Applications of Neutron Beam Technology

A. Introduction to Neutron Beam Methods

A.1. Historical Developments and Key Milestones

1. Neutrons have played an important role in the characterization, development and testing of materials over the past 60 years. Neutrons interact with different materials in very different ways and by careful measurement of the transmitted and scattered neutrons the structure and dynamics of materials can be characterized in a unique way to gather important and sometimes unique information about the materials under study. The 1994 Nobel Prize in Physics [www.nobel.se], awarded to Clifford G. Shull and Bertram N. Brookhouse, for example, is the reflection of recognition of the impact of neutron scattering.

2. In the early days, the utilization of research reactors was closely linked to the development of the nuclear energy industry as an enabling tool to gain nuclear expertise. However, emerging applications have driven the evolution of neutron beam technology to tackle increasingly diverse and intricate challenges. The development of secondary sources - hot and cold neutron sources - is an outcome of the need and realization of the power of neutron beams in the field of science and technology. It is now possible and becoming common to insert a cryogenic cold neutron source into the reactor reflector tank. This typically consists of a vessel of several litres of liquid hydrogen or deuterium, which will produce long wavelength neutrons (0.5 nm to 2 nm and sometimes longer) which are much sought after in research for studies on soft matter, such as polymers and biological species. It is also possible, but less common, to insert a 'hot neutron' source into the reflector tank, typically consisting of a thermally isolated graphite chamber, which will produce short wavelength neutrons (less than 0.1 nm) which are valuable for the study of disordered solids and for liquids.

3. Neutron beam science took a major step forward with the commissioning of the first purpose built reactors in North America and Europe in the late 1960s. The High Flux Beam Reactor (HFBR) at the Institut Laue-Langevin, Grenoble, France, for example is an international venture. It has become the benchmark for neutron beam science facilities and has been host to a number of innovations in neutron beam technology leading to the further development of applications of neutron beam technology.

A.2. Neutrons as a Probe: The Basic Properties

4. The key properties of thermal neutrons which make them important as a probe are that: i) neutrons are electrically neutral and so can interact directly with the nucleus of atoms; ii) they have a magnetic dipole moment; which make them an ideal probe for magnetic materials iii) their energy can be readily adjusted in the range 200 MeV – 500 MeV, which is comparable with the energies of excitations (vibrational, rotational and diffusional motion) in matter; and iv) their wavelengths can be readily adjusted in the range 0.05 – 2.0 nm, which spans the range of atomic and molecular spacings in condensed matter. In consequence, neutrons will often provide information which is complementary to that obtained from X-ray and electron spectroscopy studies. For example, in biological sciences, hydrogen and deuterium can be selectively exchanged to functionally distinct molecular sites and thus it is possible to tell by differences in the observed neutron scattering which sites are active and which are not in certain bio-chemical reactions. Neutrons are also ideally suited to in-situ measurements in materials in extreme environments of temperature, pressure or magnetic/electric fields.

A.3. Current Status of Neutron Beam Utilization

5. Currently there are more than 270 operational research reactors world-wide. Over 100 have a thermal power greater to or equal to 1MW and thus have the potential for effective beam line application. Many well-known research reactors are overbooked, some are upgraded, new reactors are under construction and with two major new spallation neutron sources under construction the prospect for further neutron beam applications is promising [1]. Nearly sixty years after its birth, the field of neutron beam applications is expanding in both breadth and depth. Modern neutron beam research reactors are purpose designed and built to maximize potential for scientific experiments. There are also a number of smaller university based or national research reactors of low or medium flux and with low or limited applications base. These reactors serve as teaching tools and assist in the maintenance of nuclear expertise. Although it is recognized that some research reactors are under-utilized, there is nonetheless growth in the industry to meet ever increasing world-wide demand for access to neutron beams to address the more difficult challenges of scientific research.

6. The following sections describe the contribution of these interactions to science and technology in the fields of neutron scattering, neutron radiography, neutron activation analysis, and emerging and future trends. New emerging trends are covered by many IAEA technical meetings on neutron beam applications [2].

