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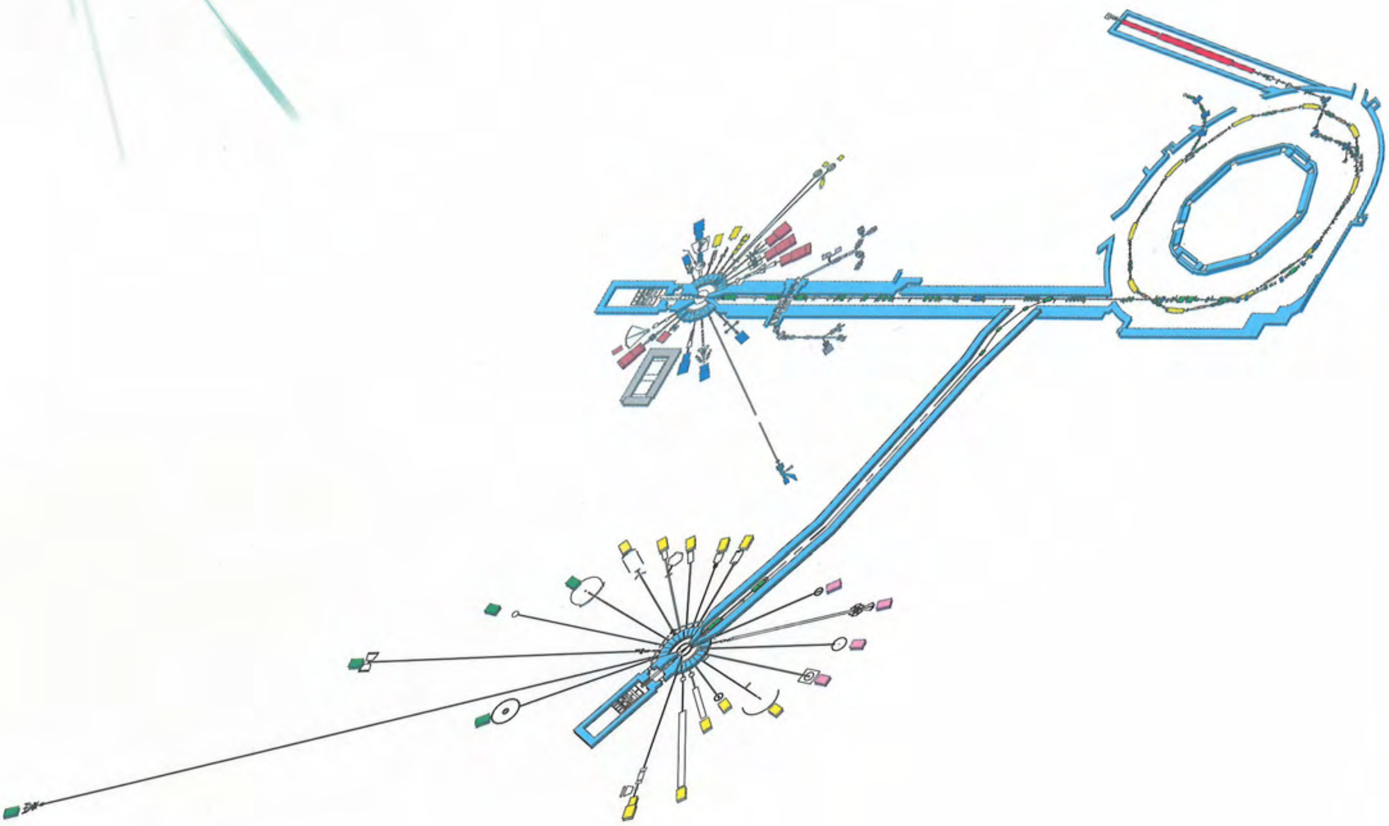
A SECOND TARGET STATION AT ISIS



NEW OPPORTUNITIES FOR INTER-DISCIPLINARY RESEARCH USING NEUTRONS

ISIS

A Second Target Station at ISIS

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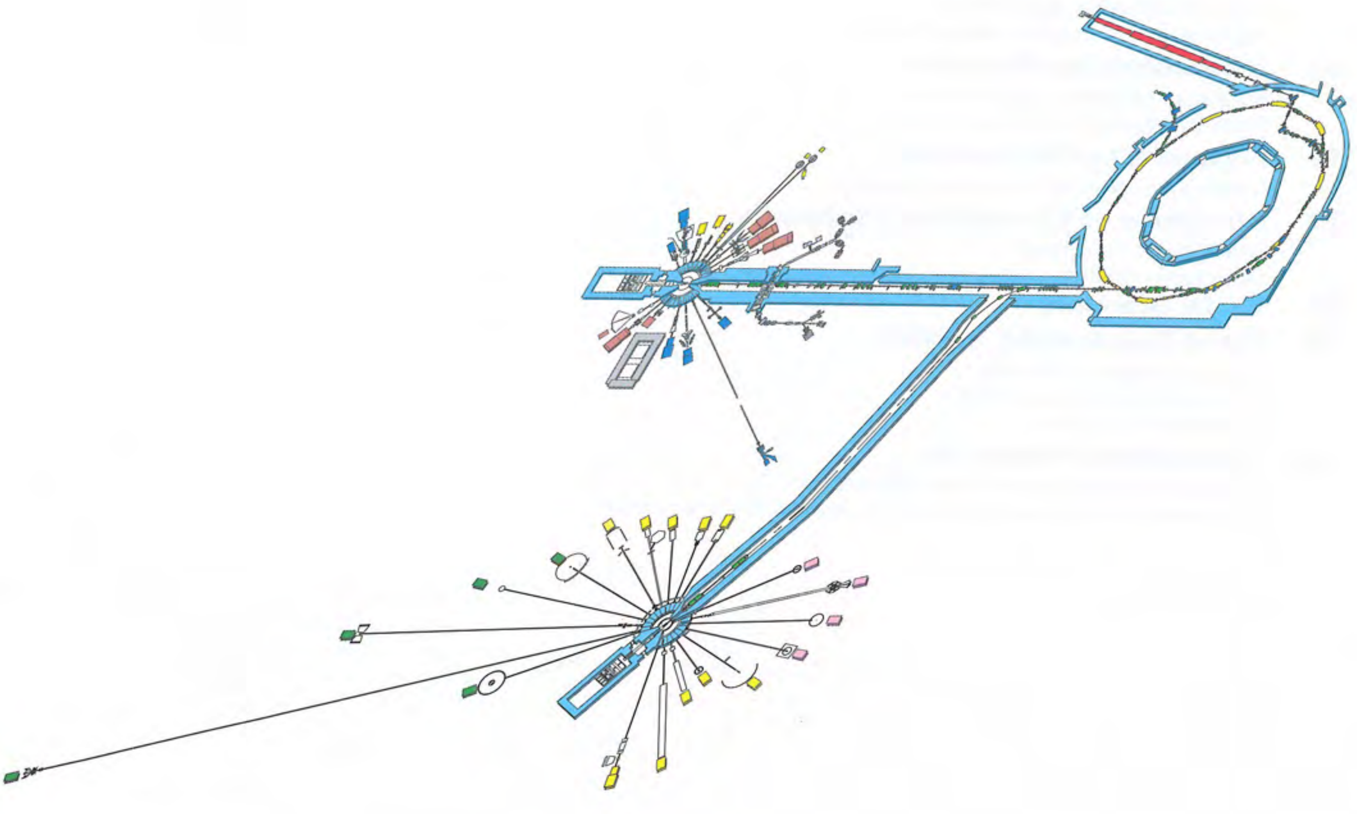
A SECOND TARGET STATION AT ISIS

The proposed Second Target Station on the ISIS pulsed neutron facility at the Rutherford Appleton Laboratory represents a new generation of neutron production target and instrument suite. It will offer unique instrumentation and unrivalled potential for structural and dynamical studies of matter, using cold neutrons and high resolution spectroscopy. Such studies will be inter-disciplinary, with applications predominantly in Soft Condensed Matter, Bio-molecular Sciences and Advanced Materials.



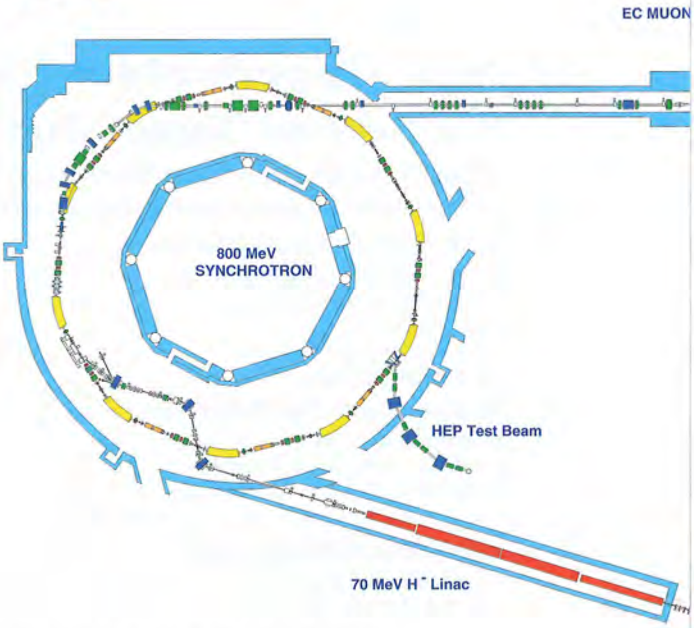
ISIS

CONTENTS

CONTENTS

SUMMARY

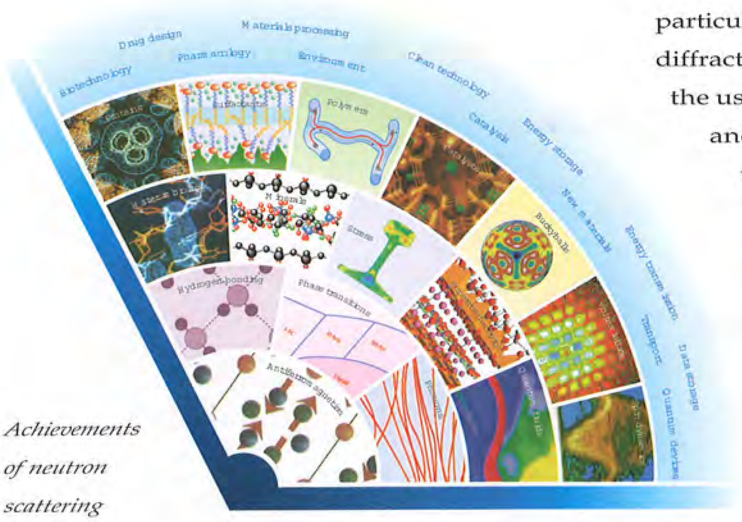
A Second Target Station at ISIS - Summary of Proposal



Introduction

Neutron scattering has made a unique and fundamental contribution to the understanding of the structure and dynamics of condensed matter. Pioneering advances were highlighted in the award of the 1994 Nobel Prize in Physics to Brockhouse and Shull. Much of this early work was based on reactor sources, but in recent years, particularly through contributions from ISIS, the potential of accelerator based pulsed neutron sources has been firmly established.

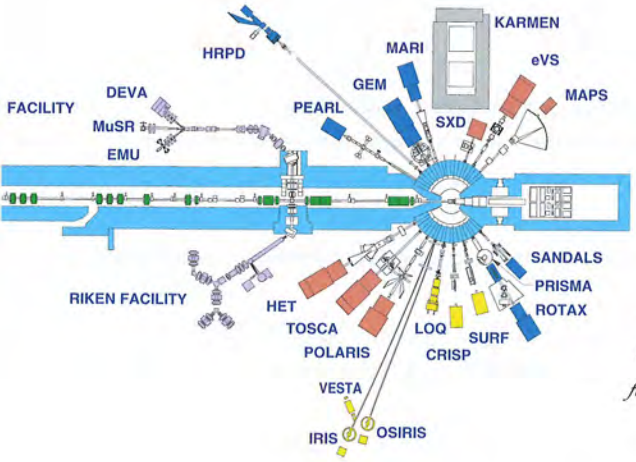
The advantages of neutron scattering studies - selectivity through isotopic labelling, the magnetic interaction, the ability to probe atomic motion, and the penetrability of the probe - are well established and documented. The effective and growing exploitation of ISIS over a broad range of condensed matter research has powerfully demonstrated the specific benefits of the time-of-flight technique in neutron scattering on pulsed sources. Such benefits include improved instrumental resolution, simultaneous wide coverage of both momentum and energy transfer, intrinsically low backgrounds, the ease of use of fixed geometry sample environment equipment, the high fluxes of epithermal neutrons and the broad spectral range. These features, coupled with the advantages of neutrons, have been responsible for the emergence of an impressive research programme, whose breadth covers topics at the forefront of Physics, Chemistry, Materials Science, and, more recently, Earth and Environmental Sciences, Engineering and Biological Sciences.



Achievements of neutron scattering

The developments at ISIS have had a particular impact on high resolution powder diffraction, magnetic inelastic scattering and the use of reflectometry to study surfaces and interfaces. Research at ISIS, using these diffraction and inelastic scattering techniques, has contributed significantly to our understanding of the properties of condensed matter, including polymers, surfactants and proteins at interfaces and in solution, giant magneto-resistance materials, high

SUMMARY



Layout of the current ISIS facility.

temperature superconductors and fullerenes.

The neutron fluxes available at nuclear reactors are close to practical limits. In contrast, recent experience at ISIS has demonstrated that there is still considerable potential for the development of pulsed neutron sources.

Just as the advent of the high flux reactor at the Institut Laue Langevin (ILL), Grenoble, in the early 1970's, with its dedicated cold neutron source, broadened considerably the appeal of neutron scattering, so the development of a Second Target Station at ISIS, optimised for long wavelength neutrons, will have a major impact on the study of complex condensed matter systems. There is very significant potential for making substantial contributions in Soft Condensed Matter, Bio-molecular Sciences and Advanced Materials.

The Potential of a Second Target Station at ISIS

A performance gain of greater than an order of magnitude will be achieved for cold neutrons and high resolution instruments. This will be produced by optimising both the target and moderator configurations on the Second Target Station. Potentially impressive gains in the capability of cold neutron scattering and high resolution studies will provide exciting new opportunities in the technologically significant areas of Soft Condensed Matter, Bio-molecular Sciences, and Advanced Materials.

In the areas of Advanced Materials and Soft Matter there is a distinct trend towards the study of complex, multi-component or multi-phase systems, the use of complex sample environments, and the investigation of non-equilibrium systems. The dimension scales of importance range from molecular to meso-scale. This dictates the need for a broad wavelength range with a particular emphasis on cold neutrons. Kinetic studies (ranging from chemical reactions to probing dynamic surface tension) require the same broad spectral range with higher fluxes of cold neutrons. Multi-component or multi-phase systems are only tractable with the parametric studies possible using enhanced flux and high resolution. The introduction of the Second Target Station at ISIS will provide a platform for such trends and offer unique, world class facilities for the UK scientific community. Significant developments in the scientific areas summarised below can be envisaged.

Adjustments being made on an experiment to study the adsorption of proteins in non-equilibrium conditions.

SUMMARY

Soft Condensed Matter

Surface, interfacial and bulk properties of complex fluids (polymers, surfactants, colloids).

- ***Interfacial studies***: self-assembly and ordering of complex mixtures of surfactants, polymers and proteins at interfaces, with emphasis on kinetic processes and multi-component systems at technologically relevant interfaces (liquid-liquid and liquid-solid), thin film devices.
- ***Processing of soft solids***: relationship between microscopic structure and bulk properties (rheology) in industrially relevant fluid fields.
- ***Self-assembly***: structure of lyotropic mesophases, micro-emulsions and vesicles, with emphasis on dynamics of structural phase changes, association and disassociation, and self-assembly in super-critical fluids.
- ***In-situ electrochemistry.***

Bio-molecular Sciences

Pharmaceuticals, drug delivery formulations, membrane-protein interactions, bio-compatibility and functionality, food technology.

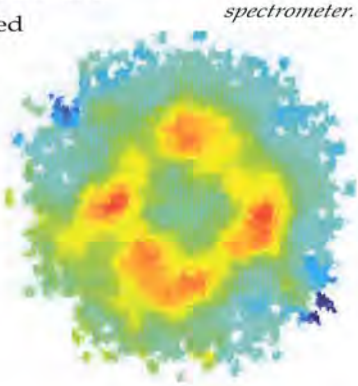
- ***Interfaces and membranes***: structural organisation of membranes and membrane-protein systems.
- ***Macromolecular assemblies***: low resolution studies on macromolecular assemblies, in systems not tractable by high resolution crystallography, viruses, glyco-proteins, protein folding, protein-nucleic acid interactions. Solvent structure. Meso-scale structure.
- ***Pharmaceuticals***: determination of new drug structures, where the role of hydrogen atoms is essential in understanding drug-receptor interactions. Molecular engineering.
- ***Food technology***: study of solvent distribution and structural changes in complex assemblies (for example starches) during processing. Protein fouling. Protein adsorption and colloidal stability. Mechanism of foam and gel formation.

Advanced Materials

Crystalline, magnetic, disordered and engineering materials, including complex inorganic and organic assemblies, clathrates, intercalates, zeolites, nano-structured materials, high temperature superconductors, giant magneto-resistance materials, magnetic films and multi-layers, spin valves, glasses, complex fluids, porous media.

- ***Structural details*** of giant/collossal magnetoresistive materials, non-stoichiometric oxides, piezoelectric, ferroelectric and negative thermal expansion materials.
- ***Composite*** multi-crystalline materials, such as mixed geological phases, magnetoresistive manganates, toughened engineering materials and high T_C materials.
- ***Studies under extreme conditions***, catalysis, in-situ chemical reactions (including electrochemistry and battery function), ultra-high pressures, novel processing routes.
- ***Structure of nano-scale materials***, sol-gel processing, ceramics.
- ***Structure of complex materials***: role of zeolites in ion exchange materials and catalysis, novel electrodes and electrolytes, clathrate formation, nano-structured materials
- ***Determination of new magnetic structures:*** materials with novel ground states.
- ***Magnetic* thin films**, multilayers, spin electronics.
- ***Nucleation and growth* processes**, molecular clustering, glasses.
- ***Complex fluids*** in porous media; oil recovery; waste disposal; supercritical fluids.
- ***Stress/strain mapping*** in engineering materials.

Spin fluctuations in $YBa_2Cu_3O_{6.6}$ taken on the HET spectrometer.



Relevance of Potential Research to the Technology Foresight Programme

The ISIS Second Target Station will be ideally placed to make a major contribution in all the areas highlighted on page 6, many of which are linked directly to the major themes and priorities identified in the last UK Technology Foresight exercise (see table) - a good indication of the relevance of the potential research programme.

Chemicals	*Bio-chemical technology, advanced materials, polymers, processing, sensors*
Energy	*Enhanced oil recovery, waste management*
Materials	*Sensors, advanced materials, adhesion, surface Engineering*
Manufacturing	*Processing, product formulation*
Defence and Aerospace	*Sensors, advanced materials, process technology*
Retail and Distribution	*Intelligent or smart packaging*
Health and Life Science	*Drug delivery, drug creation, pharmaceuticals, immune response manipulation, metabolic pathways*
Food and Drink	*Material changes during processing*
Agriculture	*Pesticides, environmental controls*

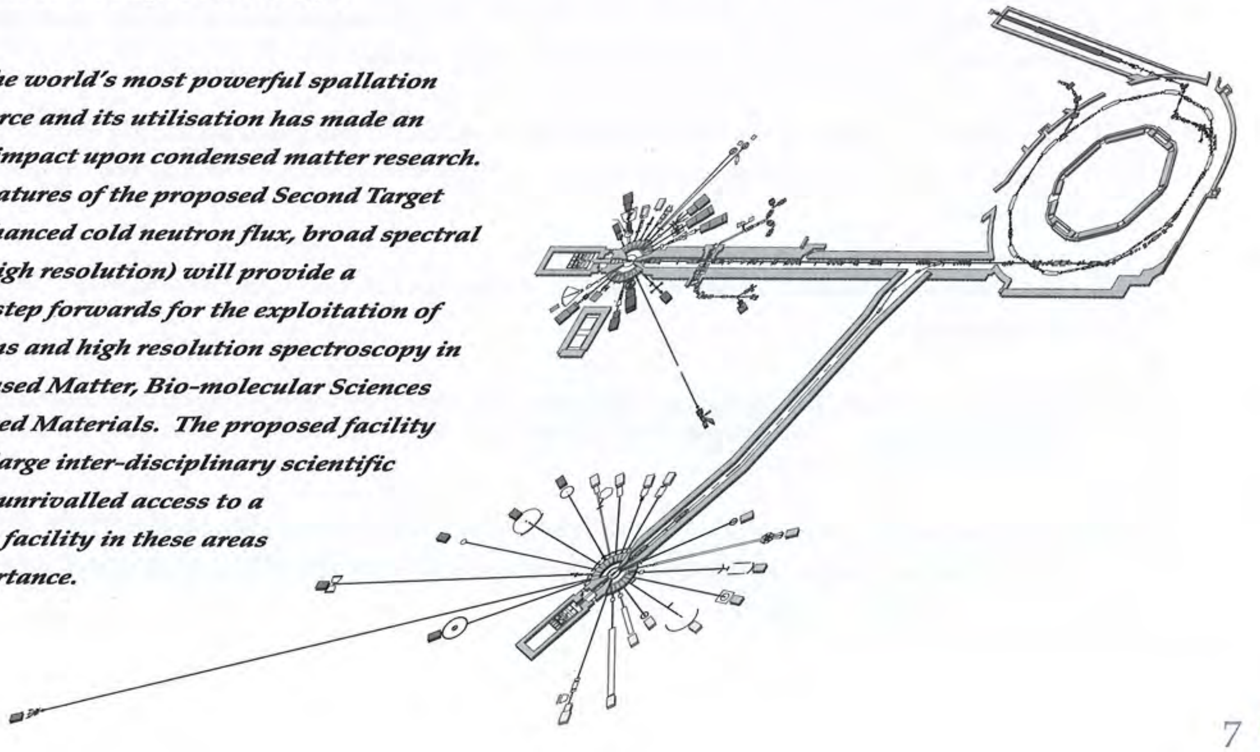
Technology Foresight themes.

