, porous fibre materials, rocks, alloys etc. [33-37].

Polarized neutron reflectivity (PNR) is a non-destructive technique used to determine the depth dependent structure and magnetic properties of multilayers. The reflectometer at Dhruva has been widely used for structural and magnetic characterization of thin film samples of technologically important hetero-structures [38-41] of Ni/Ti, Ni/Al, Fe/Pt/Cu *etc*. Recently, interface induced magnetization in Gd/Co heterostructures [Fig. 9] has been studied using polarized neutron reflectivity. Interface structure and morphology tend to play important roles in magnetic properties of such systems. Figure 9 shows a comparison of macroscopic magnetization, and magnetization at the interfaces of a pristine Gd/Co multilayer and the multilayer annealed at 400 °C for 0.5 hr. PNR data suggested alloying at interface and reduced magnetization for interfacial alloy layer at both the interfaces (Co/Gd and Gd/Co) [41].

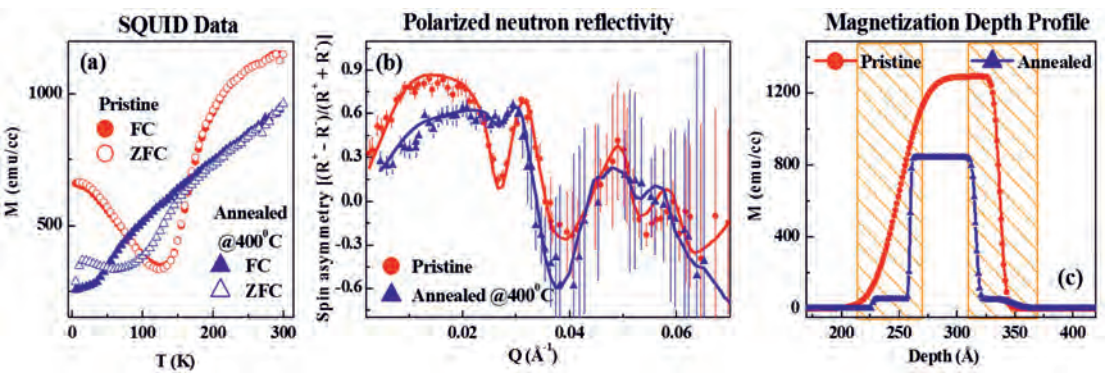

Figure 9: (a) Magnetization (M) data from as-deposited (pristine) Co/Gd multilayer and the multilayer annealed at 400 °C. (b) polarized neutron reflectivity (PNR) data from the multilayer and (c) magnetization depth profiles at two interfaces obtained from PNR.

After the illustrations on structural characterization of materials using neutrons, let us focus on the capability of neutron to probe the dynamics in condensed matter. Determination of the characteristics of the movement (dynamics) of the constituents (atoms and molecules) is important to understand the behaviour under various thermo-dynamic parameters such as, temperature, pressure *etc*. Thermal neutrons with energy (meV) of the order of excitations

(movement) of atoms/ molecules in condensed matter are a unique probe for such microscopic investigations. The time scales covered range from 10^{-11} (rotations) to 10^{-13} (lattice vibrations) seconds. Neutrons may either lose energy to or gain energy from the sample under investigation. Hence these processes are referred as 'inelastic", as opposed to the "elastic" scattering events (that provides structural information, as mentioned above). Inelastic neutron scattering is a technique complementary to spectroscopic techniques like Raman scattering, infrared spectroscopy and NMR spectroscopy. Such inelastic neutron scattering measurements are conventionally carried out by determining the wavelength of the neutron, or by ascertaining the neutron time-of-flight method.

The applications of neutron spectroscopy encompass many materials, such as minerals, inter-metallics, magnetic, semiconductors, catalyst, soft matter including biological systems (polymer, proteins, *etc*.) to determine properties (phonon dispersion relation, phonon density of states) related to thermodynamics, phase transitions, leading to knowledge about inter-atomic bonding, interlayer bonding, magnetic excitations, *etc*.

Triple-axis spectrometer at Dhruva (Fig. 10) is equipped to measure phonons (quantum of periodic lattice vibrations) in functional materials showing anomalous thermal expansion, multiferroic compounds, alloys, *etc*. Material compression with increase in temperature (negative thermal expansion, NTE) and material expansion with increase in pressure (negative linear compressibility, NLC) are unusual phenomena of interest both for fundamental research as well as applications like temperature detectors and shock absorbers. The origin of NTE, was understood by temperature dependent inelastic neutron scattering [42] (Fig. 10) in metal–cyanide framework compound, $KMnAg_3(CN)_6$, an NTE material. Such peculiar behaviour is found to arise from low-energy phonon modes involving the folding of Mn–NC–Ag–CN–Mn linkage about Ag atoms.

Neutron can also provide useful information about the stochastic dynamics in materials [43-44]. The technique used for this purpose is known as quasi elastic neutron scattering (QENS). Quasi-elastic spectrometer at Dhruva (Fig. 11) has been employed to study random motions of molecules inside the pores of a porous material, biological membranes, and vesicles as well as dynamics in micelles and diffusion in lipids. Recently, the change in the dynamics of metallosome with and without incorporation of cholesterol was studied [43] by QENS (Fig. 11), using MARX spectrometer, Dhruva. The study showed that cholesterol acts as a stiffening agent.