B. Neutron Scattering

7. The application of neutron scattering spans many branches of science, for example physics, chemistry, materials science and life sciences and also many branches of engineering research and development. The following sections describe some of the applications that may be broadly appreciated for their industrial, societal or other potential benefits.

B.1. Atomic Structure Determination for Materials Design

8. Most monochromatic neutrons leave the sample with unchanged energy (elastic scattering) and a preference for certain directions (diffraction). By counting the neutrons in a rotatable detector, a diffraction pattern is obtained which shows the relative positions of the atoms in the sample. Neutron diffraction is often used for advanced materials characterization, for example in mixed metal oxide systems where detailed knowledge of the role of oxygen is required, such as high temperature superconductors, colossal magneto-resistance manganates, ferroelectrics etc., extending and complementing the use of X-ray diffraction.

9. Neutron diffraction can reveal the response of structural ceramics to applied stress as in Ceria doped Zirconia (Ce-TZP), which is a ceramic that displays the ability to resist thermo-mechanical stress to a far greater extent than most materials. Neutrons reveal the two mechanisms responsible for this: ferroelastic switching, whereby the crystallites are reoriented when compressive stress reaches the critical value (~1.2 GPa) reducing their length in direction of the stress, and the second is by transformation from tetragonal to monoclinic symmetry, further reducing the length in the stress direction. Neutrons also revealed that these mechanisms are largely reversible, that is when the stress is removed the crystallites will largely return to their original state [3].

B.2. Studies of Material Response to Extreme Environments

10. An emerging application is in fast reaction kinetics, which studies responses in real time even under extreme environments of temperature, pressure, magnetic or electric fields, to understand what is driving the reaction. In order to explore possible clean (pure) and economical synthesis methods, a real time study of combustion synthesis of the refractory material Titanium silicocarbide (Ti3SiC2), using neutrons was performed. Titanium silicocarbide is a unique ceramic in that it possesses ceramic and metallic characteristics meaning that it is suited to both mechanical and electrical applications. For example, it exhibits high temperature stability, high electrical and thermal conductivities as well as having a moderate resistance to oxidation. It has high fracture toughness at high temperatures and can be machined using hardened steel tools. Neutron scattering clearly showed the steps that occur in the recrystallizing in the form of Ti3SiC2 – some of which were completely unanticipated, but which must be understood to refine synthetic process [4]. In this experiment the pre-cursors Ti, C (graphite) and SiC are rapidly heated inside a furnace in the path of a neutron beam. The diffraction patterns (300 milliseconds/pattern) clearly show some phase transitions to intermediate phases, and then at a critical temperature (~900° C) exothermic reactions in the sample produce instantaneous self heating to a temperature in excess of 2 000° C, at which point the material self destructs immediately recrystallizing in the form of Ti3SiC2, which has many potential industrial applications.

B.3. Hydrogen in Metals and Hydrogen Storage

11. The fundamental and technical challenges associated with a hydrogen based energy economy are substantial at all stages of the cycle - hydrogen generation, storage, transport and energy conversion. Because of the sensitivity of neutrons to hydrogen, the use of neutron beams to investigate potential hydrogen 'energy' materials is emerging [5]. Current research is involved in the investigation of potential hydrogen storage materials, such as nickel metal hydrides, alanates (compounds that contain aluminium, (stored) hydrogen, and a metal like sodium or lithium) and more exotic materials such as nano-carbons and metal organic framework lattices.

12. There is much interest in fuel cell development, particularly in polymer electrolyte membrane cells and the ceramic oxide based varieties. Many of the challenges of hydrogen generation, for example by water-splitting, involve materials design challenges in titania based and zirconia based materials. Alanates can release hydrogen at temperatures close to 353K, the temperature at which a so-called Proton Exchange Membrane (PEM) fuel cell would extract energy from hydrogen in a car, for instance. Neutrons are an ideal instrument to observe the stored hydrogen and the release mechanisms in compound materials.