Proposed Instruments

The Second Target Station is based on proven designs and technologies. Its construction and commissioning can be completed in three years, assuming an optimal funding profile. The key areas of instrumentation that will benefit most are neutron reflectometry, small angle neutron scattering (SANS), very high resolution spectroscopy, non-crystalline diffraction, high resolution diffraction and large scale crystallography. An imaginative instrument suite to exploit these new opportunities is being developed. Seven key instruments have been identified to exploit the scientific opportunities: a low energy chopper spectrometer, LET; a high resolution powder diffractometer, HRPD-II; a small angle scattering instrument, SANS; a neutron reflectometer, INTER; a single crystal diffractometer, LMX; a high energy resolution crystal analyser spectrometer HERBI; and a toroidal focusing mirror SANS instrument, fm-SANS. Further instrument concepts for the Second Target Station are detailed in the section on instrumentation.

An additional but important feature is that the relocation of instruments most suited to the Second Target Station from the existing target station will provide new opportunities for instrumentation exploiting the characteristics of the High Power Target.

ISIS is the world's most powerful spallation neutron source and its utilisation has made an impressive impact upon condensed matter research. The main features of the proposed Second Target Station (enhanced cold neutron flux, broad spectral range and high resolution) will provide a significant step forwards for the exploitation of cold neutrons and high resolution spectroscopy in Soft Condensed Matter, Bio-molecular Sciences and Advanced Materials. The proposed facility will give a large inter-disciplinary scientific community unrivalled access to a world-class facility in these areas of key importance.



Neutron Scattering in Condensed Matter Science

Neutron scattering provides information at a microscopic level on the structure and dynamics of condensed matter in fields as diverse as Physics, Chemistry, Biology, Materials Science, Earth Science and Engineering. Much of the early impact and success of neutron scattering centred around Condensed Matter Physics: phase transitions, phonons, magnetic structures and hydrogen bonding. The 1994 award of the Nobel Prize for Physics to Brockhouse and Shull was a fitting recognition of their pioneering contribution to this early work in the 1950's and 1960's. Since then, applications of neutron scattering have broadened considerably into Chemistry, Biology and Materials Science. Significant contributions have been made to our understanding at a microscopic level of technologically relevant materials such as polymers, plastics, proteins, liquid crystals, magnetic materials and superconductors.

The unique properties of neutrons as a scattering probe which allows such a broad exploitation are listed in the box below.

Until the mid 1980's the development of neutron scattering was based on steady state reactor sources. The impressive broadening of applications from Solid State Physics to Chemistry, Biology and Materials Science arose primarily from the provision of cold neutrons, especially at reactor sources such as the Institut Laue Langevin, Grenoble. More recently the potential of accelerator based pulsed neutron sources, such as ISIS, has been recognised. The advantages of the time of flight technique on a pulsed source have now been powerfully demonstrated, and this has given rise to an effective and growing exploitation of ISIS over a broad range of condensed matter research.

- **The wavelength of thermal neutrons is similar to atomic dimensions, providing structural information from interatomic bond lengths to macromolecular length scales.**
- **Neutrons are uncharged and interact with nuclei rather than atomic electrons. The scattering cross-section varies randomly throughout the periodic table and between isotopes. This allows us to see light atoms in the presence of heavier ones, to distinguish between neighbouring elements, and to exploit isotopic substitution (contrast variation) to isolate or highlight particular features or components.**
- **The energies of thermal neutrons are similar to those of atomic and molecular dynamics, enabling motions from polymer reptation to molecular vibrations and lattice modes to be probed.**
- **The neutron magnetic moment enables magnetic structure and fluctuations to be investigated.**
- **Neutrons are highly penetrating, enabling the use of complex sample environments; neutrons are also non-destructive, allowing studies of delicate biological materials without damage.**
- **Neutrons perturb the material under study only weakly. This greatly aids theoretical interpretation, making analysis generally straightforward and direct. This usually leads to an unambiguous result.**

Properties of neutrons as a scattering probe.

View of the current ISIS experimental hall.

Particular benefits of the time-of-flight technique on a pulsed neutron source arise from the factors given in the box below.

The present generation of pulsed neutron sources was optimised primarily for thermal and epithermal neutron applications, and have been outstandingly successful for high resolution diffraction and high energy inelastic scattering. Although the current ISIS target station is far from optimum for cold neutrons, the impact of techniques such as reflectometry and high resolution spectroscopy is equally impressive. Furthermore, this first generation of cold neutron instruments (micro-volt spectroscopy, SANS and reflectometry) has clearly demonstrated the potential of cold neutron scattering techniques on pulsed neutron sources. In the particular case of reflectometry, it is on pulsed sources rather than reactors that the technique has been pioneered and where the scientific impact has been the more impressive.

Preparation of micro-emulsion samples for a SANS experiment on the LOQ instrument.

- **The broad spectral range (significant fluxes at wavelengths up to 20 Å), resulting in a simultaneous wide coverage of both momentum and energy transfer.**
- **Improved instrumental resolution over a broad range of length scales and kinematic space.**
- **Intrinsically low background.**
- **High flux of epithermal neutrons.**
- **The ease of use of fixed geometry sample environment equipment.**

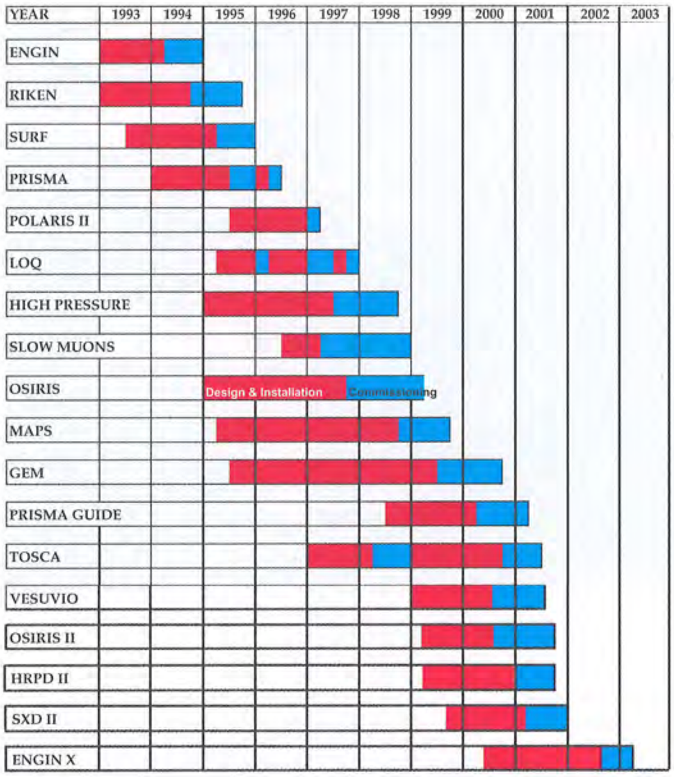
Benefits of the time-of-flight technique on a pulsed neutron source.

Science at ISIS

ISIS is the world's most powerful spallation neutron source. It is designed primarily for the study of condensed matter, using neutrons as a scattering probe. The nuclear spallation process induced in a tantalum target by an 800 MeV proton beam is highly efficient, releasing 30 MeV per neutron compared with 190 MeV per neutron for fission, and hence the target dissipates a modest 160 kW from a 200 μA proton beam at a repetition rate of 50 Hz. The fast neutrons produced in the target are slowed down by a range of hydrogenous moderators to feed currently 20 different neutron scattering instruments. Three different moderators, 20K liquid hydrogen, 100K liquid methane and ambient water, provide different spectral characteristics for a wide range of applications. The target/moderator assembly is 'decoupled'; that is, the neutrons are under-moderated, and retain a sharp pulse structure. Hence the present target station emphasises the thermal / epithermal region of the spectrum, and the 20 millisecond time frame limits band width for cold neutron applications and for instruments requiring long flight paths.

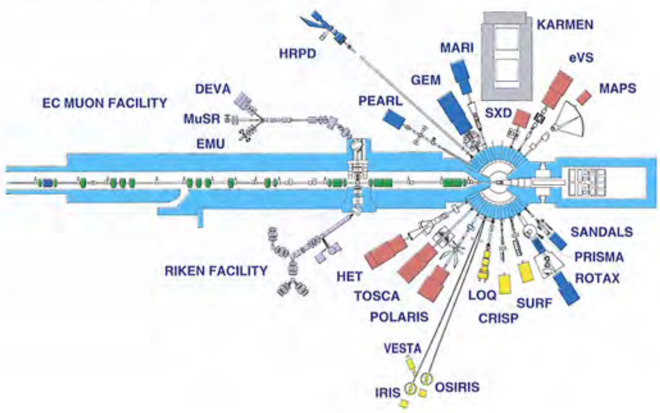
The Present Instrument suite

A suite of instruments for techniques which include inelastic scattering, diffraction, spectroscopy, small angle neutron scattering (SANS) and neutron reflectometry (NR), is used for a broad range of investigations in Condensed Matter Physics, Materials Science, Chemistry, Biology, Earth Science, and Engineering (see instrument summary opposite). The instrument suite has developed substantially over the past 10-15 years, and new instrumentation and instrument concepts



The ISIS instrument development programme over a 10-year period.

have emerged: reflectometry, high resolution powder diffraction and high energy magnetic inelastic scattering, for example. More recently, advances in detector technology, neutron optics, polarisation, data acquisition and computing have given added impetus to the development of improved instrumentation, and a second generation of instruments (SURF, MAPS, GEM, OSIRIS, and TOSCA) has been developed and introduced (see above). These have been funded predominantly through Collaborative Research Grants in the UK, and through collaborations with overseas international partners.



The Present ISIS Instrument Suite

HRPD
High Resolution Powder Diffraction

POLARIS
High Intensity Powder Diffraction

SXD
Single Crystal Diffraction

PEARL
Engineering and High Pressure Instrument

GEM
General Purpose Diffraction, Liquids and Amorphous Structures

SANDALS
Small Angle Liquids & Amorphous Diffraction

CRISP, SURF
Neutron Reflectometry

LOQ
Small Angle Scattering

HET
Excitations at Low Momentum Transfer

MARI
S(Q,ω) Vibrational & Magnetic Spectroscopy

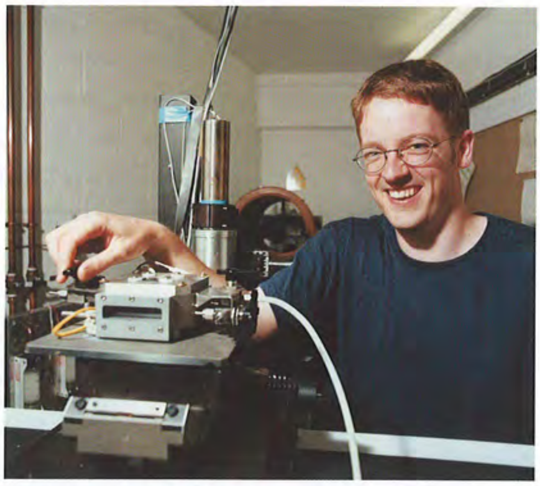
PRISMA
Coherent Excitations and Critical Scattering

ROTAX
Multiple Purpose Diffractometer

MAPS
Single Crystal Excitations

IRIS
Low Energy Spectroscopy Long d-spacing Diffraction

TOSCA
Molecular Spectroscopy and Crystal Fields



Setting up the CRISP reflectometer for studies of water penetration in float glass surfaces.

eVS
Electron Volt Spectroscopy

OSIRIS
Polarisation Analysis Spectroscopy and Diffraction

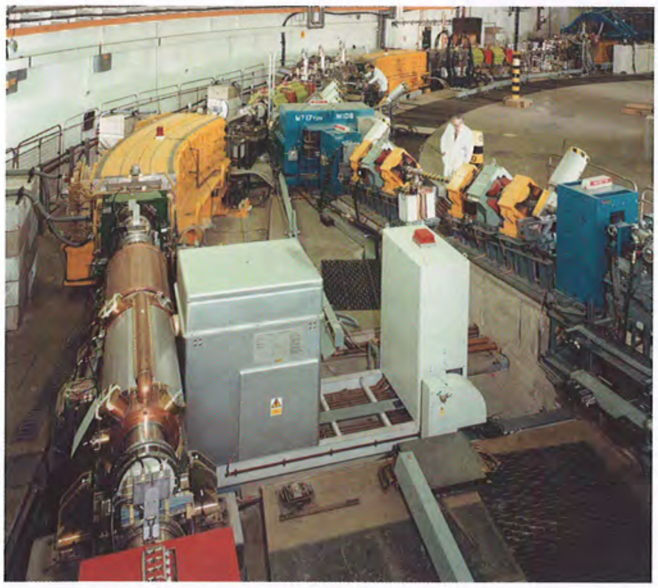
MuSR
Transverse and Longitudinal Field Implanted Muon Spectroscopy

EMU
Implanted Muon Spectroscopy optimised for Longitudinal Field

DEVA
Muon Development Beam

RIKEN
Surface and Decay Line Muon Facility

KARMEN
Neutrino Facility

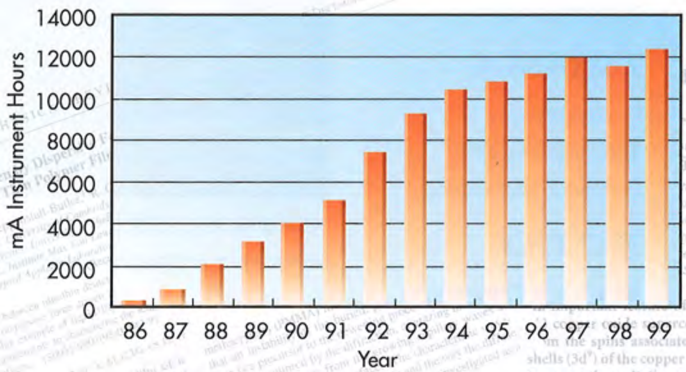


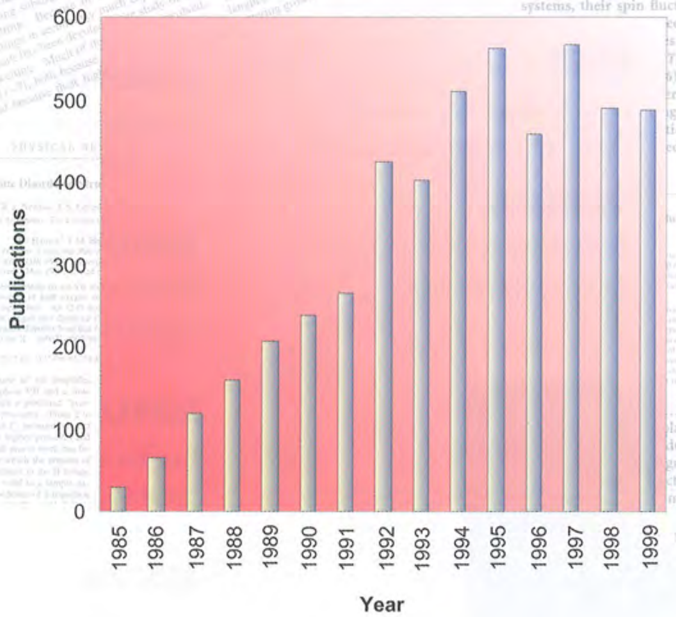
The ISIS synchrotron.

SCIENCE

ISIS Performance

The facility delivers beam for the experimental programme for ~ 170 days per year. Academic users from the UK and 10 other countries with major programmes at ISIS make up the ~1200 scientists who visit annually to perform some 600 experiments. The majority of the instruments are substantially over-subscribed by more than a factor of 2. This international and interdisciplinary research programme has resulted in some 4,000 publications over the past decade - by any measure a vibrant and highly successful programme.





This broad research programme covers a variety of topics at the forefront of Physics, Chemistry and Materials Science, and, more recently, Geology, Biology and Engineering. Notable examples include the determination of the high resolution structures of high T_c superconductors, fullerenes, GMR materials, lithium battery electrodes and negative thermal expansion materials; magnetic excitations in high T_c superconductors and heavy fermions, and excitations in quantum fluids; the behaviour of complex fluids (surfactants, polymers, colloids, proteins) at interfaces and in bulk solution; high pressure studies, both of crystalline materials and super-critical fluids; and determination of the structures of metallic glasses and ceramics. Highlights of this diverse programme are presented in the remainder of this section.

Soft Condensed Matter

Soft condensed matter encompasses a wide range of molecular materials, such as polymers, surfactants and colloids. The physical and chemical properties of these systems are of increasing technological importance, and this is an area often driven by the commercial significance of products which are highly complex mixtures and structures. Although a variety of experimental techniques is used to investigate both bulk and surface properties, the specific advantages of neutron scattering (principally 'contrast', and the 'weak interaction' - see table on page 8) makes the neutron a particularly powerful probe in this area. As a result, soft condensed matter studies using neutrons have grown enormously in recent years. Some recent examples from ISIS involving the structure of polymer films, surfactant adsorption, polymer intermolecular interactions and aggregation (self-assembly) in complex extreme environments are presented.

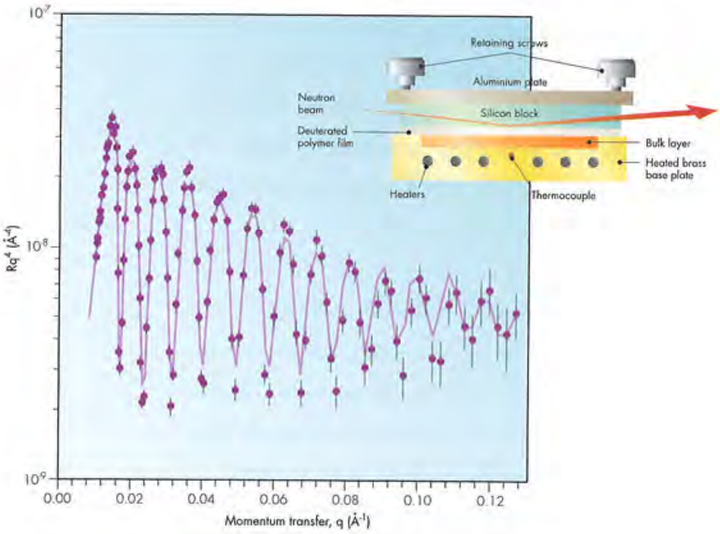


Figure 1. Neutron reflectivity data from the interface of deuterated polystyrene and polyethylene. Inset: schematic of the cell used for investigation of polymer interfaces in the melt.

Polymer-polymer inter-diffusion

Polymer-polymer interfaces are important as they play a dominant role in the nature of polymer blends and in surface wetting, as well as applications such as adhesion, welding and mechanical strength. To understand polymer behaviour in such applications, the interface must be characterised at a microscopic level. In combination with deuterium labelling, neutron reflectivity provides unprecedented detail on interfacial shape and width at molecular resolution. This has been exploited in the study of the interface between a range of immiscible and partially miscible polymers, such as polystyrene-poly(methyl methacrylate). Industrially important polymers, such as polypropylene and polyethylene, are semi-crystalline, and a specially constructed 'hot cell' has been developed to study interfaces involving such polymers, at temperatures above their melting point (figure 1).

The interfacial widths measured by neutron reflectivity are greater than that predicted by theory, due to broadening by thermally induced capillary waves. Capillary waves produce a measured interfacial width which varies logarithmically with film thickness. The cut-off for observing short wave vector waves is determined by the in-plane coherence of the neutron beam for thick polymer films, and by dispersion forces across the film in thin films. This logarithmic dependence has been verified for measurements of the polystyrene – poly(methyl methacrylate) interface, for different polystyrene film thicknesses.

In-situ real time measurements provide the opportunity to measure the early stages of inter-diffusion at a polymer-polymer interface, and open up the potential for studying oligomer and solvent ingress (role of plasticisers). Using a development of the 'hot cell' described earlier, measurement times of <60 s have been achieved. The white beam time-of-flight method on a pulsed source makes such an approach possible, in that the entire reflectivity profile can be obtained simultaneously. The geometry is chosen to match the region most sensitive to the interfacial width, and the resolution relaxed to match the length scales in the bilayer. The variation in interfacial width and position of the interface for oligomeric styrene diffusing into high molecular weight polystyrene was measured. At early times the interface moved into the thicker high molecular weight film due to the styrene diffusion, and at later stages the interface broadened.

Surfactant adsorption (complex mixtures at interfaces)

The combination of neutron reflectivity and H / D isotopic substitution has been shown to be a powerful tool for the study of surface-active molecules at interfaces. Through deuterium labelling it is possible to highlight or isolate the contribution from particular components or fragments at the interface. This enables adsorbed amounts to be determined over a wide range of solution concentrations, measurements not possible by other techniques. This is especially important in the study of the interfacial behaviour of complex mixtures.

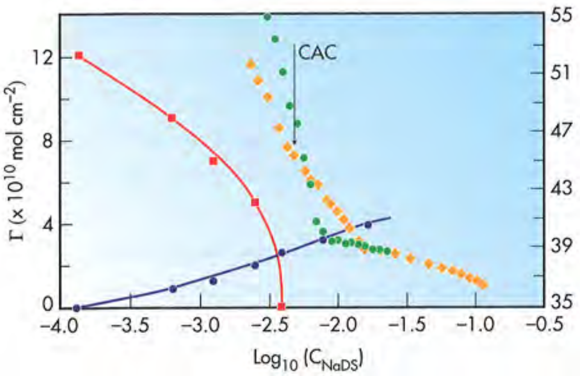


Figure 2. Adsorption isotherm (from neutron reflectivity) and surface tension behaviour for poly(ethylene oxide)/sodium dodecyl sulphate (PEO/SDS) at 35 ºC. Red/blue: SDS/PEO adsorption. Green/orange: SDS/PEO surface tension.

The widespread technological, industrial, and domestic applications of surfactants invariably use mixtures, and neutron reflectivity is now providing a unique insight into the surface behaviour of such complex samples. In many formulations, such as shower gels and hair shampoos, polymers are also frequently incorporated, as viscosity modifiers, stabilisers or deposition aids. Such polymers often interact strongly with surfactants in aqueous solution, giving rise to a rich pattern of behaviour in properties such as surface tension. However, without additional information about chemical activities, only techniques such as neutron reflectivity can unravel what is happening at the interface (see figure 2), and often this is very different from the bulk behaviour.