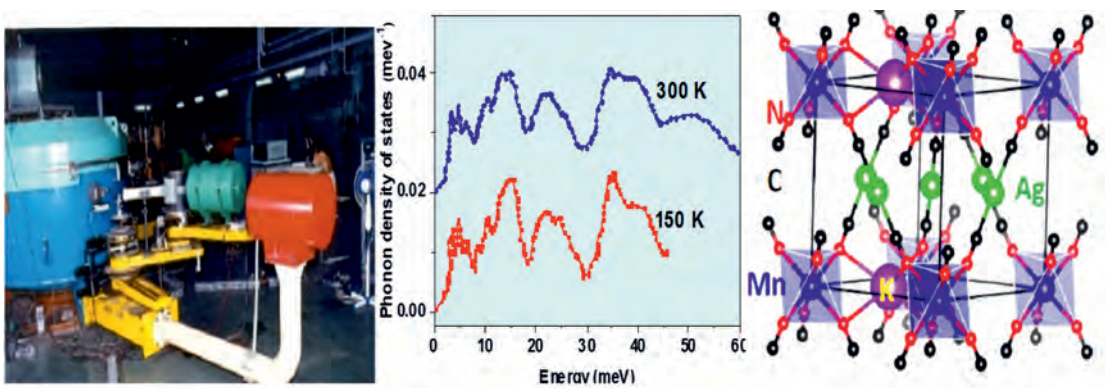

Figure 10: Left to right: Triple axis spectrometer. Structure of $KMnAg_3(CN)_6$, a NTE material. Experimentally measured temperature dependent inelastic neutron scattering spectra [42] of $KMnAg_3(CN)_6$.

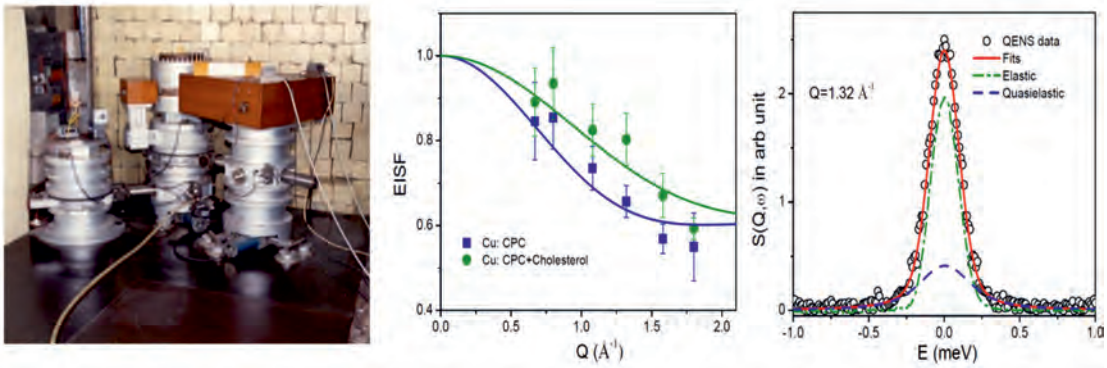

Figure 11: Left to right: Quasi-elastic spectrometer at Dhruva. Typical fitted QENS data at momentum transfer Q = 1.32 Å^{-1} with the model scattering law S(Q, ω)-A(Q)δ(ω)+[1-A(Q)]L(Γ,ω). Elastic Incoherent Structure Factor (EISF) for metallosome with (f:0) and without(f:0.5) cholesterol in that ratio.

It is needless to say that detection of neutrons is challenging as neutron does not have any charge. For all the facilities mentioned above, the detectors including the position sensitive detectors are also developed at Solid State Physics Division, BARC. Such detectors are also supplied to various institutes in the country. In addition to standard neutron counter and 1-dimensional position sensitive detector (PSD), research and development of two-dimensional position sensitive neutron detectors are underway.

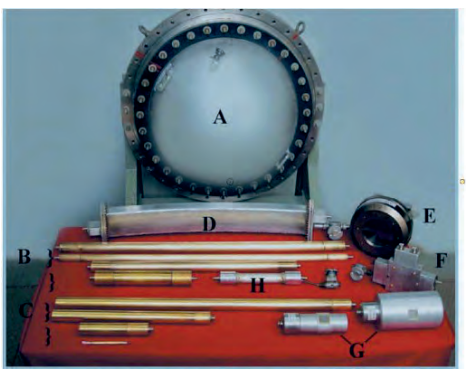

Figure 12: Various neutron detectors developed at SSPD, BARC, A) prototype 2D detector,B,H) Linear position sensitive detectors, C,G) Proportional counters, D) Curved detector, E) Multi-wire 2D PSD, F) Micro-strip based neutron PSD.

In brief, neutron scattering technique is unique owing to the special properties of neutrons and it provides the crucial information on structure and dynamics in materials. A strong and focussed neutron scattering activity, encompassing interdisciplinary fields, is going on at Bhabha Atomic Research Centre based on the indigenously developed facilities at the Dhruva reactor involving single crystal diffraction, powder diffraction, small-angle scattering, high Q diffraction, reflectivity, in-elastic and quasi-elastic scattering. A wide variety of technologically relevant materials, including ceramics, alloys, glass, magnetic, biological, porous, nano-structured materials are being investigated by these facilities. Under NFNBR, a large number of user communities from Universities and institutes, all over the country, use these facilities apart from the dedicated in-house research. In addition to the existing Dhruva based neutron scattering

facility, a newly proposed high flux research reactor (HFRR) [45] at BARC, Vizag will be a new horizon for neutron scattering activity in the country which was initiated almost six decades ago. The upcoming high-flux neutron facility will open up possibilities for more sophisticated neutron scattering experiments enabling scientific breakthroughs related to novel materials, energy & its storage, health, environment addressing some of the most important societal challenges.

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