B.4. Magnetism and Superconductivity

13. Most knowledge of the magnetic structure of materials comes from neutron diffraction. The technique has revealed magnetic order across a broad spectrum of magnetic phenomena, ranging from simple metallic magnets, soft and hard magnets such as ferrites, and the modern high performance Neo-max (Nd2Fe14B), which is the permanent magnet of choice for high performance electro-magnetic devices, such as compact electric motors. The scope also includes a wide range of exotic magnetic systems such as antiferromagnets, where the orientation of magnetic moments is complex, and two-dimensional and even one-dimensional. There is also widespread activity in materials where the interplay between magnetic and electrical properties offers the promise of powerful microelectronic devices, e.g. colossal magneto-resistance (CMR) compounds.

14. In the field of research and development of superconductivity, neutrons have provided many insights into structure and function. The superconducting behaviour of high temperature metal oxide

superconductors strongly depends on the concentration and position of oxygen atoms in the structure, which has been revealed by neutron diffraction.

B.5. Excitations and Dynamics

15. Neutron inelastic scattering has traditionally been used to determine collective excitations translational motions of atoms in solids phonons (a quantized mode of vibration occurring in a rigid crystal lattice, such as the atomic lattice of a solid) thus revealing the underlying drivers of phase transitions etc., and also molecular vibrational or librational motions and diffusional motions. This can lead to rare and valuable insights into materials properties, such as proton transport mechanisms. One key application is in magnetic transport spin waves, helping to understand how magnetism can be shared and/or transmitted.

B.6. Structural Biology and Biotechnology

16. Biologists have a direct interest in the study of molecules and molecular assemblies at low resolution, such as proteins in solutions, viruses, liposomes, and also protein complexes with, for example, polyelectrolytes such as DNA. Neutrons are unique, as they are able to determine the structure and dynamics of biological macromolecules and their complexes. The similar scattering signal, using small angle neutron scattering (SANS) from deuterium, carbon, nitrogen and oxygen allows the full determination of the positions and dynamics of the atoms in biological structures. The non-destructive method using neutrons is an ideal complementary tool of investigation if combined with X-ray scattering and/or nuclear magnetic resonance (NMR) measurements.

17. Contemporary research areas in biology and the life sciences include membrane biophysics, drug delivery systems and pharmacology, dental and medical composites, fillings and implants. In enzyme catalysed processes, protein folding and denaturation due to pH balance and temperature changes can be revealed to enable a further understanding of the relationship between structure and function. The complementary application of neutrons using neutron single crystal diffraction has directly revealed the location of hydrogen in myoglobin, a protein in heart muscle.

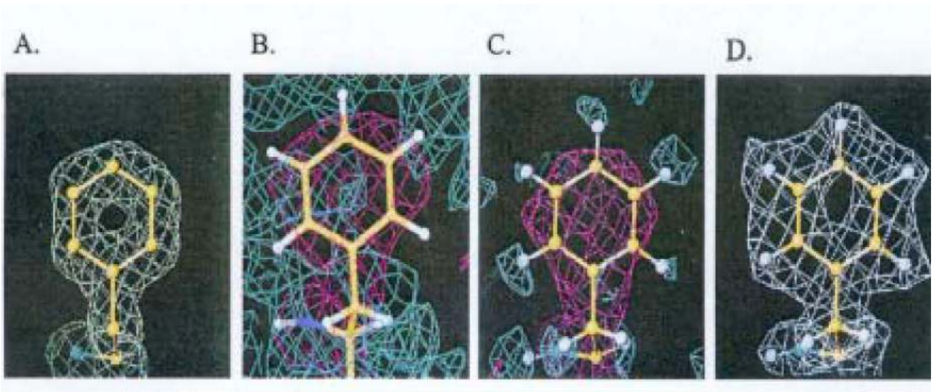

Figure 1. Locating the hydrogen atoms in a residue of myoglobin, (A) using X-rays, (B) using neutrons and an unlabelled sample, (C) the calculated structure equivalent to (B), and (D) using neutrons and a sample where hydrogen has been fully replaced by deuterium [6].