More powerfully, detailed structural information on the surface layer can be obtained, and such studies have provided a new insight into the monolayer structure, revealing 'surface layering' at higher solution concentrations, and the onset of 'surface micellisation' in amphiphilic block co-polymers. More detailed labelling schemes enable the mean conformation of a surfactant molecule at the interface to be determined, and the contributions of capillary waves to the structure to be identified. For surfactant mixtures at the interface, detailed labelling schemes have provided a detailed description of the surface structure of such mixed monolayers (see figure 3).

Complex fluids under shear

The behaviour of complex fluids under shear flow (figure 4) is important in the context of product processing and delivery. Understanding the microscopic origins of the complex rheological behaviour that exists in these fluids requires techniques such as SANS.

Measurements on LOQ of the lamellar phase of the non-ionic surfactant $C_{16}E_6$ in a

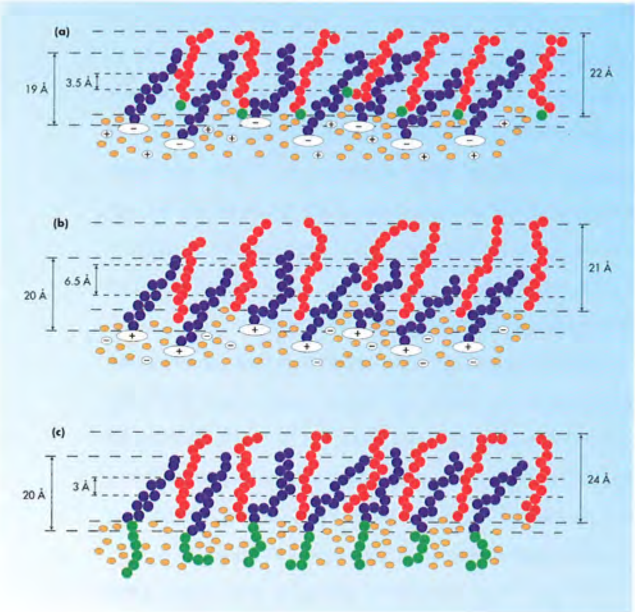


Figure 3. $C_{12}EO_5$ surfactant monolayer with adsorbed dodecane (top) compared with SDS/dodecane and $C_{12}TAB$/dodecane (bottom).

well defined Couette flow show a complex behaviour. At low shear rates the lamellae are aligned parallel to the flow-vorticity plane, whereas at higher rates they are aligned in the shear gradient-flow plane. At intermediate rates a complex interplay between the two extremes of behaviour exists.

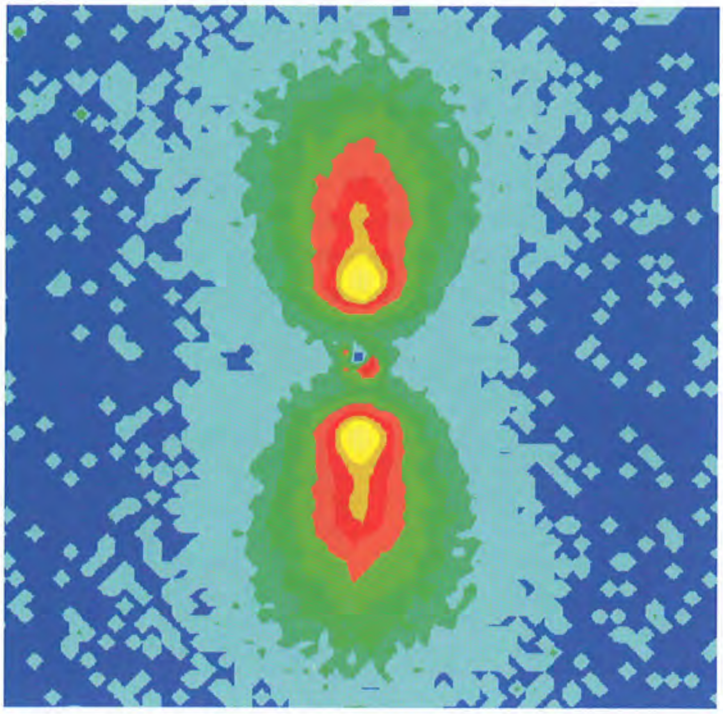


Figure 4. LOQ data from mixed surfactants under shear: 3% $C_{16}E_6 + C_{16}TAB$.

For the di-chain cationic surfactant, 2HT, under shear flow, a different type of behaviour is seen. At very low shear a highly aligned lamellar phase is observed, ordered in the flow-vorticity plane. At a critical shear rate, which is dependent upon concentration and increases with increasing concentration, the scattering becomes isotropic, consistent with the formation of multi-lamellar vesicles. In the ordered lamellar region, measurements at different scattering geometries with respect to the flow and gradient axes show an inconsistency which is reconciled by a distribution of orientational order across the Couette cell gap.

Aggregation in super-critical fluids (complex environments)

Liquid or super-critical carbon dioxide is a non-toxic, chemically inert, non-flammable fluid. As such it provides a safe alternative to potentially hazardous petrochemical solvents, and is being exploited commercially in processes involving flavour extraction (e.g. hops), decaffeination of coffee beans and dry cleaning. The high compressibility of CO_2 near the critical point enables a wide density variation which can be exploited to fine-tune solvent properties and enhance selectivity. A limiting factor in the solvent properties of CO_2 is the low solubility of 'polar molecules', which normally dissolve well in water. This problem has been approached by the addition of polar co-solvents, such as ethanol, to enhance solubilities, or by exploiting the nature of the C-F bond compared with C-H.

There is now growing interest in the formation of water-in-CO_2 micro-emulsions which are stable dispersions of nanometre-sized water droplets coated by a monolayer of surfactant (figure 5). Since the water droplets can solubilise polar molecules, micro-emulsions can provide a 'universal

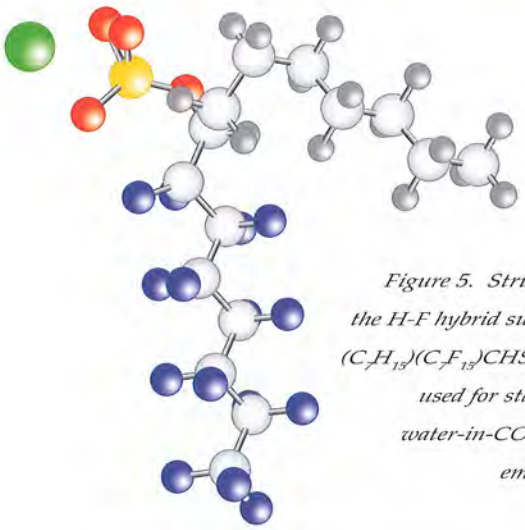


Figure 5. Structure of the H-F hybrid surfactant $(C_7H_{15})(C_7F_{15})CHSO_4^-Na^+$, used for stabilising water-in-CO_2 micro-emulsions.

solvent' for both polar and apolar molecules. SANS on LOQ has been used to investigate the structural features of these new systems to establish whether discrete micro-emulsion droplets are formed as opposed to simply a homogeneous solution of water in CO_2. The penetrating power of neutrons makes the design of a pressure cell for such measurements straightforward, and SANS is a powerful technique for characterising aggregates such as micelles and micro-emulsions. Measurements have shown that the hybrid H-F di-chain surfactant does result in micro-emulsion formation in super-critical CO_2 above a certain critical pressure. The SANS measurements have provided structural information that has been vital to the design of new surfactants for the production of water-in-CO_2 micro-emulsions at optimal operating temperatures and pressures.

Bio-molecular Sciences

There is a modest but important user base in Bio-molecular Sciences at ISIS. The first example below is the determination of the structure of the antibody Immunoglobulin A in solution. This demonstrates how SANS is used to obtain low resolution (mesoscale) structural information of important macromolecular assemblies. It also illustrates the frequently-used complementarity of neutrons and X-rays, leading to a model which provides an improved understanding of the functional properties of the antibody. The second example demonstrates the potential of an emerging area, the use of specular neutron reflection for the study of surfaces and interfaces of biological relevance.

Non-crystalline diffraction methods provide access to local solvent-solute, solvent-solvent correlations in such systems; for example, the effect of protein stabilisers or denaturants on the solvation shells of particular chemical groups on proteins.

High resolution single crystal and powder diffraction measurements are presently limited to smaller biologically-relevant molecules. Neutron diffraction provides accurate hydrogen positions and thermal ellipsoids. In conjunction with high-resolution X-ray data, this gives access to accurate electrostatic interaction parameters essential to understand, for example, drug-virus interactions.

Structure of Immunoglobulin A

Antibody or immunoglobulin molecules play a critical role in our body's immune defence against bacteria and viruses. Immunoglobulin A (IgA) is not only present in blood but is also the predominant antibody class found in mucosal surfaces of the lung and the gastrointestinal tracts, and so forms a critical first line of defence against

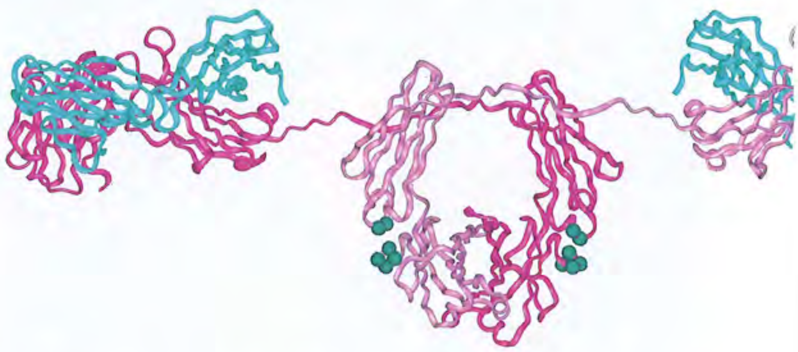


Figure 6. Ribbon diagram of the 3-D structure of immunoglobulin A1.

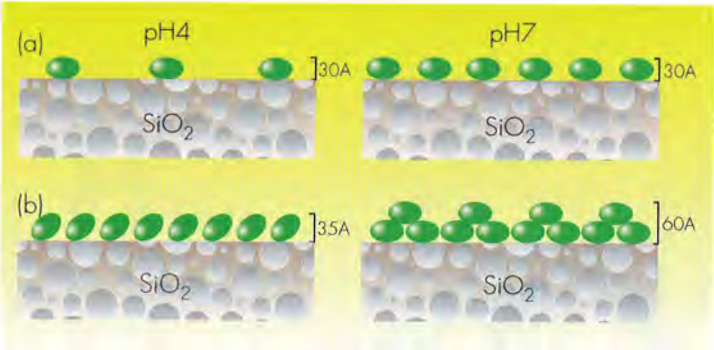


Figure 7. Schematic diagram of the surface coverage and orientation of lysozyme molecules adsorbed at the silica-water interface at different concentrations (a,b).

many invading pathogens. However, despite the abundance and importance of IgA, surprisingly little is known about its three-dimensional structure and how this relates to its role in immunity. A combination of small angle neutron and x-ray diffraction has been used to reveal the intermolecular arrangement, and in turn provide a better understanding of its functional properties.

Like other immunoglobulins, IgA has a basic monomeric structure composed of two heavy chains and two light chains, arranged into two identical side lobes and a central region, separated by a flexible hinge. The tips of the side lobes are responsible for recognising and binding to structures (antigens) on the surface of foreign cells or molecules. This region interacts with specific receptors on the surface of phagocytic immune cells to trigger mechanisms such as engulfment and cell killing to eradicate invaders.

Structure at medium resolution was determined using small angle diffraction, and from a refinement of complementary X-ray data and neutron data from samples in D_2O and H_2O. This medium resolution structure, to a resolution of about 10 Å, was developed by a novel, highly-constrained technique, in which molecular-dynamics simulations of IgA1 (one of the sub classes of IgA) hinge peptides were used to create 12,000 randomised IgA1 molecules. Each one was built from homology models for the central and side regions connected by the hinge peptides. After filtering to reject poorly-fitting models, the first averaged solution structure for serum IgA1 was obtained as a superposition of 104 best-fit models. A T-shaped structure was revealed in which the hinge of each heavy chain holds two side lobes apart with a very high side lobe centre-to-centre separation of 170 Å (figure 6).

The model leads to an improved appreciation of the functional properties of IgA1. The two side lobe antigen sites are widely spaced apart at 230 Å, and this enables them to access more widely spaced antibody targets on foreign particles compared with other antibodies, thus permitting a greater diversity of immune targeting in the blood.

Protein adsorption at interfaces

Protein adsorption is involved in a wide range of processes and applications. The deposition of blood proteins on to medical devices and implants, and the subsequent modification of their biological response, the bacterial fouling of ships hulls and the blockage of filtration membranes in bio-separation processes are examples of the unfavourable aspects of protein adsorption; the extensive use of proteins in food stabilisation and in the fabrication of bio-sensors are widespread applications.

Pioneering experiments on two common and well-characterised globular proteins, lysozyme and bovine serum albumin (BSA), have shown how neutron reflectivity can contribute to our understanding of protein adsorption at interfaces, and have opened the way to study more complex processes such as membrane-protein interactions (figure 7).

Adsorption at hydrophilic surfaces involves both the electrostatic interaction between the surface and the hydrophilic parts of the protein, and between protein molecules at the interface. Further driving forces are the entropic changes associated with dehydration of the protein or with its structural rearrangement. Strong surface interactions may damage the native state of the protein, and lead to deformation or denaturation, the extent of which will depend upon the nature of the surface and the relative stability of the protein. Neutron reflectivity offers the opportunity of measuring not only adsorbed amounts, but the surface structure of adsorbed proteins to reveal the nature of those surface interactions.

Specular neutron reflectivity, combined with appropriate water contrast variation, was used to obtain a direct determination of the structural distribution of lysozyme and BSA at the air-water and liquid-solid interfaces. For lysozyme at the air-water interface the protein remains intact. At low concentrations the protein has its long axis parallel to the surface and is about 50% immersed in the solvent. At higher concentrations the long axis is vertical, and the protein is about 75% immersed in the solvent, consistent with some of the amino acid side chains being shifted out of the water. At the hydrophilic silica surface the adsorption of lysozyme was completely reproducible with respect to concentration, but irreversible. The structure normal to the surface was determined by varying the isotopic content of the water. The adsorption was completely reversible with pH, and the variation with pH implies that the surface excess is dominated by interactions between protein molecules rather than with the surface. Changes with pH and concentration indicate that the protein undergoes a transition from 'side on' to an 'end on' configuration, and that at higher concentrations layering occurs. The measurements also indicate that the lysozyme retains its tertiary structure at the hydrophilic interface. This is in marked contrast to adsorption at the hydrophobic interface, where denaturation does occur. Similar observations have been made for the protein BSA.

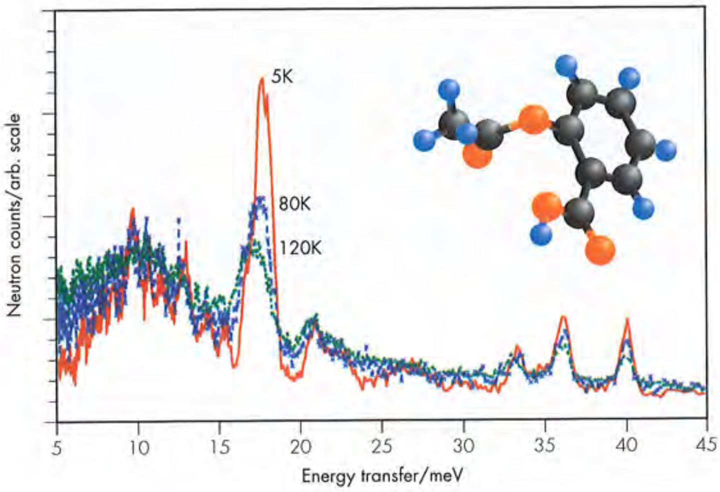


Figure 8. Inelastic scattering spectrum of aspirin. The strong feature is due to the fundamental transition of the methyl torsional motion.

Dynamics of Molecules

The energies of thermal neutrons are similar to those of atomic motions. This enables molecular vibrations, lattice modes and diffusional motion to be probed (figure 8). The momentum transfer and energy transfer space (Q, ω) accessible by neutrons translates to a real time space of 0.1 – 1000 Å and 10^{-8} - 10^{-15} s. Vibrational spectroscopy is a frequently used method, and the neutron technique is complementary to the optical (infra-red and Raman) methods, often leading to a better understanding due to the absence of 'selection rules' and the simplicity of the interaction. Measurements over a broad (Q,ω) range also give spatial information on the vibrational wavefunction. This is a particularly powerful method for hydrogen-containing systems, and, as illustrated in the example below, provides the sensitivity to study catalytic surfaces. The broad (Q,ω) range also provides access to a variety of other processes such as quantum excitations, the rotational tunneling of small molecules, diffusional motion of small molecules through solids such as zeolites and crystal field effects.

Catalyst poisoning by methyl groups

Catalysts are an integral feature of everyday life, from catalytic converters on vehicles to the refining of crude oil to petrol. Most commodity chemicals can only be produced economically by the use of catalysts. Vibrational spectroscopy is extensively used to characterise species on catalyst surfaces, and inelastic neutron scattering (INS) performed on TFXA (now replaced by the TOSCA instrument) has helped to reveal why an industrial catalyst became deactivated in-process.

A palladium catalyst that had been used for a process involving the hydrogenation of C-O groups to C-OH groups of functionalised aromatic and polyaromatic systems, but which had become deactivated, did not show evidence of coke formation (the most common cause of deactivation) although some carbon was present. Palladium is the catalyst of choice because it readily dissociates hydrogen; however, measurements of the surface area and hydrogen capacity were normal. Investigation by a range of analytical techniques did not show any clue as to the cause of the loss of activity. Raman spectroscopy, using either visible or near-infrared excitation, and infrared spectroscopy were both unable to resolve the problem.

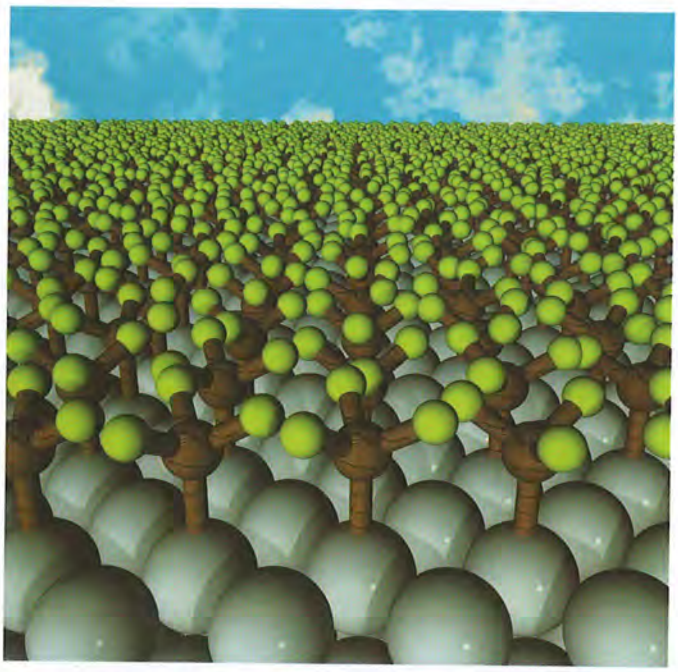


Figure 9. Idealised view of the palladium catalyst surface covered by methyl groups.

The INS spectra, from TFXA, of a deactivated catalyst showed a series of sharp peaks which were completely absent on an active catalyst. The most striking feature of the spectrum was its simplicity - there were remarkably few bands. This implied either a highly symmetric structure with many degenerate modes, or a small molecule. The intense 302 cm^{-1} feature suggested a methyl torsion, and a detailed analysis showed that the spectrum was consistent with a layer of methyl groups in an on-top site configuration on the surface.

The sharpness of the peaks suggest that the methyl groups are bound to the top-most atomic layer of the catalyst, and the intensities suggest that most of the non-extractable carbon was present as methyl groups. The relative intensities, when normalised to the amount of sample in the beam, showed an excellent correlation with the degree of deactivation of the given catalyst sample. The large quantity of bound methyl groups presumably lowered the catalyst performance by a surface blocking effect (figure 9).

Structure of crystalline materials

Understanding the exploitable properties of new materials requires a detailed knowledge of their structure. Neutron scattering has played a principal role in developing this understanding in almost every area of modern structural Chemistry and Physics.

High resolution allows phase transitions, subtle structural effects and complex structures to be studied. High flux at medium resolution enables rapid data collection in complex environments and for in-situ processes. The time-of-flight Laue technique provides access to structural and disorder information in single crystals. The following examples cover this broad range of diffraction applications.

Solid State Chemistry

The broad range of d-spacings accessible with good resolution using the white beam time of flight method has enabled powder diffraction at ISIS to make a real impact in the area of structure-property relationships for a range of technologically important materials.

The ability of neutron diffraction to probe the location of light atoms in the presence of heavier ones is particularly important in the study of lithium batteries, where the synthesis of new cathode materials is an important aspect of the development of high power density, light-weight cells for applications such as laptop computers and mobile phones. A detailed knowledge of the crystal structure is required to understand the dynamics of the Li^+ intercalation mechanism, including the effects of repeated charging/discharging, to determine those factors which limit the ultimate lifetime of the battery (figure 10).

The recent ab-initio structure determination of the polymer electrolyte poly-(ethylene oxide)$_6$: $LiAsF_6$ is a notable example of the complex structures that can be determined from powder diffraction on a pulsed neutron source. Polymer electrolytes consist of salts dissolved in solid, high molecular weight polymers, and represent a unique class of solid co-ordination compounds. Their importance lies in their potential for the development of truly all-solid-state rechargeable batteries. The structure of the 6:1 complex is particularly important, as it is a region where the conductivity increases markedly and is distinct from all known crystal structures of PEO : salt complexes. The Li^+ cations are arranged in rows, with each row located inside a cylindrical surface formed by two PEO chains, with the PEO chains adopting a previously unobserved conformation. Furthermore the anions are located outside the PEO cylinders and are not co-ordinated with the cations.

Neutrons uniquely are able to probe the nature of the magnetic ordering within crystalline solids. In recent years this has been illustrated by the considerable interest in the so-called Giant Magneto-Resistance (GMR) materials, which exhibit significant changes in their electrical resistance under applied magnetic fields and thus have potential applications as, for example, magnetic storage media.

The absence of a form-factor for neutron scattering allows diffraction data to be collected to significantly lower d-spacings than its X-ray counterpart. This, coupled

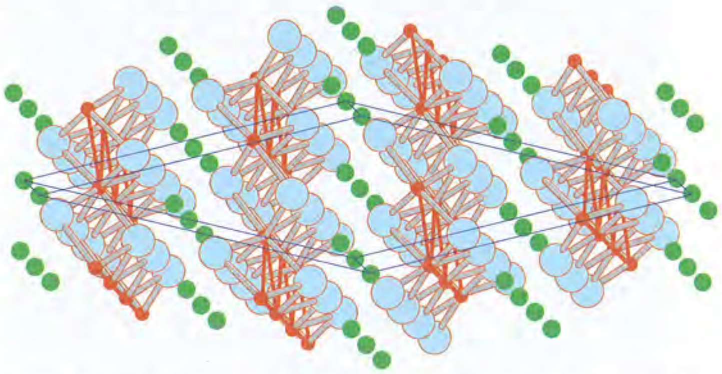


Figure 10. The structure of $LiMoO_2$, a potential cathode material for advanced Li batteries.

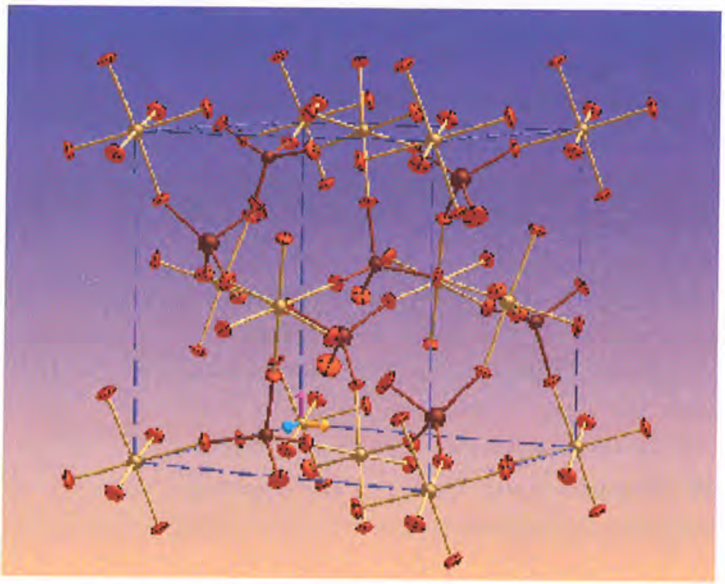


Figure 11. Structure of the negative thermal expansion material ZrW_2O_8

with the high flux of epithermal neutrons produced by the ISIS source, allows the chemical occupancies of partially filled sites within the crystal structure to be accurately determined. Such crystallographic information is important for the design of optimised materials using selective chemical doping, such as increasing the superconducting temperature in high T_c materials or maximising the mechanical strength of engineering ceramics.

The HRPD instrument at ISIS collects diffraction data with an extremely high resolution of $\Delta d/d \sim 4 \times 10^{-4}$ which is essentially independent of d-spacing. This unique facility has found many applications, including the study of subtle phase transitions and small structural distortions. Such a high intrinsic resolution allows the variation of crystal structure parameters to be determined accurately as a function of temperature, pressure and chemical composition. In a recent study neutron powder diffraction was able to provide a unique insight into the mechanisms of the negative thermal expansion material zirconium tungstate, which contracts on heating over its entire temperature range as a solid (0-1050K) (figure 11).

Structure of solids under very high pressures

The study of materials under extreme pressures has long been an important topic in condensed matter research (figure 12). Pressure and temperature are complementary thermodynamic variables. Temperature allows the effects of entropy to be studied, whereas pressure monitors changes associated with enthalpy. The application of hydrostatic pressure is a very effective means of changing the distances between atoms in a solid. The general 'rule-of-thumb' states that cooling to absolute zero brings about a reduction in sample volume comparable to the application of a pressure of ~1 GPa, which is straightforward to achieve experimentally. As a consequence, experimental data describing the evolution of the crystal structures of the elements and simple (i.e. binary) compounds under compression is routinely used to test the reliability of potentials derived ab-initio.

The most effective means of subjecting samples to extreme pressures (greater than, say, 5 GPa) is to use relatively small samples (typically less than 1 mm^3). Neutron diffraction studies of samples under these conditions are desirable to locate light atoms

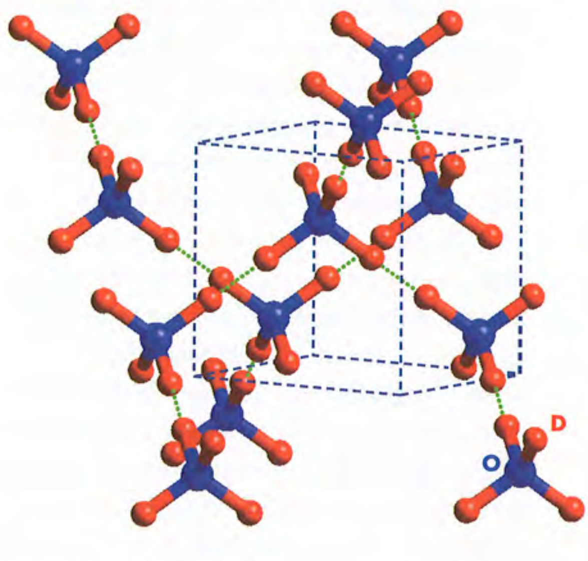


Figure 12. The structure of cubic ice VII, formed at pressures above 2GPa.

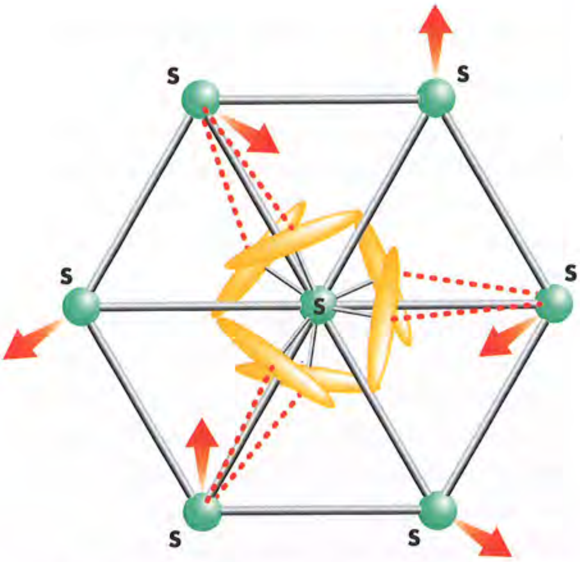


Figure 13. Part of the D_2S high pressure phase I′ structure showing a central sulphur atom, the six associated deuterium sites (ellipsoids) and the six nearest neighbour sulphur atoms.

in the presence of heavier ones and to study magnetic ordering. However, the requirement to use small samples suggests intuitively that neutron diffraction will be at a distinct disadvantage compared to its X-ray counterpart for such studies. The flux of X-ray photons produced by a third generation synchrotron source is many orders of magnitude higher than the flux of neutrons at ISIS, even though it is the world's most intense pulsed neutron source. However, over the last decade a collaborative project at ISIS involving the Universities of Edinburgh and Paris has shown that this is not the case. In particular, novel design of the pressure cell allows sample volumes of ~100 mm³ to be routinely pressurised to greater than 10 GPa. Moreover, optimised design of both the cell and the diffractometer to exploit the inherent characteristics of the ISIS pulsed source allows high-quality diffraction data to be collected in relatively modest counting times (typically a few hours). Specifically, the diffraction experiments at a pulsed source such as ISIS are performed at fixed scattering geometry. The pressure cell then only requires narrow apertures for the incident and scattered beams, because the detector is not scanned as it would be if the experiment were performed at a monochromatic (reactor-based) diffractometer. Using scattering angles close to 90° and suitable collimation of the incident and scattered beam apertures, it is possible to define an effective scattering volume which lies entirely within the sample volume. This enables the collection of a diffraction pattern from the sample (under its extremely non-ambient conditions) without contamination by spurious peaks from the surrounding pressure cell components.

Earth Sciences

In the Earth Sciences, the application of neutron techniques benefits from the same advantages (see table on page 8) as in many of the areas of materials research. In spite of this, the Earth and Environmental Sciences have until recently remained relatively uncharted territory for neutrons, perhaps because earth materials are considerably more complex structurally, and frequently they are composed of a number of different phases and compositions. Also, the conditions under which they must be studied push the experimental techniques for attaining high temperatures and pressures to their limits.

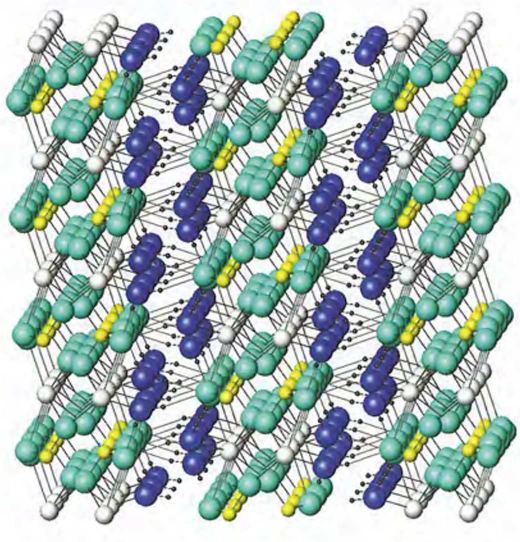


Figure 14. The structure of gypsum.

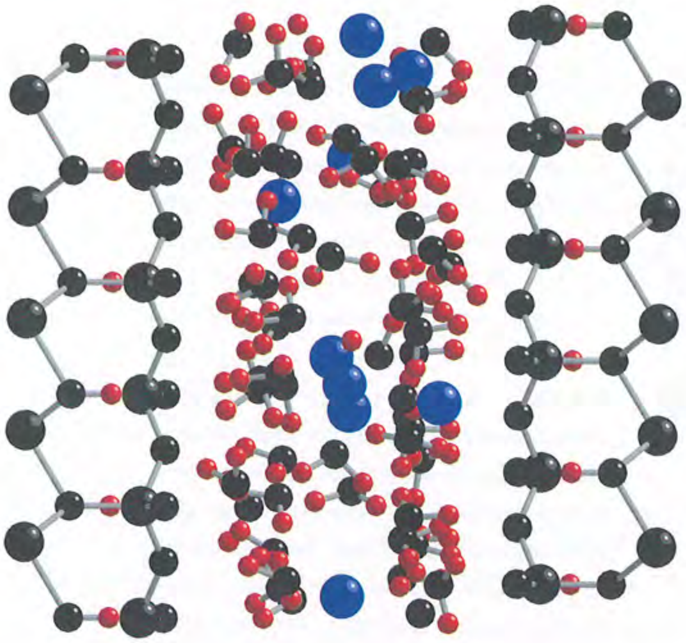


Figure 15. Molecular graphics snap-shot of water within a swelling clay.

However, there has recently been a dramatic expansion of the use of ISIS in this area, and the available instrumentation and analysis techniques have enabled these challenging problems to be tackled.

A notable recent example is the work on cation-ordering in olivines (figure 16), which illustrates how neutron single crystal and powder diffraction can be used to elucidate features of mineral structures and their geophysical relevance. Olivine is a major constituent of the Earth's mantle and it has long been appreciated that the ordering of the cations onto distinct sites in olivine structures is strongly dependent on temperature - this provides clues as to the conditions under which olivine-containing rock was formed. However, attempts to quantify this have long foundered on the requirement for studying quenched samples using, for example, X-ray diffraction techniques. Quenching methods are based on the assumption that rapidly changing a thermodynamic variable (temperature, pressure) can lead to the retention of the phase of interest but under conditions which are more amenable to study. There are many cases where this method can be applied, but in the case of olivine the re-ordering processes are now accepted to be faster than the accessible quench rates and such studies can be highly misleading - the cation ordering does indeed alter on quenching and the samples studied do not accurately represent the pre-quench conditions. With the possibilities offered by neutron diffraction of performing routine, in-situ studies, the veil has now been lifted on some of the mysteries of this important system. A further advantage of neutron diffraction is the high contrast available between cations close in atomic number, for example Fe and Mn, which are all but indistinguishable using X-rays.

ISIS has been used in both single crystal studies of natural olivines of meteoric origin, and in neutron powder diffraction studies of synthetic olivine samples. In the first case, Italian and ISIS scientists studied a Mg-Fe sample of an olivine from Southern Ireland and discovered that not only was there a gradual preferential ordering of the cations up to ~900 °C, but beyond this temperature the preference was reversed, a hitherto unsuspected and unobserved feature which has challenged thermodynamic models of this system. The powder diffraction measurements confirmed these observations in synthetic Fe-Mn samples. The improved quantification of the precise temperature dependence of the effect in the latter case led to the concept of 'Geospeedometry' by which the cation ordering found in olivine

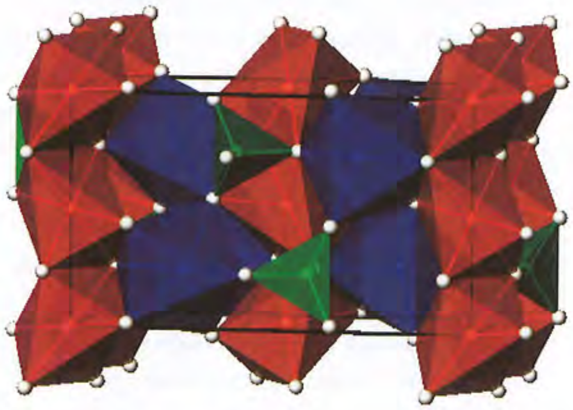


Figure 16. Olivine structure.

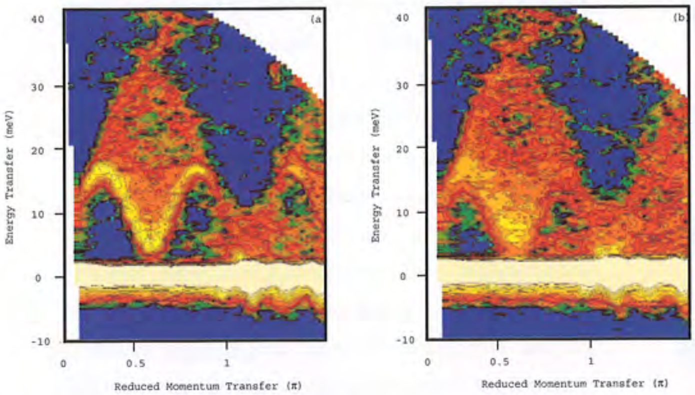


Figure 17. The excitation spectrum of the spin-Peierls system $CuGeO_3$ at 10K and 20K.

samples can be used to gauge the rate of cooling upon formation of the rocks in which they are found.

Hard Condensed Matter

The impact of neutron scattering in Condensed Matter Physics, which includes magnetism, superconductivity, quantum systems, lattice dynamics and phase transitions, has been of outstanding importance. Neutron scattering often provides the basic atomic and magnetic structural and dynamical information for new materials, such as heavy fermion systems and high-temperature superconductors, and is often concerned with the solution of basic questions in quantum and statistical mechanics in the solid state. One of the highlights of the ISIS scientific programme over the past fifteen years has been the correct determination of the structure of the high temperature superconductor $YBa_2Cu_3O_{6+x}$. Neutron scattering is the only method for the determination of magnetic structures.

Inelastic neutron scattering experiments also provide rich information on fundamental magnetic interactions that cannot be obtained by other probes and which can be compared to detailed theoretical models. The ISIS spectrometers have made a major impact in this field, in areas like CMR and high temperature superconductivity. The ability to collect large data sets allows the simultaneous measurement of entire dispersion relations, for example the data collected from the spin-Peierls compound $CuGeO_3$ (figure 17). Chopper spectrometers allow access to energy transfer ranges that cannot be reached at other neutron sources; consequently the ISIS instruments have been used to study materials such as one-dimensional chains and spin ladders that cannot be investigated anywhere else in the world. Two examples - the impact of inelastic scattering in the understanding of High-T_c superconductors and CMR materials - are described here in more detail.

High-T_c Superconductors

The parent compounds of high-T_c superconductors, such as La_2CuO_4 (figure 18) or $YBa_2Cu_3O_6$, are insulating

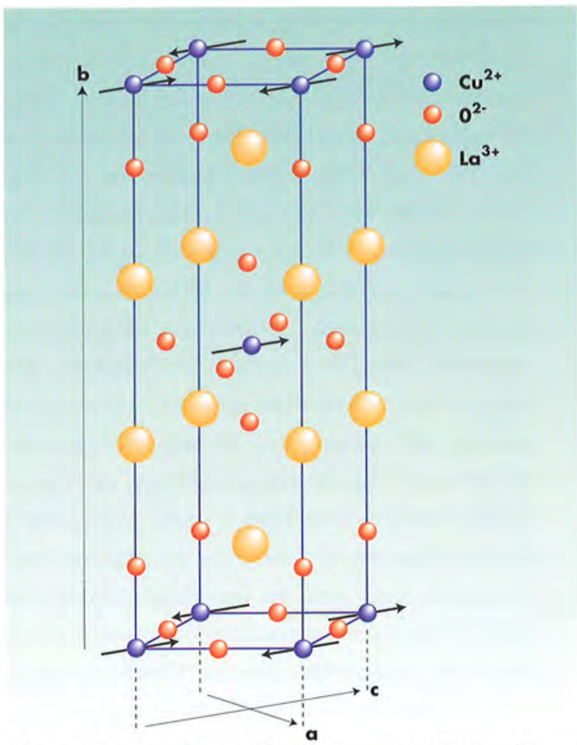


Figure 18. Structure of the antiferromagnetic insulator La_2CuO_4.

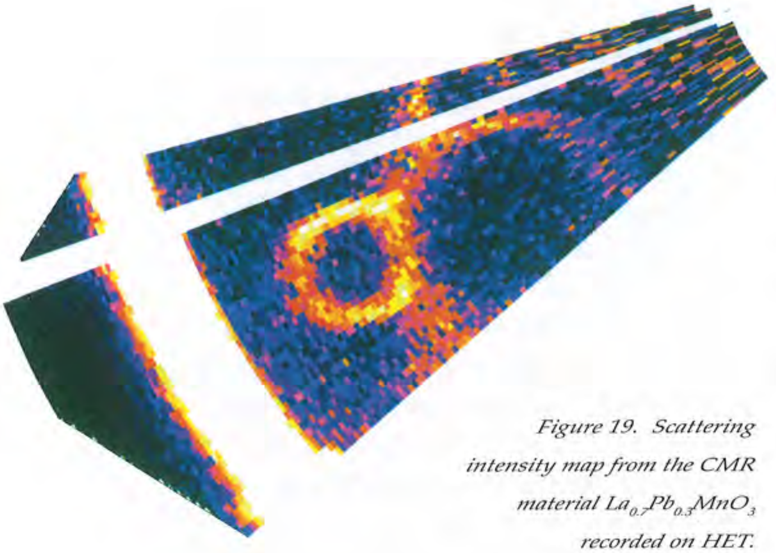


Figure 19. Scattering intensity map from the CMR material $La_{0.7}Pb_{0.3}MnO_3$ recorded on HET.

antiferromagnets, which, when chemically doped with Sr or O to remove electrons from the Cu-O planes in the crystal structure, become metallic and superconducting. While the superconducting compositions no longer show long-range magnetic ordering, strong antiferromagnetic correlations between the fluctuating magnetic moments remain. It is widely believed that these magnetic fluctuations are responsible for the bonding between pairs of electrons responsible for superconductivity. However, the nature, energy spectrum and precise role of the fluctuations in the superconductivity still remain incompletely resolved. Until recently, the spectrum of magnetic fluctuations in the two most widely studied examples, $La_{2-x}Sr_xCuO_4$ and YBa_2CuO_{6+x}, appeared to be very different at the energy scale of the bonding between the two electrons in the superconducting pairs. Use of the recently installed position sensitive detectors on the HET spectrometer at ISIS allowed images of the fluctuations to be recorded in the Cu-O planes of YBa_2CuO_{6+x}. The data show a pattern of 4 peaks with the same intensity, orientation and peak separation as in $La_{2-x}Sr_xCuO_4$, revealing a unifying feature in the magnetic fluctuations and suggesting a common explanation for the origin of high T_c superconductivity.

Colossal magnetoresistance

In recent years there has been a huge revival of interest in manganese oxides with the perovskite structure $RE_{1-x}A_xMnO_3$, where RE is a rare earth, for example La, Pr or Nd, and A is a Group-II metal such as Ca, Ba, Sr, Pb. This is because of the massive change in electrical resistance they can display when placed in a magnetic field – over two orders of magnitude in a few Tesla. The interest is not just because of the fundamental physics of metal-to-insulator transitions, but because of the potential technological applications. As with the high temperature superconductors, the undoped parent materials (i.e. x=0) are antiferromagnetic insulators, but with sufficient doping, x=0.2-0.4, the materials are ferromagnetic metals which become insulating above the Curie temperature. The colossal magnetoresistance (CMR) is displayed at temperatures near T_C, typically 200-400K. Knowledge of the magnetic fluctuations is vital to test models of the interactions between the current-carrying electrons and the magnetic moments of the Mn ions, which lie at the heart of the CMR phenomenon. Several groups from around the world are using the spectrometers at ISIS to investigate these materials. One question is the extent to which a model for the interactions dating from the 1950's is a good starting point to understand the ferromagnetic state. Data taken on HET (figure 19) for $La_{0.7}Pb_{0.3}MnO_3$ show good agreement with the predictions of the model, with a realistic value for the metallic band-width.