B.7. Nanomagnetic Materials for Nanotechnology

18. Neutron scattering can play a unique role in the field of nanomagnetic materials. The physical and magnetic properties of magnetic materials can change drastically when they are reduced to small particle sizes. Recent studies of nanomagnetic particles have shown some intriguing evidence of

macroscopic quantum magnetic tunnelling, which may shed light on the scalar limits of quantum mechanical processes. Nanomagnetic materials are of great interest in the magnetic recording industry in the never-ending search for faster, more compact and more reliable magnetic recording media.

B.8. Polymer Science and Engineering

19. Polymers are long chain molecules that play an important role in biotechnology, medicine and nanocomposites. The physical and chemical properties of polymeric materials are determined by their nanostructure. SANS can be used to study crystallization kinetics, polymer blend structure, and response to shear forces [7]. New materials derived from crops are expected to revolutionise the plastics industry by removing the packaging waste problem. The rates of degradation of these new polymer materials are of crucial importance and may be effectively studied by SANS. For example, SANS revealed the response of polystyrene to the conditions of the extrusion process. It is through such understandings that polymer mechanical properties can be optimised.

B.9. Residual Stress Measurements for Engineering and Industrial Applications

20. Residual stress measurements using neutrons provide a sensitive tool for determination of strain on the atomic level. By analogy with the more common X-ray residual stress method, the technique is characterized by the high penetration depth of the neutron beam, making it possible to measure residual stress deep inside industrial components. The information obtained underpins knowledge of materials strength, fracture toughness, creep resistance and provides early warning of potential failure, which is a vital application in for example, aero-space (turbines and stress points); transport industry (rails, gears) and pressure vessels, including pipelines [2].

C. Neutron Radiography

21. A unique characteristic of neutrons is that they “see” the nuclei, rather than the diffusive electron cloud, which is seen by X-rays. Neutron radiography uses this aspect to view light atoms (e.g. hydrogen) in the presence of heavier ones. As neutrons are neutral and deeply penetrating, neutron radiography is a non-destructive evaluation technique which possesses certain unique features that distinguish it from photon radiography. The interaction of neutrons with matter is governed by nuclear, rather than electronic characteristics of the medium, and is complementary to X-ray radiography.

22. Neutron radiography has many industrial applications, as for example, in motor industries for inspection of running engines. The main application remains, however, in nuclear materials inspection for examination of irradiated nuclear fuels and power reactor components.

23. New detector developments including real time dynamic imaging technique using devices like Charge-coupled Device (CCD) cameras make neutrons an even more versatile instrument of investigation of materials. Some of the recent developments can be found in the conference proceedings [8, 9].

C.1. Nuclear Fuel Technology

24. Neutron radiography is used for non-destructive characterization of nuclear fuels both freshly fabricated and post-irradiation (Post Irradiation Examination known as PIE). Characteristics that can be examined include distribution of fissile isotopes, compositional changes in the fuel, physical integrity of the fuel pellets, dimensional measurement and changes occurring in the cladding materials and other reactor components. This technique has been used for characterization of zirconium hydride blisters detection of hydrogen in zircaloy. It has also demonstrated its usefulness in the non-destructive characterization of nuclear fuels containing enriched uranium and plutonium for determination of the homogeneity of the fissile material. Both thermal and fast reactor fuel pins have been examined to study the performance of the fuel in the reactors. In the context of advanced fuels for new reactors, neutron radiography has an important role to play in the development and characterization of nuclear fuels.

C.2. Aerospace Industry

25. Inspection of turbine engine blades, adhesives in metallic honeycomb structures, monitoring of corrosion in aircraft wings and inspection of pyrodevices used in space launch vehicles has been carried out by neutron radiography for the non-destructive evaluation of these critical components. Neutron radiography provides important information about the proper loading of pyrocharges (as used in airbags) and investigation of aircraft parts as, for example, honeycomb structures, which are used in aerospace industries as the preferred core material for buckling and bending sensitive sandwich panels and structures.