Magnetic thin films

Polarised neutron reflection, PNR, is a sensitive probe for the study of both structural and magnetic properties of magnetic thin films and multi-layers. The neutron spin-dependent reflectivities provide an absolute and sensitive measurement of magnetic moments, distribution and orientation in thin films. It has now also been demonstrated that 'off-specular' scattering provides direct information about domain sizes and orientation.

The power of PNR as an absolute layer-sensitive vector magnetometer has been impressively demonstrated in the recent work on the Cu/FeNi/Cu/Co/Cu/FeNi/Cu cobalt / permalloy spin valve structure. At high values of an external field the moments are ferromagnetically aligned. The spin dependent reflectivity data at low fields (50 G), after reverse-saturation, show that the Co layer rotates by 35° from the anti-aligned case. Further 90° rotations of the sample do not reveal the expected four fold symmetry, suggesting that a small but significant uni-axial anisotropy exists. This shows how sensitive measurements are essential for understanding the behaviour in such devices and their extensive applications as sensors, random access memory and disk read-write heads.

Disordered Materials

Disordered materials, which include liquids, glasses and disordered crystalline materials are common in our environment. The control of many modern materials' properties is through disorder and defects. Neutron scattering provides a unique probe of local order and dynamics in such substances, and increasingly it is being applied to more complex samples related to real applications.

The exploitation of isotopic substitution in such neutron diffraction measurements provides a powerful advantage over other scattering probes in determining local structure. The examples described here use the optimisation of the non-crystalline diffraction instruments at ISIS for H/D isotopic substitution measurements.

Structure of high and low density water

The local structure of water (figure 20) has attracted a revival of theoretical interest in recent years. These new approaches all predict an anomaly in the thermodynamics at 220K and 1 kbar, indicative of a structural phase transition.

From measurements on SANDALS using H/D isotopic substitution, the O-O partial structure factors were obtained at three different densities. The changes seen in these partial structure factors have all the hallmarks of a structural phase transition occurring within the liquid as the density of the liquid increases. The principal finding is that, whereas the inner near-neighbour shell of water molecules is almost unchanged with the application of pressure, the second and subsequent shells collapse inwards by about 1 Å for the higher density form of water compared to the low density form. It is therefore clear that whatever may happen at lower temperatures near the postulated anomaly, where the liquid is only metastable or may not exist at all, some form of structural

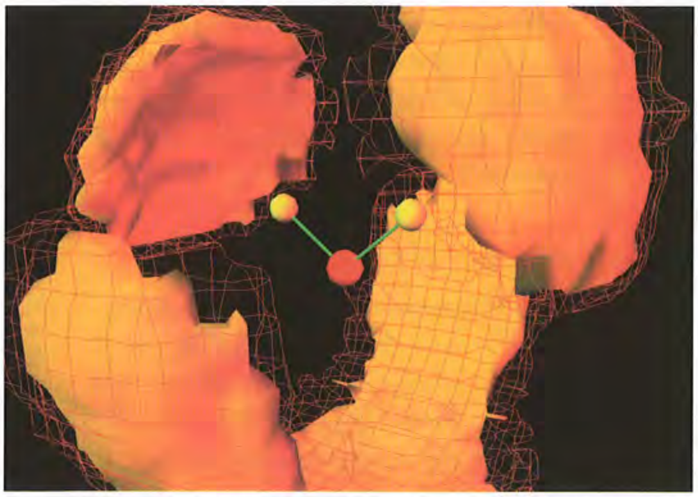


Figure 20. Distribution of molecules around a central molecule in water.

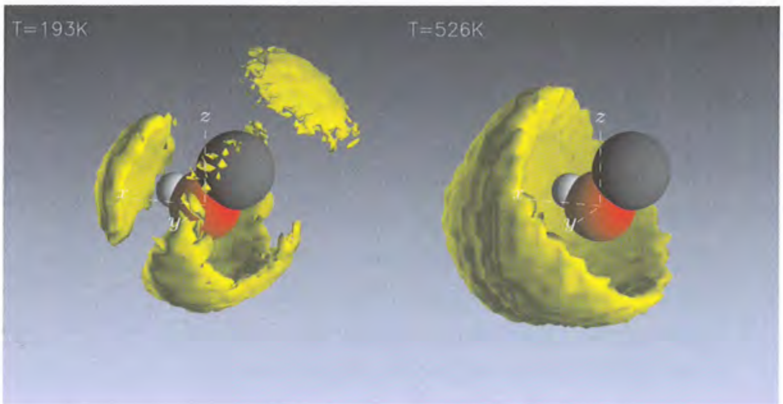


Figure 21. Spatial desnsity of methanol molecules around a central molecule.

transition does occur even in stable water with increased pressure. Other recent diffraction evidence indicates that the equilibrium between the two structures is easily affected by adding ions and other solutes to the water. It is very likely that the structural balance between the two forms of water plays a pivotal role in determining the degree to which solutes will phase separate, associate or dissolve when mixed with water.

Structure of water around ions and molecules in solution

Non-crystalline diffraction and H/D isotope substitution allows specific ionic or molecular sites to be identified, and the structure of water around those sites to be explored.

Around positive ions such as sodium, water molecules tend to line up with their dipole moment vectors pointing away from the central ion. There is, however, a distinct distribution of orientations, and it is found that while the most probable orientation is along the ion-water molecule axis, significant fluctuations about this direction occur, with the water molecule rotating about an axis parallel to its own plane, making the average orientation closer to 20°. For negative ions such as chlorine on the other hand, the hydrogen bonding nature of water molecules prevails, with one of the OH bonds on the water molecule pointing directly towards the ion, albeit again with a significant distribution of orientations, primarily this time about an axis perpendicular to the plane of the water molecule.

For hydrophobic species such as methane, the observed orientations are exactly out of phase with those seen for ions. In this case, for orientations which derive from rotations about the axis parallel to the water molecule plane, the dipole moment tends to point more towards the solute, while for orientations derived from rotations about the perpendicular axis, the dipole moment vector tends to point away from the solute. Thus in either case no hydrogen bonds form with the central methane. Quantification of these orientations from neutron diffraction data presents a powerful constraint on possible models of the aqueous systems under consideration.

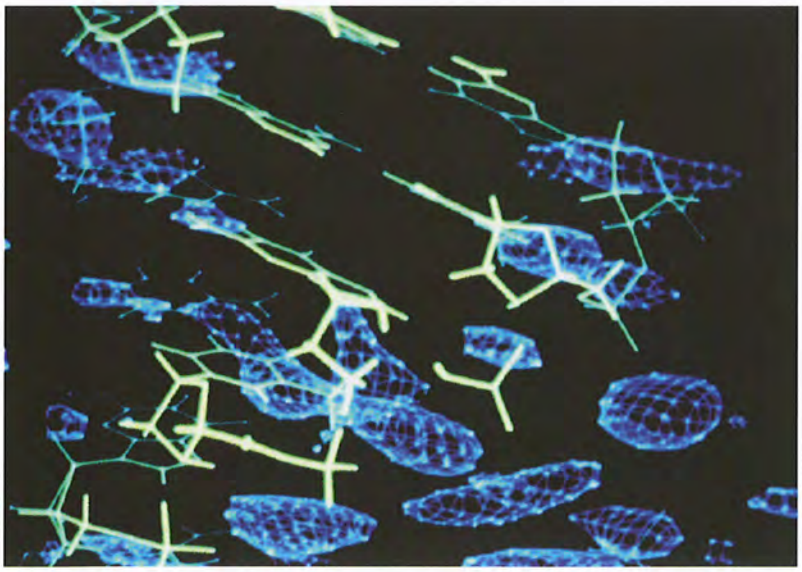
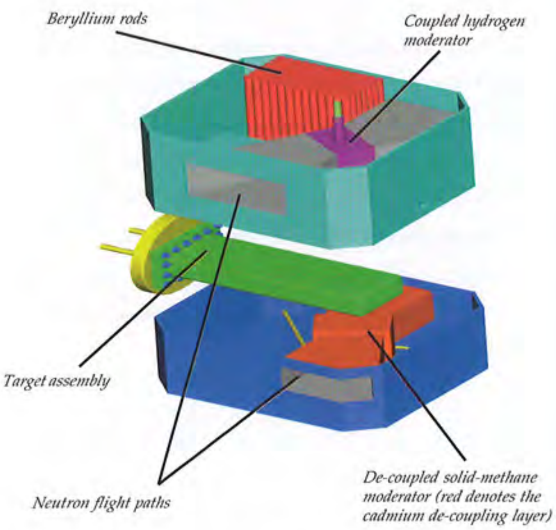


Figure 22. Structure of water around DNA.

New Opportunities for Inter-disciplinary Research on the Second Target Station.

Summary of Potential Gains in Performance

Substantial gains in performance of more than an order of magnitude over the High Power Target Station can be achieved for specific cold neutron and high resolution studies at low repetition rate, by optimising the design of both the target and moderators for these applications alone. The original High Power Target suffers from compromises made to service a broad instrument suite. The Second Target Station enables optimisation to be made for low power levels, producing significant gains in cold neutron flux. For example, at low powers highly efficient moderators such as solid methane can be used which are not viable on the High Power Target. The geometries of the moderators and target can also be optimised for cold neutron production. The removal of adsorbing 'decoupling' material also provides a significant enhancement in cold neutron flux. This latter effect also results in a broadening of the neutron pulse width which is not acceptable for some high resolution applications, and gives rise to a variation in the potential 'gain factor' possible on the Second Target Station, depending upon the particular type of instrumentation. The design of the Second Target Station will be optimised for applications which require a high cold neutron flux with a relaxed time structure and a broad wavelength band (SANS, reflectometry, neutron spin echo), and for high resolution applications requiring a broad wavelength band (using 'deoupled' cryogenic moderators for μeV spectroscopy and high resolution diffraction). All these techniques will experience significant gains.

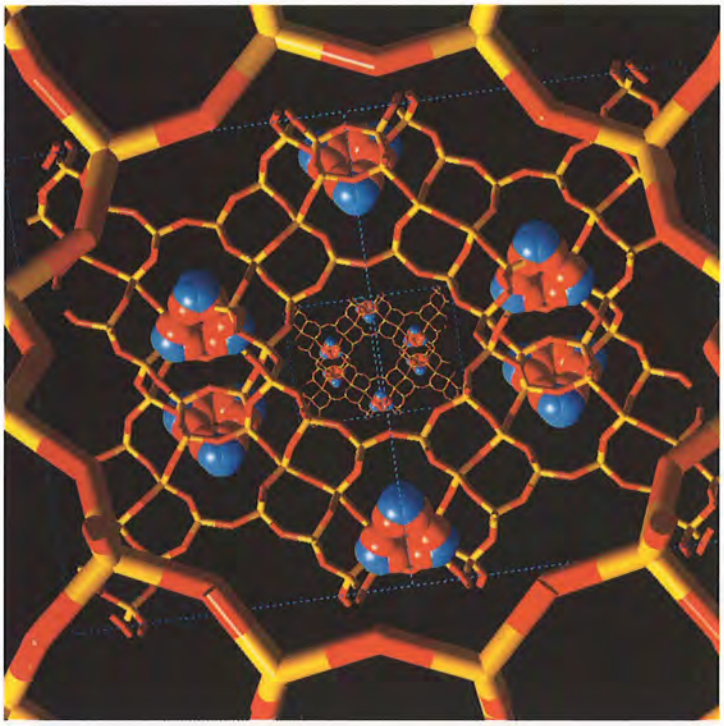


Target and moderator details.

Overview of Important Scientific areas

The main features of the Second Target Station, of enhanced cold neutron flux, broad spectral range and high resolution, are particularly well matched to many of the emerging areas in Soft Condensed Matter, Bio-molecular Sciences and Advanced Materials. The gains will provide exciting new opportunities in these technologically significant areas which relate directly to the major themes and priorities identified in the last UK Technology Foresight exercise as given in on page 7.

In the particular areas of Soft Matter and Advanced Materials there is a trend (already highlighted in the previous section on the recent achievements of ISIS) towards the study of complex multi-component or multi-

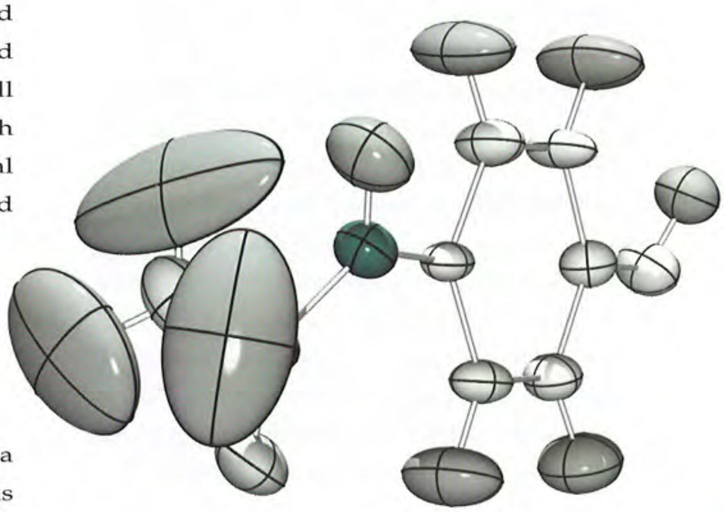


phase systems, the use of complex environments, and of non-equilibrium studies. In many of these systems the dimension scales of importance range from molecular to mesoscale, and this dictates the need for a broad wavelength band and a greater emphasis on cold neutrons. Kinetic studies (ranging from chemical reactions to probing dynamic surface tension) also require a broad spectral range and a higher flux of cold neutrons. Multi-component or multi-phase systems are only tractable with parametric studies possible using the enhanced flux and resolution. The gains and advantages afforded by an optimised Second Target Station (see page 28) will hence provide an exciting platform for such measurements, and some of those potential scientific developments are now described in more detail.

Soft Condensed Matter

Soft Condensed Matter encompasses a wide range of molecular materials, such as polymers, surfactants, colloidal suspensions, thermotropic liquid crystals, micelles, and lyotropic mesophases and micro-emulsions. The physical and chemical properties of these systems are of increasing technological importance. They have a wide range of applications related to macroscopic and surface / interfacial properties, including visco-elasticity and surface tension, which control practical phenomena such as rubber elasticity, detergency, adhesion and lubrication. Research interests are increasingly motivated by the commercial significance of products that are complex mixtures of components and structures, and this is currently a research area of considerable growth and potential.

All these materials share the same basic properties: weak interaction between structural units, large thermal fluctuations, large numbers of internal degrees of freedom, a macroscopic softness indicative of a weak modulus of elasticity and a sensitivity of their phase diagrams to external constraints. A detailed characterisation of such systems involves a wide range of distances and time-scales. SANS and neutron reflectometry are established as key techniques for the characterisation of soft matter at a molecular level, in terms of both the bulk and surface or interfacial behaviour. The ability to



manipulate refractive index (through H/D isotopic substitution) allows complex multi-component systems to be studied. The penetrating nature of the probe means that buried interfaces can be investigated and that complex environments can be easily accommodated. On pulsed neutron sources the white beam time-of-flight technique is used in both SANS and NR. This allows a wide range of scattering vectors (and hence length scales) to be probed simultaneously at high resolution, and this is the key feature for soft matter studies. The neutron scattering techniques provide information that not only complements other techniques but, importantly in many cases, is essential for the detailed understanding of such complex systems.

The broad scientific programme already established at ISIS shows trends towards the study of complex multi-component systems, the use of complex environments and the response to changes in environment such as temperature, pressure, external forces (e.g. shear flow), and there is an increasing realisation that the time-scales of some important time-dependent phenomena are now accessible. These trends are all closely linked to improving the understanding and ultimate control of ever more realistic but complex materials. The selectivity, through H/D isotopic substitution, the intrinsically good resolution and the broad range of length scales accessible in a single measurement, are features that will be fully realised by the incorporation of a Second Target Station optimised for cold neutrons. These features, coupled with the substantially enhanced flux, will have an enormous impact upon the emerging priorities in soft matter and complex fluids.

The Second Target Station at ISIS, with the expected substantial increase in flux and the even wider length scales and time ranges accessible, will have a profound affect on the scientific opportunities in soft matter:

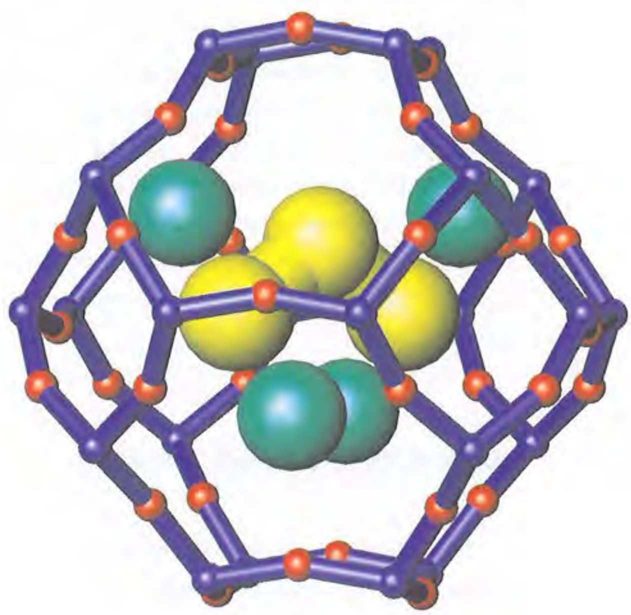
- Extensive parametric studies (the need to make measurements in a wide variety of conditions; such as isotopic labelling, temperature, pressure, external force field, pH, concentration, composition and so on in complex systems) will be possible with better resolution, in shorter times, on more dilute samples, and with more detailed isotopic labeling schemes.

- Rapid screening of the mesoscale structure of novel multi-component materials produced from combinatorial synthesis methods.

- More complex, multi-component systems (more closely resembling industrially-relevant materials), providing the opportunity to investigate the critical role of minor components.

- More complex environments (that are relevant to conditions faced in different processing routes, for example, in electrochemistry and extrusion), and more complex interfaces, will be accessible.

- New opportunities will be presented to utilise off-specular scattering, grazing incidence diffraction and SANS to probe



lateral inhomogeneities and self-assembly at interfaces, together with surface inelastic scattering to characterise the low frequency dynamics of the interfaces.

- Measurements of time-dependent processes will open up poorly explored fields of dynamics and kinetics of surfaces, thin films and anisotropic systems of polymeric and surfactant origin, including the microscopic details of complex rheological behaviour.

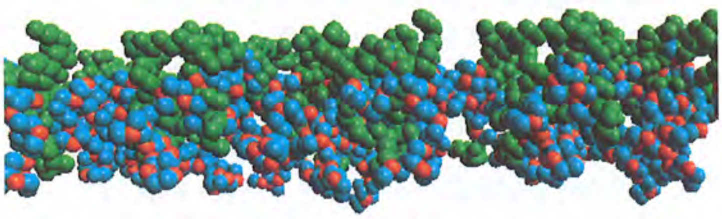
- Such measurements will extend our understanding of adsorption at the air-liquid, liquid-liquid, and liquid-solid interfaces. The ability to use smaller sample areas will dramatically extend the range of systems that can be studied. Liquid-liquid, and a wider range of solid-liquid interfaces will become routinely accessible.

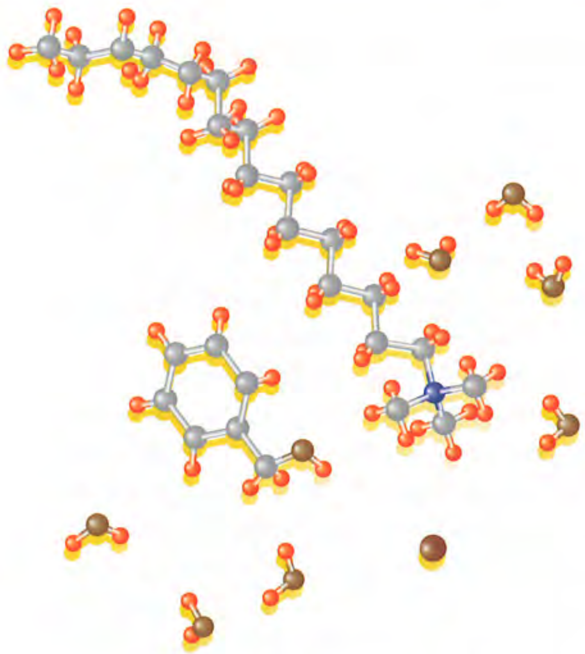
These potential areas of study will provide a much needed insight into a wide range of technologically important materials such as detergents, paints, inks, coatings, composite polymeric materials, emulsions, foams and gels found in foods and other products, and lubricants and fuel additives.

The following broad themes, identified as examples of areas of soft matter research that will benefit enormously from the proposed developments, are described in more detail below.

Surface adsorption and ordering

The interfacial properties of surfactants, polymers and complex mixtures is a vast and rapidly growing field, in which all aspects of disordered and semi-ordered interfaces have been opened up by specular and off-specular neutron reflection. The higher flux available on the second target station will enable the study of more complex multi-component systems through contrast variation methods, and will provide more detailed structural information through more complex isotopic labeling schemes. Time-dependent measurements will provide access to kinetic processes at the interface, associated with, for example, relaxation in compressed monolayers, dissolution, surface phase changes, and the migration / ingress of oligomers or solvent in polymers films. The more technologically relevant interfaces, liquid-solid and liquid-liquid, will become more accessible, and the surface behaviour of more complex molecules such as polyelectrolytes, bio-molecules and liquid crystalline polymers, will be routinely studied. Off-specular scattering will provide information on the lateral order in such systems on a length scale unobtainable by other techniques. The new information will impact upon our understanding and manipulation of detergency, lubrication, coatings, adhesion, and foam and emulsion stability; influence the design of sensors, display devices, functionalised surfaces, smart packaging, composite materials and the emerging area of polymeric opto-





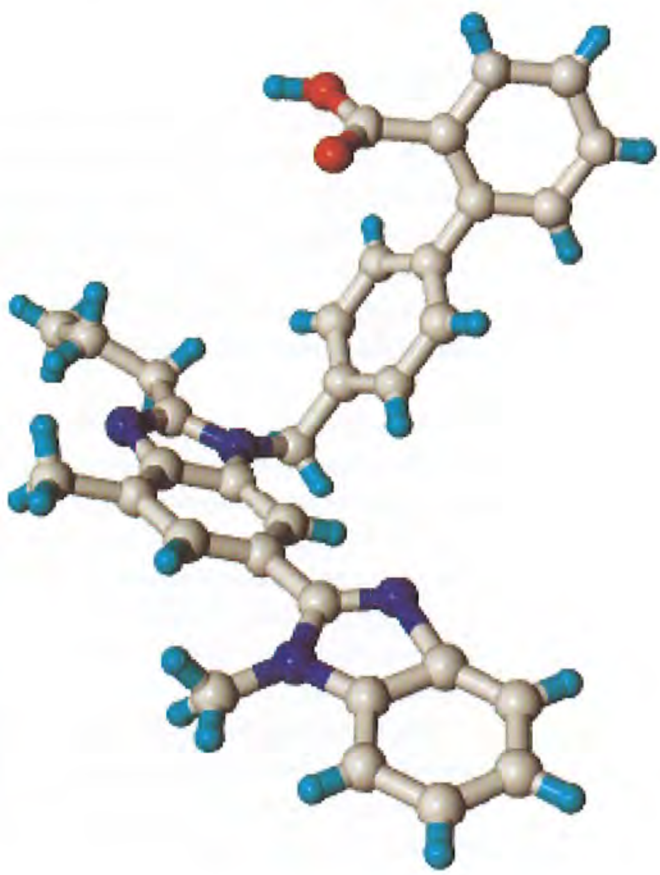
electronics and devices; and help in corrosion control (protective coatings).

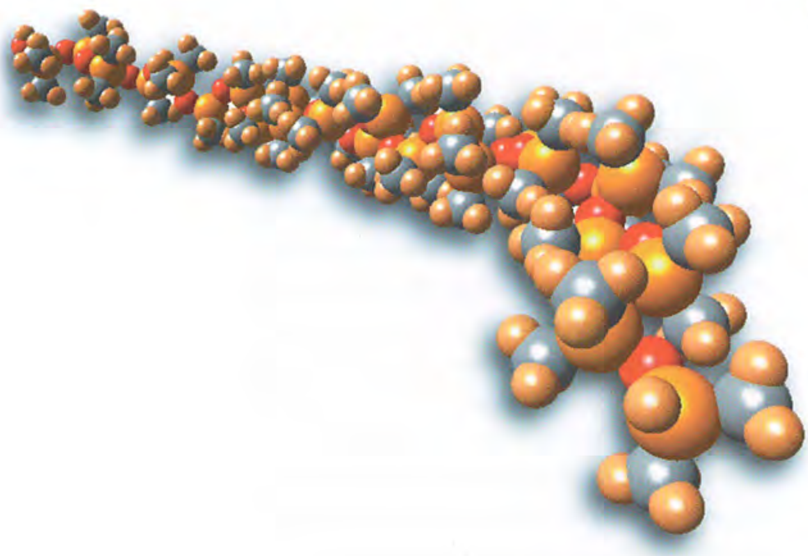
Flow and processing

The processing of a variety of soft solids in many important industrial processes involves flow, from component mixing in pipe flow, to extensional flow for the deliberate alignment by extrusion. The flux available on the Second Target Station will make it possible to study the mechanisms controlling these processes at a molecular level in realistic conditions and geometries, through the full range of fluid fields encountered. Measurements as a function of position to map the response to complex spatial distributions of velocity and stress within the extruder will be possible. With the partial labeling of parts of a polymer molecule, measurements of polymer melts under flow will develop the structure - rheology relationship in polymer processing and address the role of features such as branching, polydispersity and additives. Similar labeling schemes will be exploited in multi-component systems, involving surfactants, polymers and colloidal particles, to provide the essential link between phase behaviour, rheology, and product formulation and stability. It is not only structural information that is important, and other neutron techniques, especially neutron spin echo, will provide access to the low frequency dynamics essential in understanding the response of systems to a range of perturbations.

Self-assembly

Self-assembly, involving lyotropic mesophases, micro-emulsions and vesicles of surfactants and amphiphilic block copolymers, is of interest in a wide variety of fields, from pharmaceutical drug delivery, encapsulation, efficient safe extraction processes and enhanced oil recovery. The increased flux available on the second target station will enable the dynamic processes associated with structural phase changes, association and dissociation to be studied. Aggregation and self-assembly in super-critical fluids are now important areas for the development of safe alternative solvents

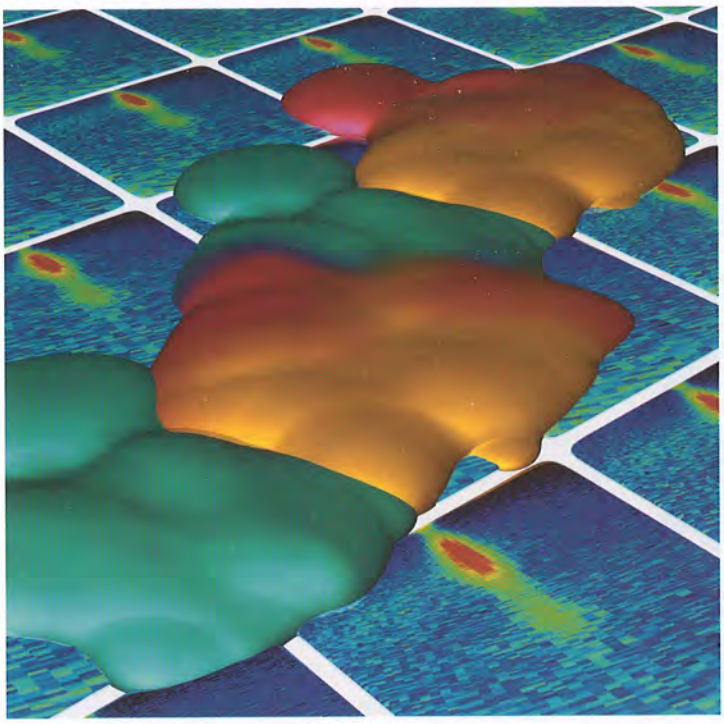




in flavour extraction, dry cleaning, controlled polymerisation; in-situ studies over this broad area will become increasingly possible.

In-situ electrochemistry

In-situ studies of electrochemical processes and the electrode surface will yield important structural information relating to transport and reaction processes. A variety of characterisation techniques provides aspects of structural information on electrochemical responses in-situ on time-scales from sub-second to minutes. This provides the opportunity to correlate structure with (redox) composition on a dynamic scale (for example, during a redox transformation). It is now important to offer neutron reflectometry information on such timescales, to give additional insights into a wide variety of electrochemical interfacial processes such as conducting polymers, redox polymers and metal oxides. Measurements of lateral inhomogeneity (using off-specular scattering) will give an insight into the nature of inhomogeneous deposits and rough surfaces found in real electrochemical applications. The impetus is to obtain information on an increasingly wider range of lateral dimension scales from Ångstroms to microns.



Thin film devices

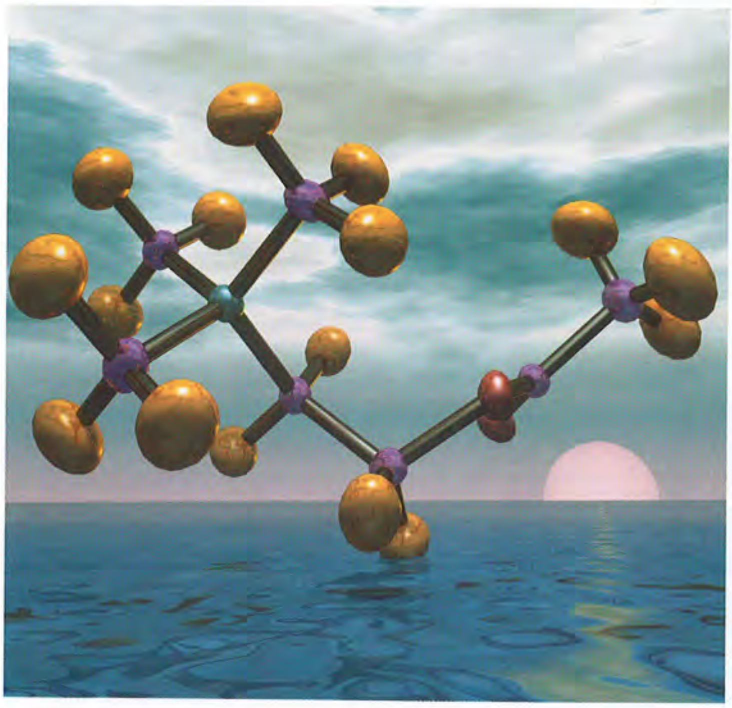
There is a range of emerging technologies, based on conducting polymers, which are rapidly showing potential for new thin film devices. These include polymeric-based photo-voltaics, light-emitting diodes and field effect transistors, and potentially a polymer-based laser. All these structures are multilayer devices and have complex morphologies which maximise interfacial areas, and the nature of the interfaces is critical to device performance. Both SANS and Neutron Reflectometry are the appropriate characterisation techniques.

There will be new and exciting prospects for the development of techniques and instrumentation. The further development of high resolution spectroscopy, and especially neutron spin echo to probe the scales from a few nanoseconds to greater than a few hundred nanoseconds, will provide unprecedented access to slow relaxation phenomena in systems such as polymer melts and blends. The possibility of combining neutron spin echo and a grazing incidence scattering geometry will provide an opportunity to probe the low frequency dynamics of surfaces and interfaces. Probing in-plane structure or inhomogeneities on a range of length scales will be possible using in-plane diffraction, off-specular scattering and grazing incidence SANS. There is a real need to extend off-specular measurements (currently probing length scales in microns) to shorter length scales. Investigating lateral structures at such length scales in ultra-thin films using SANS requires the increased flux that will be available. The response of the systems to a variety of external stimuli is a growing field; systems undergoing cyclic processes such as oscillatory flow, stroboscopic measurements, stop-flow measurements and responses to a range of other parameters such as pH, pressure and temperature.

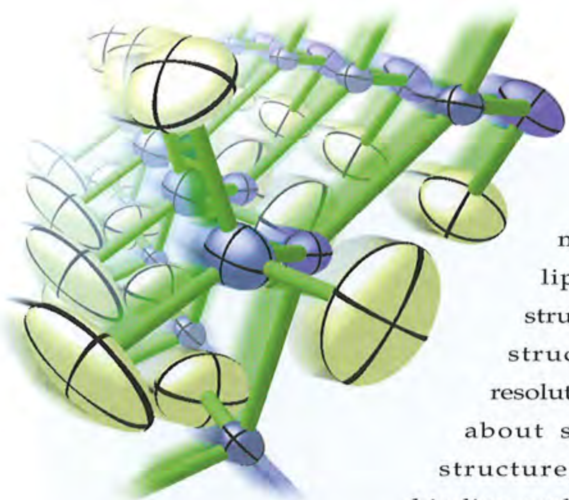
Bio-molecular Sciences

The Life Sciences are currently making an immense impact, with many health-related issues underlying this importance. The success of X-ray crystallography in solving complex protein and virus structures to high resolution has been critical for understanding structure-function relationships, and this has been further boosted by the Human Genome Project and the prospects of post genome research on a large number of newly identified protein sequences. The role of neutron crystallography is secondary to these high resolution X-ray studies. However, there are important contributions that neutrons will make in, for example, determining water or hydrogen location at lower resolution.

In the broader context of the biological remit, in the fields of pharmacy, food science, sensors, bio-compatibility and bio-functionality, there is much similarity with the problems in Soft Matter; and here SANS and Neutron Reflectometry are poised to make an impact. Indeed, one of the exciting features of Bio-molecular Sciences is the diversity of important issues and problems. In the last UK Technology Foresight exercise, in the areas of Health and Life Sciences, Food and Drink, Agriculture, and Natural Resources and Environment, priorities in drug creation and delivery, pharmaceuticals, metabolic pathways, immune manipulation, material changes during processing, and pesticides, are all closely linked to understanding membrane structures, membrane-protein interactions, the structure of macromolecular complexes, aggregation in gels and emulsions, surface adsorption and structure, self-assembly, bio-compatibility, and bio-functionality.



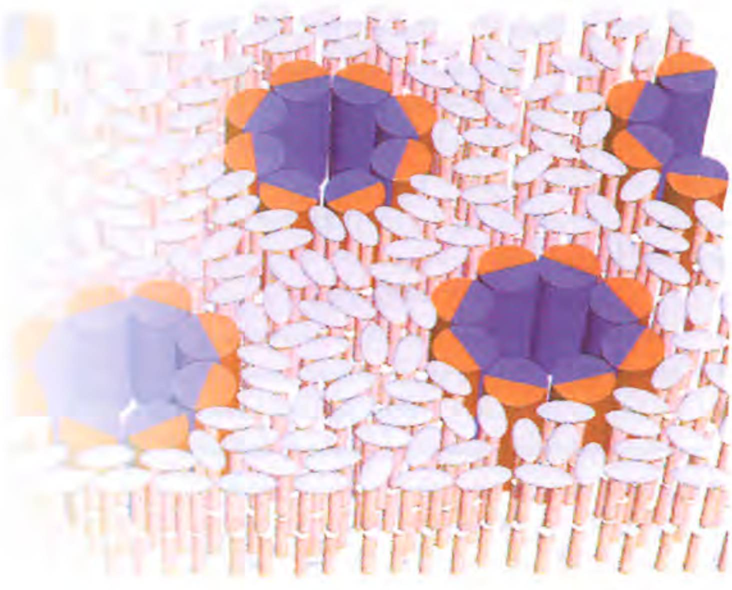
There is a small but established usage of neutron scattering in the Biological Sciences on which the enhanced capabilities of the Second Target Station can build. Neutron scattering can give either low resolution information about large features, such as lipids surrounding



a macro-molecule, micelle structure, lipid-membrane structure, or vesicle structure, or high resolution information about solvent water structure, hydrogen binding and precise active site geometry in proteins. Neutron scattering is the method of choice for the highest resolution studies of small bio-active molecular systems, neuro-transmitter molecules, energy-rich molecules and pharmaceutical materials. Neutron reflectivity has also shown its potential for the study of model membranes, lipid-protein interactions and surface adsorption / structure. Advances in membrane - protein interactions and protein folding will impact upon our understanding of a variety of diseases, such as HIV, Parkinson's and Alzheimer's. Some of the diverse areas are highlighted in more detail below.

Macromolecular assemblies

Low resolution studies, using SANS, are already established as an important method for characterising the conformation and functionality of macromolecular assemblies or complexes, where only the detailed crystalline structure of fragments or components is known, not the overall configuration (the highlight in the previous section on the structure of immunoglobulin A1 and its impact on functionality is a good example of the potential of the technique). The development and synthesis of new macromolecules, with known secondary or tertiary structures, will benefit enormously from a greater application of such methods. The importance of larger complexes, such as the binding of antigens to large antibodies like IgM, and the link between protein folding/unfolding and the formation of fibrils in amyloid-based disorders (Parkinson's, HIV, Alzheimer's) stresses the need for mesoscale structural information. Extending SANS to lower Q's, whilst maintaining the wide Q-range, provides an exciting opportunity for neutron scattering to probe larger complexes with improved spatial resolution.



Interfaces and membranes

Grazing incidence and conventional diffraction will provide information on the in-plane organisation of membranes and membrane-protein systems; using isotopic labeling and the higher fluxes available, higher resolution information will be accessible. Specular reflectivity in conjunction with isotopic labeling of component lipids will substantially enhance our understanding of membrane structures, membrane-protein mechanisms and changes in conformation of peptide molecules loaded into bilayer membranes. Analogous surface structural details and the characterisation of the adsorption of proteins and lipids at interfaces will be used in the development of bio-compatible and bio-functional surfaces, and in the biochemical and biomedical engineering of surfaces.

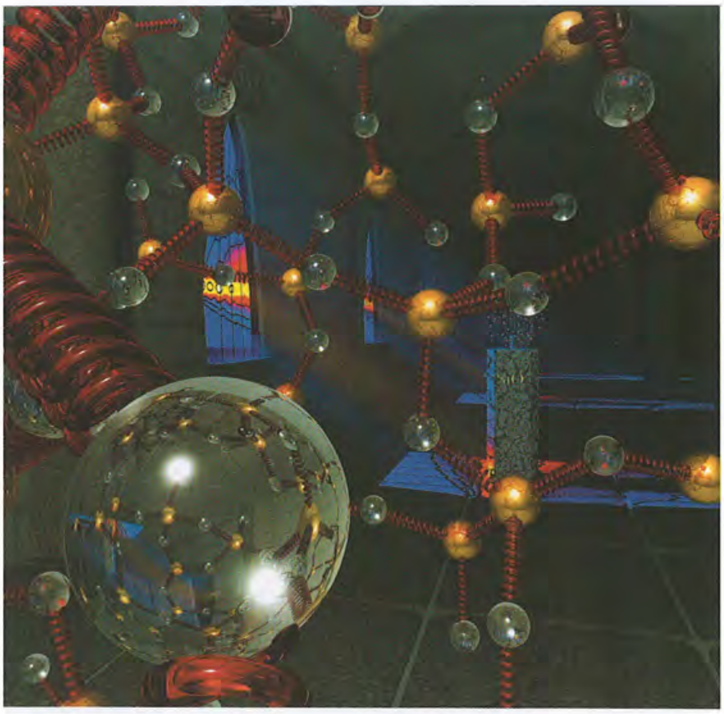
In pharmaceutical applications, the highly detailed information that will become available on self-assembled molecular layers and aggregates will be exploited in the design and optimisation of systems for drug encapsulation and delivery, and through membrane-protein interactions and drug mechanisms.

Food science and technology

Although practical systems in food science are extremely complex, SANS will permit the study of solvent distribution and material changes (denaturing) during processing, and considerable progress can be made on relevant model systems. Kinetics is an extremely important issue central to the understanding of food colloidal stability, protein fouling during processing (the competitive inter-play between small molecules and proteins) and in the structural changes that occur during processing. The kinetics of these processes are difficult to access using other techniques. The flux available on the Second Target Station will make a wide range of these in-situ kinetic studies accessible for the first time. The nature of the adsorption of proteins and lipids is key to the understanding of colloidal stability, and foam and gel formation, and the necessary parametric studies will be possible.

Low and high resolution crystallography

The anticipated flux increases will revolutionise low and high resolution crystallography, making studies on membrane protein-detergent crystals, organisation in virus particles, the structure of glycoproteins, protein folding, and other macromolecular complexes (such as protein-nucleic acid interactions) routine at ISIS. In particular, from high resolution studies the specific location of critical hydrogen atoms and hydrogen bonding interactions, and



other functionally important water molecules or other solvent molecules, will be determined. The vast improvement in data collection times will allow, for the smaller macromolecules, accurate determination of solvent structures, temperature dependent studies, and investigation of active site interactions.

The massively enhanced flux of cold neutrons will have an important impact on the use of single crystal determination of new drug structures, where the hydrogen atoms are so crucial in understanding the mechanisms, energetics and electrostatics of drug-receptor interactions. Neutron crystallography is an important 'niche' market, which will effectively complement X-ray measurements where detailed information on hydrogen atom location and mobility is required.

In many cases the potentially important experiments in these areas are flux limited; many of the systems of interest are weak scatterers, and there is a need to study dilute systems, kinetics and small samples, and to make parametric studies. The enhanced flux of cold neutrons and the wide simultaneous coverage in Q that are anticipated will provide an exciting platform for the increased application of neutron scattering techniques in the study of membranes, pharmaceuticals, macro-molecular assemblies and food technology.

Advanced Materials

The characterisation of Advanced Materials using neutron scattering, including new magnetic materials, composites, the use of extreme conditions, nano-scale structures, complex materials such as zeolites and novel electrolyte materials, requires both high resolution crystalline diffraction methods and non-crystalline diffraction.

Crystalline materials

More new materials have been synthesised in the past 20 years than in the rest of history. During this period there have been two strands in materials development, increasing complexity and increasing subtlety, involving competition between magnetic, electronic and structural ground states.

High resolution diffraction with enhanced performance will impact on the investigation of a broad range of novel structures in solid state chemistry and advanced materials:

- GMR/CMR materials, the characterisation of mixed metal oxides of manganese which show a marked change in electrical resistance with applied field.
- Complex structures of small samples; structural molecular materials.
- Novel optical materials: photonics, opto-electronics, non-linear optics.
- Ferro-electric materials: relationship between macroscopic properties and subtle crystallographic changes.
- Battery materials, including work both on electrodes and electrolytes, along with in-situ studies.
- Catalytic materials, including micro-porous solids.
- Non-stoichiometric solids, the link between defect structure and electronic properties.
- Disordered crystalline structures, incommensurate materials and quasi-crystals.

- Ionic conductors, location of Li^{+} ions, cation distributions in ternary sulphides, and correlation of structure with magnetic and electronic properties.

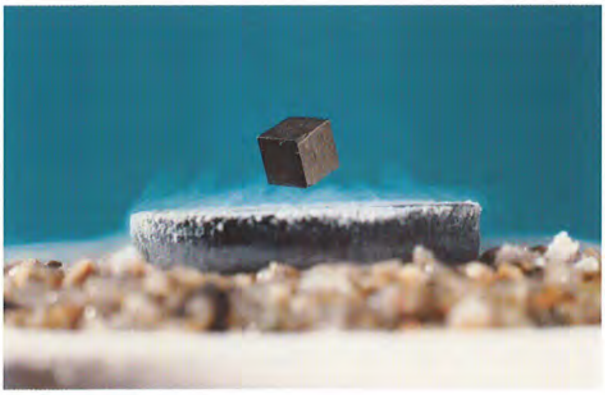
- Molecular sieves, zeolites, alkali metal chemistry, catalysis and ion-exchange, clathrate formation, and quantum wire and dot systems.