C.3. Biomedical and Agriculture

26. Neutron radiography has also found its uses in biomedical and agricultural applications. Distribution and transport behaviour of neutron absorbing elements such as gadolinium, samarium and cadmium in leaves, the effect of exhaust gases on the growth of plants and materials detection of micro-organisms are some of the uses of this technique. The effectiveness of protective coatings used for conservation of wood is studied using neutron radiography. Water movement in plants and soil, which is important for growth studies, has been studied using neutron radiography. In bio-medicine the monitoring of boron distribution in boron capture therapy for cancer, the inspection of heart valves and materials for dentistry are also valuable applications.

C.4. Other Potential Applications

C.4.1. Civil Engineering

27. Detection of water absorption in building materials and use of protective agents in brick samples have been carried out employing neutron radiography [10], which is also used for investigating geological materials, detection of cracks in concrete and detection of pores in rocks. Though non-destructive testing of the concrete is being carried out mainly by ultrasonic techniques, neutron radiography holds good promise for the study of rocks and construction materials.

C.4.2. Ordnance Industry

28. Explosive charges in steel casings are examined using neutron radiography, which has immediate relevance for the detection of explosives in packages.

C.4.3. Real time Radiography

29. Real time radiography has been used for visualization of two phase flow in components, such as reactor vessels in refineries, where study of the flow pattern instability is required to develop thermal hydraulic models. Visualization of the two phase flow and measurement of void fraction have been carried out using neutron radiography.

C.4.4. Cultural Heritage

30. Neutron radiography of various art objects yields important information about the method of fabrication and history of precious art objects.

D. Neutron Activation Analysis

31. Neutron activation analysis has been used for determination of elements by measuring the characteristic radiation emitted by nuclides formed in the material when irradiated with neutrons, and is capable of a high level of accuracy for trace elements. This technique has a variety of applications in agriculture, food and nutrition, forensic sciences, basic research and various industries. Detection of trace elements in materials, characterization of archaeological specimens, and examination of art objects such as paintings have been done using neutron activation analysis. A novel application has been reported in the analysis of forensic samples, where the technique provides a characteristic signature for the samples. Prompt gamma neutron activation analysis has been carried out for assay of low atomic elements such as hydrogen and boron.

E. Emerging and Future Trends

32. Quasi-Laue Diffraction - a diffraction measurement which matches the wavelength and bandpass to the individual requirements – for rapid structure determination in single crystals has emerged over the last decade and speeds up data acquisition for complex crystal structures from months to hours. This method is likely to revolutionize the application of neutron diffraction for determination of small scale chemical structures and catalyse application of the technique to protein crystallography (traditionally an application where X-ray diffraction is commonly used) and will have the greatest impact in understanding the role of hydrogen in functionality.

33. Ultra-SANS, is emerging as an alternative tool to conventional ‘pin-hole’ SANS for large scale structure determination, and could be used for the dynamics of hydration of cement.

34. The next generation of spallation neutron sources are anticipated to open up a new era of ground breaking international scientific achievements, providing enhanced performance of investigations using neutrons. New instrumentation used with these new sources could potentially give a gain in sensitivity up to about a factor of 100 [1]. In order to succeed, this effort must be underpinned by good research reactors to allow testing of new instrumentation and training in enabling knowledge for the big facilities.

35. Advances in imaging and detectors have made it possible to have good resolution neutron radiographs. CCD based detectors are being used for real time imaging of neutrons [11]. Useful information about the nature, location and orientation of the objects is being extracted by neutron tomography techniques. A number of new neutron radiography facilities are also being commissioned with new features. With the development of high resolution detectors and image processing techniques, three-dimensional neutron tomography has become a powerful tool for investigating complicated structures.

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