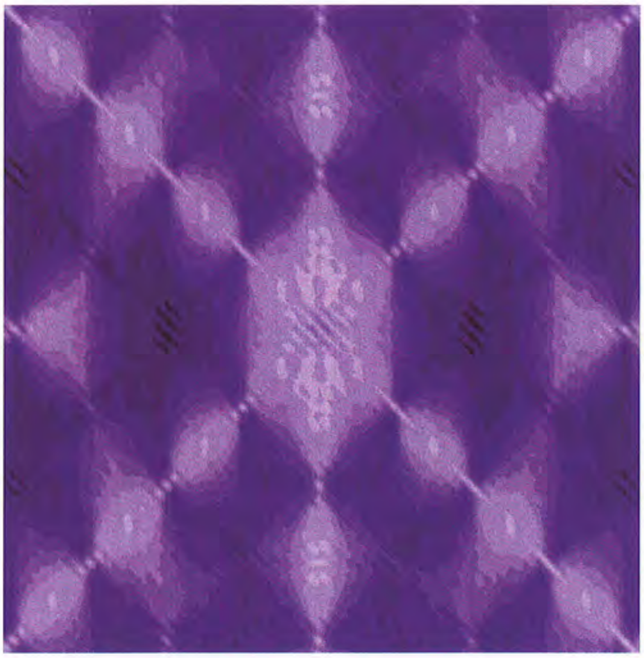
- Composite multi-crystalline materials, such as mixed geological phases.

- Geological specimens, in addition to good structural resolution, require good spatial resolution to scan through inhomogeneous specimens.

- In-situ studies of rock deformation; relating stress and strain tensors to the development of earthquakes.

The flexibility of sample containment for neutron diffraction leads to a natural exploitation of extreme or exotic sample environments. Temperatures from milli-K to thousands of K and pressures of tens of GPa are routine, but there is an increasing tendency to combine such extremes for the study of materials such as super-ionic conductors. For high pressure work, reducing the required sample volume naturally leads to the ability to reach higher pressures (for the study of the high pressure phases of ice, metal-insulator and superconductor transitions, and synthetic

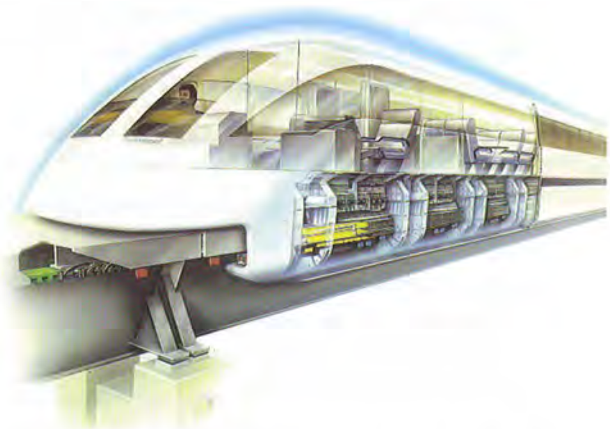




geological specimens). The possibilities offered by optimised high flux are obvious, but in the case of magnetism at high pressures, an area under-exploited to date, the optimised cold neutron flux and the specific provision of very long wavelength, high resolution diffraction facilities, will dramatically widen the horizons.

The penetrating power of neutrons makes the use of bulky equipment to generate a range of extreme environments more tractable, and, for example, provides the opportunity to study real, reactive chemical systems in-situ. Such methods will allow a wider range of materials to be synthesised in the beam, giving the opportunity to identify reactive intermediates. Hydro-thermal cells and catalytic rigs will be exploited further. There would be impact in areas such as sol/gel processing of ceramics, including piezoelectrics; CMR/GMR materials; high T_c materials; in-situ hydro-thermal processing of fine particles for pigments and recording media; in-situ growth techniques like CVD; kinetics of chemical reactions; and intercalation reactions. The pressing need here is to reduce data collection times in order to match the characteristic time of the reaction under study.

Development of stroboscopic measurements, with switchable electric and magnetic fields, would enable potential applications for the investigation of switching in ferro-electrics and in the study of piezo-electric transducers.



Magnetic materials

Virtually everything we know about magnetic structures has been revealed by neutron diffraction, and neutrons will continue to be the method of choice for studying new magnetic materials. This is true not only for the field of magnetism, with its fundamental and technological implications, but also for our understanding of the structure and electronic properties of materials.

The opportunities offered by the improved performance for high-resolution diffraction and spectroscopy will allow an increased impact in many areas:

- Metallic, organic and ionic magnetic materials, many of which show ordered, often incommensurate, magnetic structures with small moments and large unit cells.

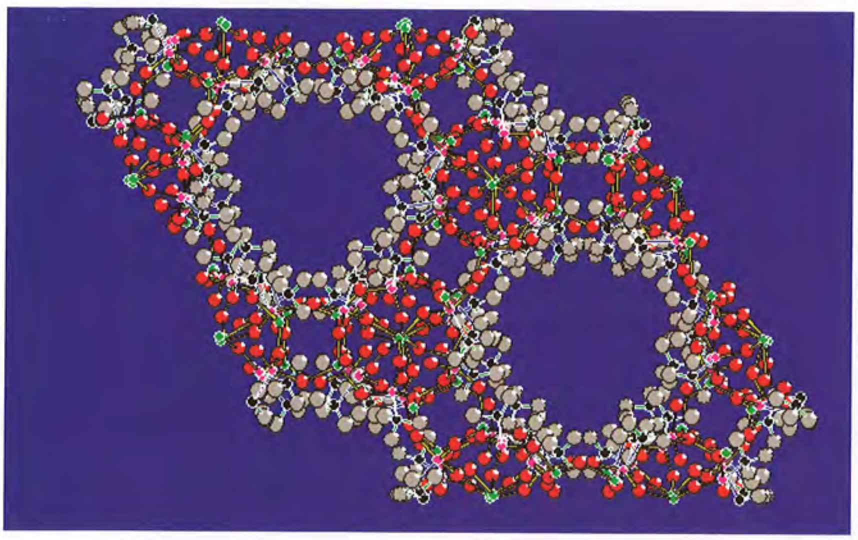
- Materials with novel ground states, such as low dimensional or frustrated systems, or that exhibit multipolar or orbital ordering, where magnetic scattering is often weak and/or diffuse.

- Processing routes of advanced permanent magnetic materials by in-situ kinetic diffraction studies.

- Strongly correlated electron systems, such as high temperature superconductors, CMR perovskites and non-Fermi liquid materials, where parametric studies are often important.

- Nanocrystalline materials: influence of high interface:volume ratio on local magnetisation, and nanoscale magnetic correlations in relation to the microstructure and the interface.

In many of these areas moments are small or delocalised, unit cells are large, the magnetic scattering is weak and samples are small, and an enhanced instrument performance is essential to make a significant impact.



Artificial structures of nanoscale dimensions are key model systems for understanding the fundamental properties of matter. At the same time they form the basic building blocks for new high technology industrial products with applications in improved magnetic recording devices and erasable

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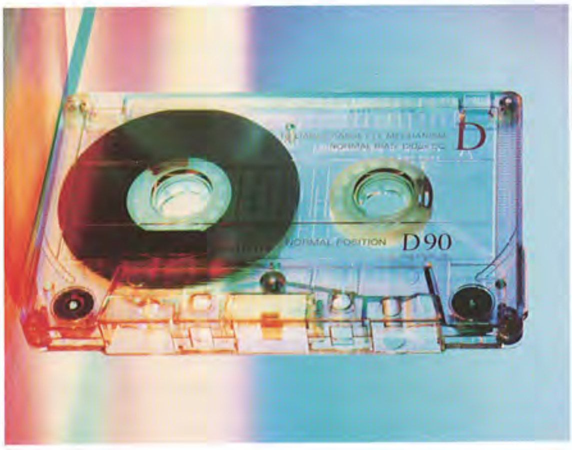
recording systems with extremely high storage capacities. Neutron Reflectometry has already demonstrated that it can contribute unique information in this field. The Second Target Station will turn NR into a mature technique for understanding and optimising systems based on spin electronics devices (polarised electron transport in spin-engineered structures such as spin valves, GMR multilayers or manganites). Reflectometry studies in-situ and in real time will become possible to follow surface oxidation, magnetic domain relaxation, in-situ growth deposition, in-plane and multilayer dynamics. Tunneling magneto-resistance (TMR) devices, based on ferromagnetic/insulator/ferromagnetic structures, are likely to replace GMR materials in applications such as MRAM. The applications of such structures are the driving force for understanding the spin dynamics in these materials. Off-specular scattering at grazing incidence and SANS will provide information on nano-structures, in-plane ordering, patterned substrates and domain structures (where depolarisation tomography will also be important).

Inelastic neutron scattering is unique in its ability to provide definitive information about the lifetime and extent of correlations between magnetic moments. Inelastic magnetic scattering is therefore a vital tool for the investigation of materials ranging from model magnetic systems, systems with novel ground states through to high



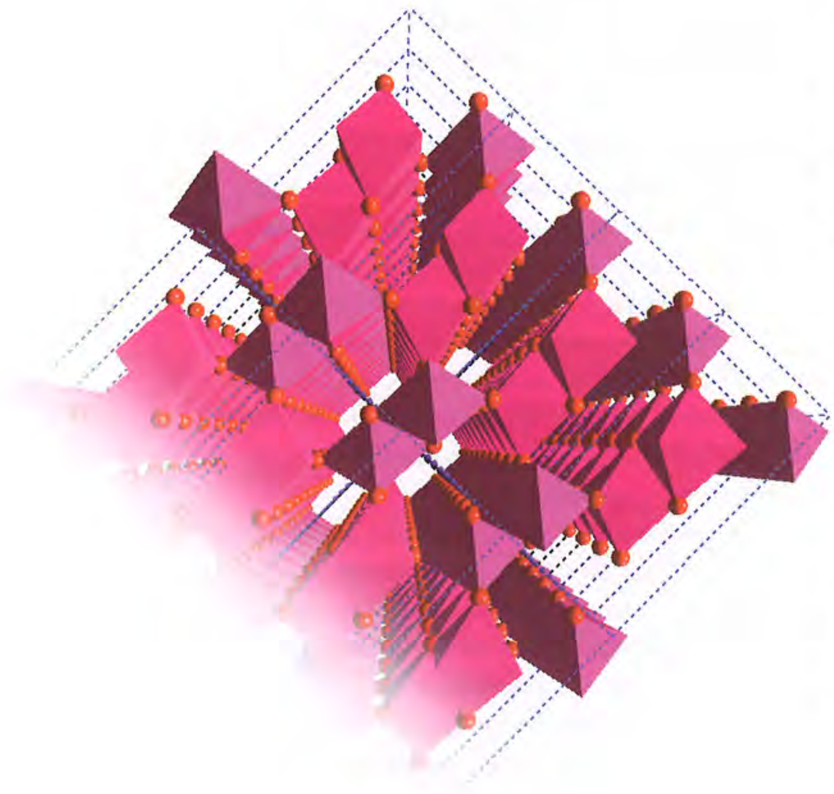
temperature superconductors and colossal magnetoresistive materials.

Magnetism arises from complex many-particle interactions. Materials where magnetic interactions are confined along a chain or in a plane, makes theoretical and numerical investigation more tractable. Neutron studies of such model systems provide an opportunity to test theory against experiment and consequently provides a vital insight into the magnetic behaviour of more complex 'real' systems. The most interesting model systems are those where the interaction strength, and hence the energy scale of the magnetic fluctuations, is comparable to the strengths of magnetic fields and temperatures that can easily be applied. This is the cold neutron energy range that where instruments on the second target station will offer a high neutron flux and excellent resolution.

The colossal magneto-resistance (CMR) perovskites have many potential applications including sensors and spintronic devices. Knowledge of the magnetic fluctuations on energy scales corresponding to typical magnetic fields in which the CMR phenomena are observed is vital to understand their origins. The energy scale of the fluctuations near the M-I transition in layered managnites, for example, is of order 1 meV, so low energy inelastic neutron scattering is the ideal tool. Despite much progress over many years, the origin of high-temperature superconductivity remains an unsolved problem. It is widely believed that the antiferromagnetic fluctuations are an important ingredient, and further exploration of the spin dynamics from μeV to several hundred meV is required to provide further insight. Furthermore, studies of the flux line lattice and its movements through a material under an applied current will provide important information, in particular for technological applications.

The second target station will have a significant impact on our ability to study the dynamics of magnetic materials, not just because the energy scales of the neutrons and excitations are well matched, but because of the unique nature of instruments at a time-of-flight source. They enable data to be gathered simultaneously over a wide range of momentum and energy transfer; a capability that is extremely powerful for studying both magnetic and structural excitations in novel materials.

In both elastic and inelastic studies, the provision of polarised beams (and polarisation analysis of the scattered beam) is of central importance. Magnetism and polarised beams are inextricably linked, and this has important consequences for the potential instrument suite.



Disordered materials

Many of the potential developments in Advanced Materials involve disordered systems, which are characterised by short range order and a poorly defined long-range structure. Non-crystalline diffraction and the use of H / D isotopic substitution on simple fluids with spherical molecules is well established, and fluids such as argon and methane are now well understood. Examples of complex disordered materials for which neutron scattering is now starting to provide important structural information include lubricants, polymeric glasses and solutions, battery electrolytes, metallic glasses, network glasses, complex ionic solutions near the critical point, super-critical fluids, solvent mixtures, and liquids under confinement (in porous or intercalated media). Isotopic substitution, the ability to use complex environments, the minimisation of inelastic corrections for light elements by using high energy neutrons, all make neutron scattering a particularly powerful tool for the study of local order in complex disordered materials. The wide Q range available in a single measurement makes a wide range of length scales, from 1 Å to 100 Å (and even larger) accessible. In the complex systems that are increasingly being studied, understanding structure/function relationships require us to follow simultaneously not only molecular level structure, but also larger length scale density fluctuations and inhomogeneities that are central to the materials' behaviour. This applies to structural changes occurring in molecular and forced processes (during manufacture). This requires a Q range from 0.01 to 10 $Å^{-1}$ and a resolution of <1%. This is often accompanied by extensive use of isotopic substitution which is only limited by the statistical quality of the data, and high count rates over the broad Q range are necessary.

Particular attention is drawn to the following areas:

- Disordered crystalline materials; mesoscale consequences of molecular-level processes.

- Nucleation and growth processes, including solid growth from solid (precipitates), solid from liquid (crystallisation), liquid from liquid (phase separation), pre-nucleation, self-assembly.

- Monitoring in-situ novel processing routes for new disordered materials, such as the sol-gel processing of ceramics. Ability to accommodate extreme or complex environments, such as high temperatures and pressures, and reactive conditions.

- Phase separation in glasses.

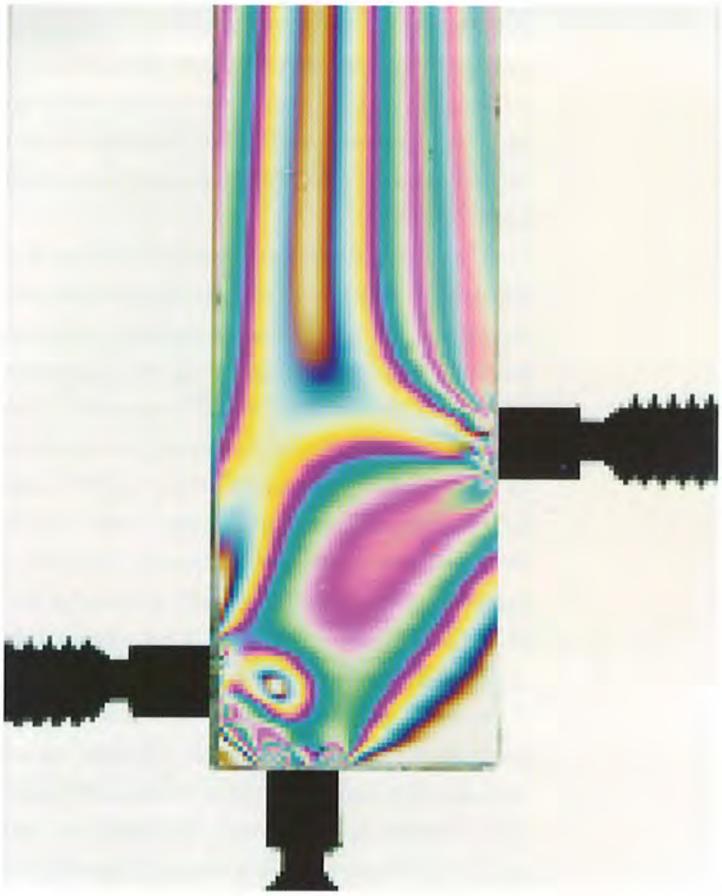
- Probing longer length scales nearer critical regions in a variety of liquids.

- Electronic liquids, containing larger length scale inhomogeneities associated with molecular clustering.

- Structure and phase transitions in two-dimensional fluids; fluid structure in phases relevant to oil recovery; waste disposal.

Many of these broad areas will impact directly on important issues in chemical engineering, such as catalysis, combustion science, separation technology, and liquid (especially oil) recovery.

Inelastic scattering investigations, for molecular vibrations (with timescales up to 10^{-15} s), through diffusive motions (10^{-11} to 10^{-9} s), down to slow relaxation process in glass transitions (~ 1 s), will also play an important role in the investigation of complex disordered materials. Here there is a particular need for high resolution (< 1 meV) over a broad range of momentum and energy transfer, and neutron spin echo for the investigation of very slow processes.



Engineering materials

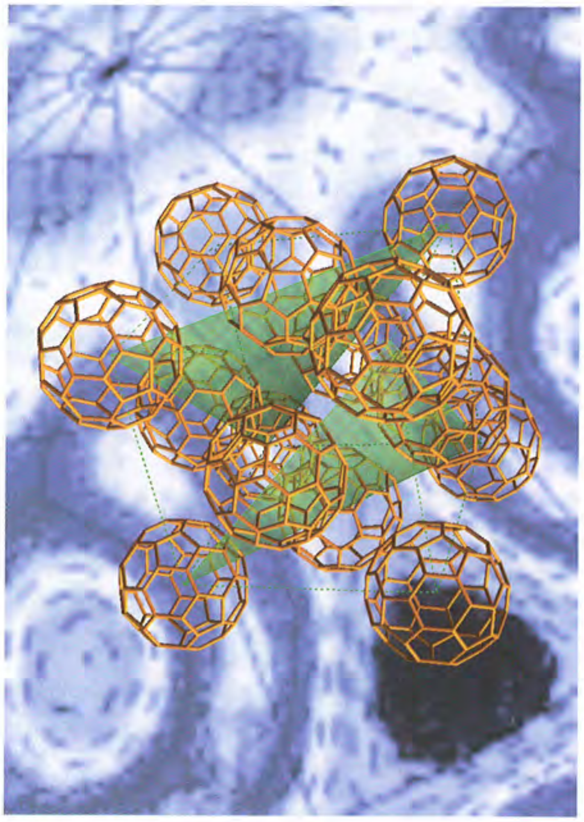
It has already been demonstrated that high resolution powder diffraction can provide a quantitative map of strain in engineering and technologically important components. Strain experiments can be broadly divided into two areas - those requiring high intensity and moderate resolution and those requiring high resolution capabilities.

Improvements in flux would allow an improvement in both the size of gauge volume, and/or in the time taken to collect data. Improvements in data collection rates are particularly important for in-situ investigations e.g. for the optimisation of heat treatments, or potentially in-situ processing. Monitoring of cooling from fabrication temperature, isothermal creep and thermo-mechanical fatigue are all of importance to real industrial components.

With smaller gauge volumes, a range of investigations can be simplified, namely residual stress measurements in coatings, buried interfaces, rivets, complex stress fields and the investigation of surface treatments. Novel joining techniques, such as the friction stir welding of large aluminium alloy sheets which will be vital for construction of the next generation of super-jumbo civil aircraft, need optimisation and characterisation. The detailed mapping of residual strains in joined materials such as ceramics-to-metals and glasses-to-metals would also be of considerable use to industry.

Areas of study requiring high resolution include microstress measurements. A high resolution strain measurement instrument would also allow accurate peak width deconvolution to provide stress/strain distributions in, for example, ceramic composite systems, as well as studies of chemically active, electrostrictive or fibre reinforced materials, or studies of shape memory/super-elastic phase transformations. Other phase transformations which are sensitive to stress occur in read/write optical disc materials.

Inter-granular stress measurement on metal-matrix composites such as Al:SiC,

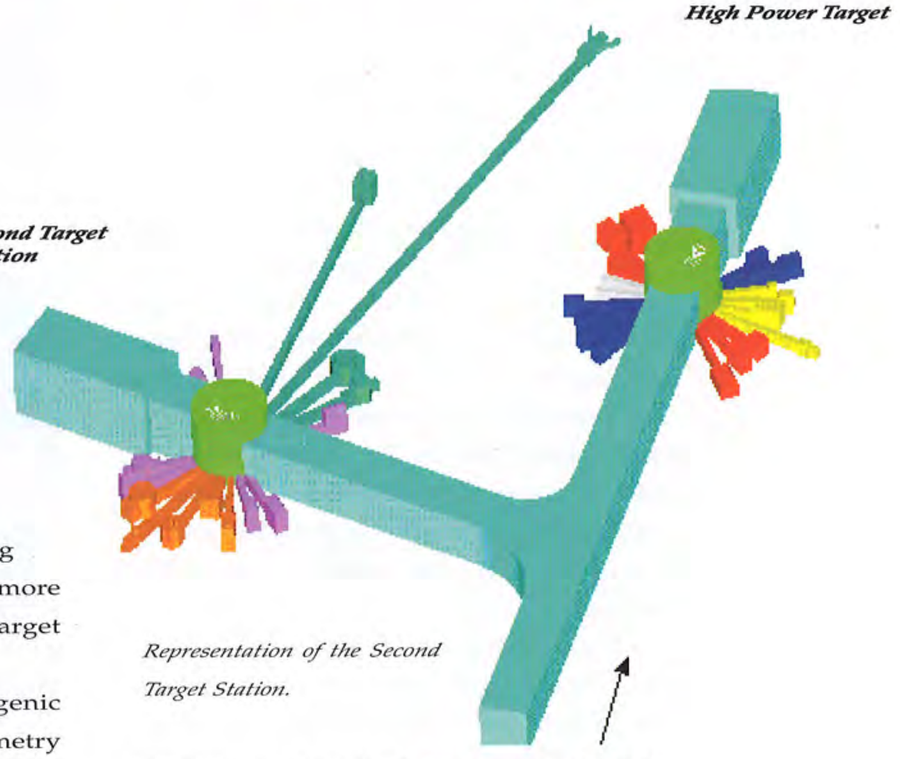


ceramic composites such as Al_2O_3:SiC as well as alloys, provide details of stresses in the respective phases or crystallographic directions, and thus insight into the deformation mechanisms involved. For example, the drive to measure intergranular stress in zirconium alloys, widely used in the nuclear industry, comes from the fact that creep and grain growth in a radiation environment are strongly dependent on texture and stress within the crystallites. Recently, interest has also grown in the effects of thermomechanical treatment such as extrusion, rolling or creep loading.

Other systems include layered materials such as ceramic multilayers (e.g. Al_2O_3:ZrO_2) which offer potential improvements in operating temperature when used for burner casings in the exit chambers of jet-engines. These multilayers exhibit crack growth resistance due to internal stresses. Coatings on more conventional structural materials, designed for wear or to resist high temperatures, are also of considerable interest.

Technical description of the Second Target Station

Introduction



Representation of the Second Target Station.

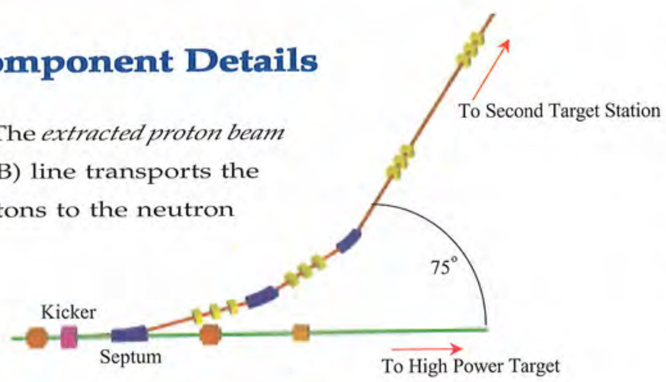
The Second Target Station will be situated on the southern side of the existing High Power Target. One pulse in five from the ISIS synchrotron will be directed into the target along a new proton beam line. The relatively low proton power will allow target and moderator designs which are optimised for the production of long wavelength neutrons and hence are more efficient than those on the existing target station.

A tungsten target with two cryogenic moderators, positioned in 'wing' geometry relative to the target, will be used to produce the neutron beams. A beryllium reflector will surround the target and moderators, and the core target components will be contained within a cylindrical void vessel surrounded by shielding. Eighteen neutron beam ports will penetrate the shield, some of which will feed two neutron guides and so serve two instruments.

Shown top right is a plan for the Second Target Station layout, including the beam lines and, schematically, the new instrument suite

Component Details

The *extracted proton beam* (EPB) line transports the protons to the neutron production target in the experimental hall. The new EPB will be extracted horizontally, at 75° to the south side of the existing one using a small angle pulsed kicker and a dc septum magnet.



The Extracted Proton Beam line.

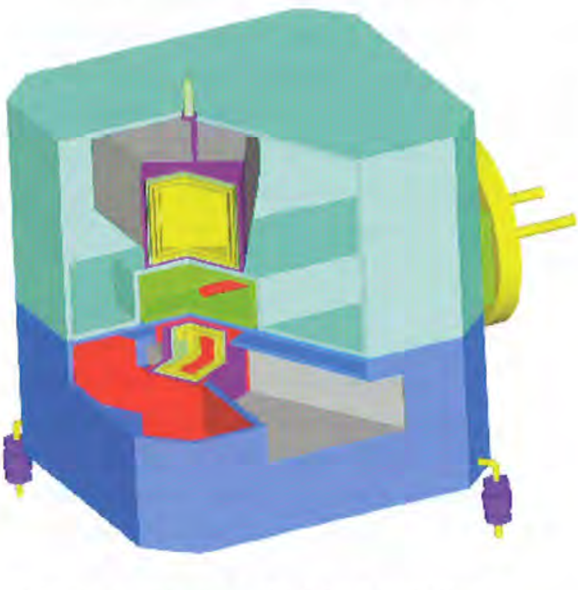
The *target* produces neutrons by the spallation process resulting from the interaction of the high-energy (800 MeV) proton beam and the target nuclei. The target will be a 50 mm diameter tungsten rod clad with tantalum to eliminate corrosion problems and cooled by heavy water. The low power of the proton beam for the Second Target Station will enable a small diameter target to be used, giving excellent coupling between the leakage neutrons and the moderators.

The *moderators* reduce the energy of the spallation neutrons to energies useful for the experimental programme. The dimensions, moderator material and temperature of each moderator for the Second Target Station have been chosen to match instrument requirements. The proposed configuration consists of two moderators:

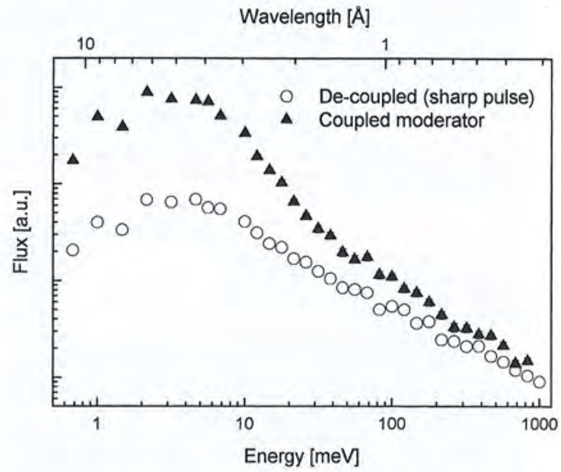
- 25 K decoupled, solid methane with an asymmetric poisoning layer producing broad and sharp pulse widths.

- 25 K coupled liquid hydrogen.

The complete target, moderator and reflector assembly is shown below. The use of solid methane is only possible at low proton beam powers - the effects of radiation damage at high powers make its use there impractical. The coupled liquid hydrogen moderator will be in 'wing' geometry with two viewed faces to give maximum intensity to those instruments for which a relatively wide time pulse is acceptable. The solid methane moderator, also in wing geometry, will be decoupled with an asymmetric poison foil which will give beam characteristics similar to the hydrogen and liquid methane moderators on the existing target station but with enhanced flux. The *reflector* scatters fast neutrons back into the moderators. It consists of a stainless steel vessel filled with beryllium rods, cooled



The target, moderator and reflector assemblies.

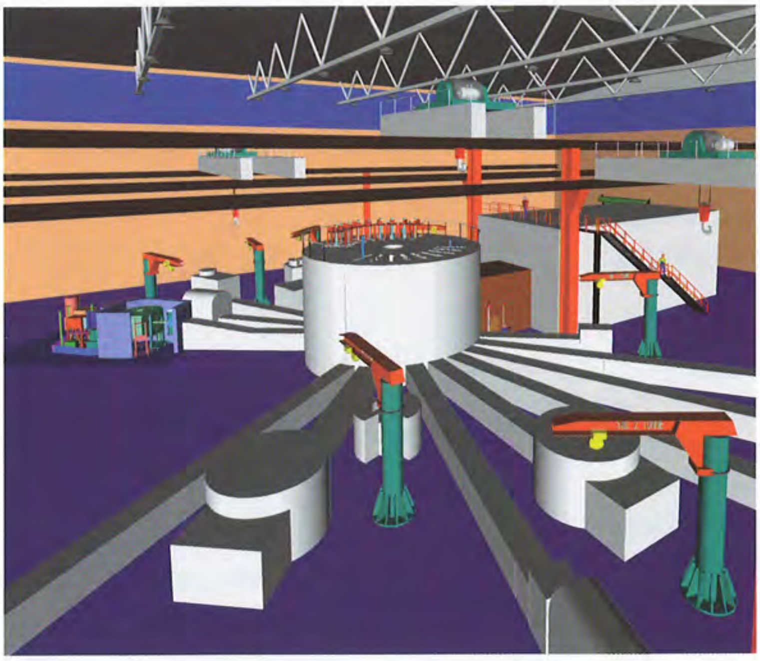


The figure shows some of Monte-Carlo calculations of the target/moderator assembly. The triangles are the flux from a broad pulse coupled moderator and the circles show the flux from a sharp pulse moderator suitable for the instruments that require high-resolution.

using heavy water.

The *void vessel* provides a controlled environment for the target and acts as a containment vessel in the case of a target component failure or water leak. The helium-filled vessel will be embedded within a 12 m diameter, 7 m high steel and concrete *shield*. The shielding will be designed to allow vertical access to the proton beam window, the neutron beam windows, the upper reflector assembly and the shutter assemblies. The target and lower reflector assembly will be installed and removed horizontally. Eighteen neutron beam holes, nine on each side of the target spaced 13° apart, will be provided through the shielding to allow the neutrons to travel to the instruments, and each beam hole will be equipped with a vertical steel and concrete shutter to open and close the beamline. The shielding will be constructed from cast and machined steel blocks with a poured concrete outer layer. To reduce soil and ground water activation to acceptable levels, there will be 2 m of steel and 1 m of concrete shielding below the target. This will also serve as the foundation to support the 8000 tonne load.

SPECIFICATIONS

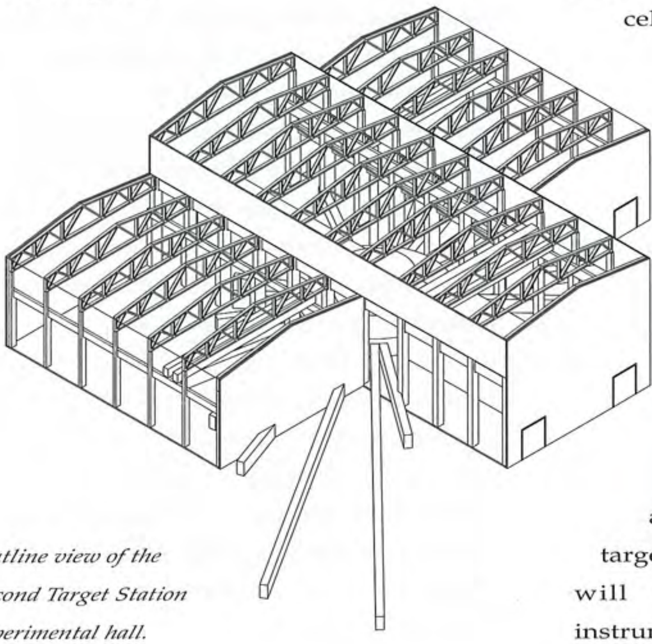


A view of the Second Target Station experimental hall.

Fundamental to the design of the Second Target Station is the ability for all principal target components (target, reflector and moderators) to be removable, to enable their replacement within a period of one week. This requirement dictates the design and configuration of the core region of the target station. The expected working life of some of the principal target components is shown in the technical specifications table opposite.

The Second Target Station has been designed to minimise the amount of remote handling through the use of modularisation and improved accessibility to components with a short lifetime. This includes vertical access in combination with containment flasks for frequently serviced components, and horizontal access and a dedicated remote handling cell for those components with a long working life. The remote handling cell will be similar to the existing ISIS cell but with some design changes to improve accessibility to core target components. The cell will be equipped with master slave manipulators and a remotely operated crane with positional readout. Lead glass windows supplemented by remotely operated cameras provide viewing for the remote handling.

The controlled handling enclosure above the target station will be served by a 100 tonne crane. The building roof will be raised in the middle to allow for the overhead handling of target components. Two 30 tonne cranes will serve the neutron scattering instruments.

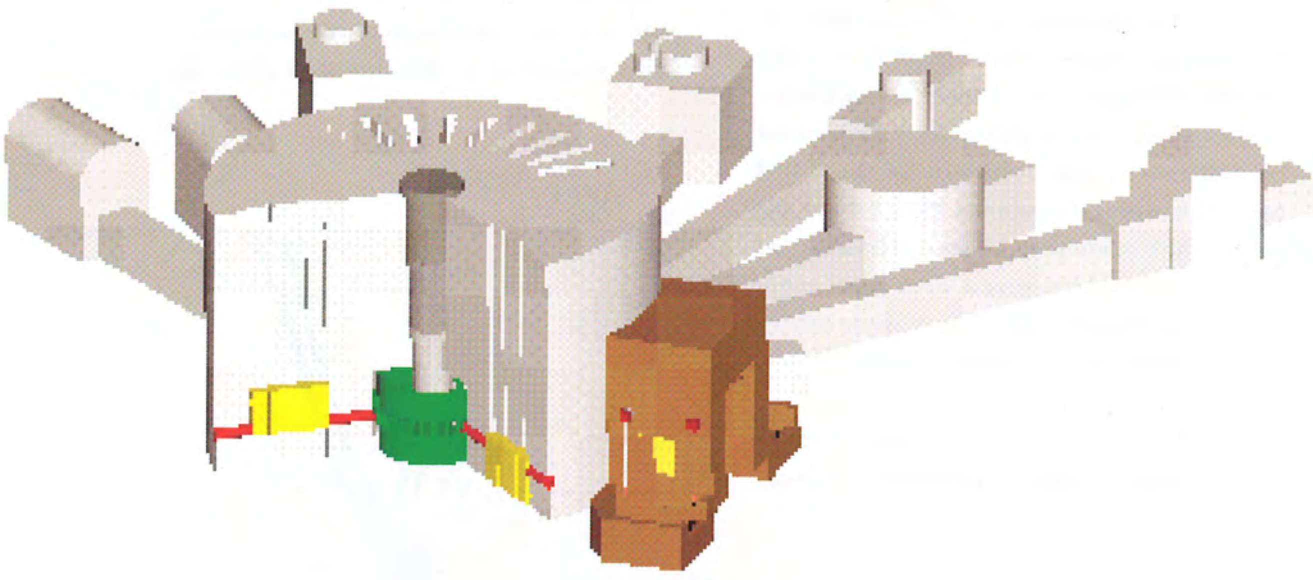


Outline view of the Second Target Station experimental hall.

SPECIFICATIONS

Proton beam	repetition rate	10 Hz
	proton extraction angle	75°
	current	60 μA
	power dissipation in target	48 kW
	proton beamline shielding	1.5 m steel + 0.75 m concrete
	beamline outer surface dose rate	<0.5mSv/hr
Target	material	tantalum clad tungsten
	size	25 mm diameter
	cooling	heavy water
	reflector	beryllium rods
	shielding	12 m diameter x 7 m high, steel + concrete, 2 m steel + 1 m concrete below target Surface dose <0.5mSv/hr.
Beam ports	number and spacing	9 on each side of the target, 13° separation
Moderators	25 K decoupled, poisoned solid methane	150 x 150 x 30 mm^3
	25 K coupled liquid hydrogen	150 x 150 x 50 mm^3
Component Service Lifetimes	Methane Moderator	6 months
	Tungsten Target	2-5 years
	Hydrogen Moderator	15 years
	Neutron Beam Window	15 years

Technical specifications of the Second Target Station.



View of the Second Target Station.

Instrumentation for the Second Target Station

The Second Target Station will offer a range of benefits for instruments:

- A wider dynamical range in momentum transfer for crystalline and non-crystalline diffraction, SANS and reflectometry, and in energy and momentum transfer for spectroscopy, by virtue of the increase of the time frame from 20 to 100 ms.

- Enhanced flux for long wavelength/ low energy neutrons arising from a fully optimised target/moderator system.

- A coupled cold moderator will provide a further enhancement of flux for those instruments that do not require a sharp pulse structure.

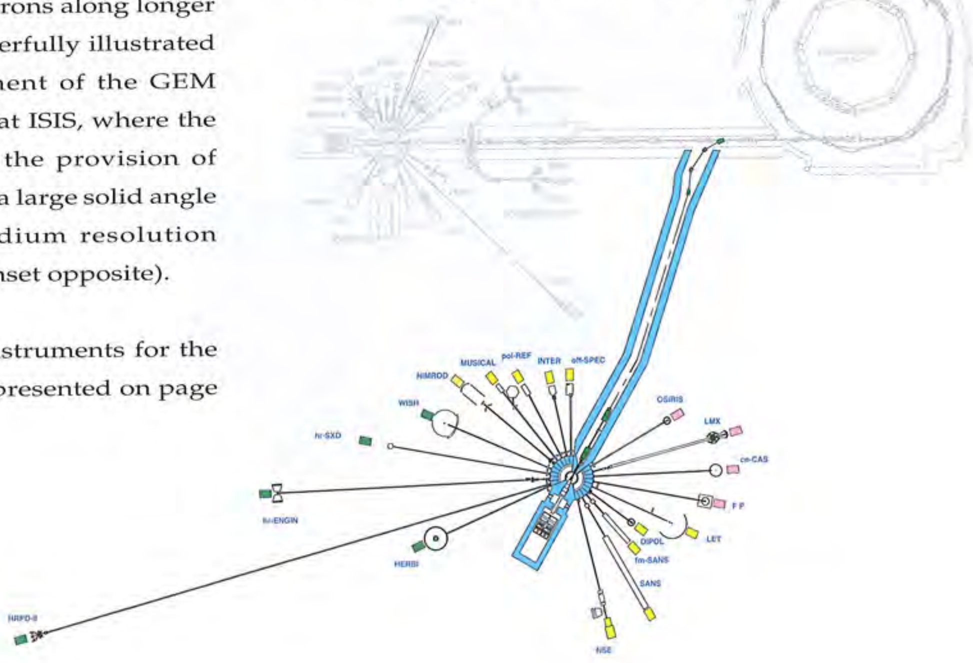
- The availability of new technologies provides the opportunity to enhance further the performance of new instruments on the Second Target Station. The larger time frame will allow resolution improvements through the use of supermirror guides for the efficient transport of neutrons along longer flight paths. This is powerfully illustrated by the recent development of the GEM Materials diffractometer at ISIS, where the significant advances in the provision of stable detectors covering a large solid angle has revolutionised medium resolution powder diffraction (see inset opposite).

A potential suite of instruments for the Second Target Station is presented on page 52.

Large Scale Structures

The broad pulse, high intensity beams of long wavelength neutrons available from a cold coupled moderator are ideal for reflectometery and small angle scattering, and these are the techniques which will benefit most from the construction of the Second Target Station. The longer time frame provides a wide bandwidth and a broad, simultaneous coverage in Q.

Two small angle scattering instruments are proposed: **SANS**, which will be a world-class instrument offering exceptionally broad, simultaneous access to Q space, and **fm-SANS** which uses a focusing mirror to provide access to very low Q and hence to length scales that overlap with light scattering measurements. Both instruments will benefit from the enhanced flux from a coupled moderator with relaxed resolution. **SANS**, however, will have an excellent resolution by virtue of a secondary flight path of 22 m, with the detector moveable to allow flexibility in the choice of resolution



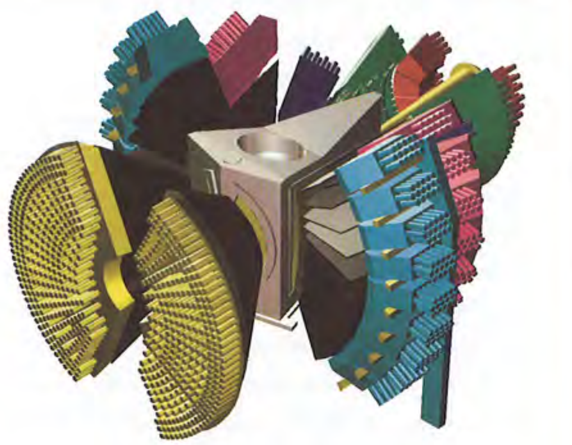
and Q coverage to suit particular problems. The novel incident beam optics used on **fm-SANS** will provide excellent access to low momentum transfers and extend neutron scattering to longer length scales.

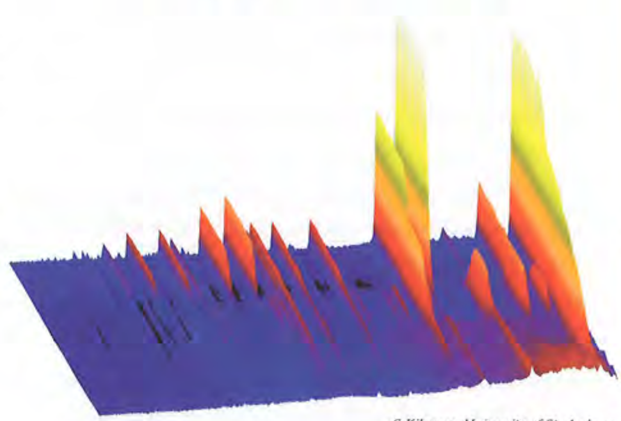
The proposed instrument suite includes three reflectometers: **INTER**, **off-SPEC** and **pol-REF**. **INTER** is optimised for studies of chemical interfaces. It has been designed to provide high flux and a wide bandwidth to allow for short data acquisition times and as a consequence will be well suited to time dependent measurements on the timescale of seconds. **off-SPEC** will offer similar performance in terms of flux and resolution, with a multi-detector suitable for vertical and horizontal scattering geometries. It will be optimised for off-specular scattering studies from a wide range of systems. Polarised neutron reflectometry has been demonstrated as a uniquely powerful tool for the study of magnetic and superconducting films and multi-layers. The polarised neutron reflectometer, **pol-REF**, will offer high flux and bandwidth like the other two reflectometers. Full polarisation analysis for both the specular and off-specular scattered beams will be available.

The scientific case highlights some specific development issues for instrumentation in this field. The optimisation of **off-SPEC** to probe lateral inhomogeneities on a shorter length scale (<< 1 micron), to measure efficiently in-plane diffraction, and to exploit grazing-incidence SANS, all require development. The combination of neutron spin echo and grazing-incidence geometry will provide unique opportunities for investigating low

The GEM Diffractometer

The performance gains that can be obtained by re-designing neutron instrumentation around state-of-the-art technology should not be overlooked. In fact, the recent experience of building the new high-intensity powder diffractometer GEM at ISIS (upper right figure) show that considerable gains can be obtained. In the case of GEM, the gain arises mainly from the very large detector surface area (12 m²). This was made possible by the development of series production of scintillator detector modules, in partnership with industry, and of fast electronics and data handling techniques. The lower right figure shows as example data set collected on GEM showing the crystallisation of magnetic Y_7Fe_3 from amorphous precursors as the sample is heated in a furnace. Each diffraction patterns was collected in 3 min, to be compared to the typical collection times of 1 or 2 hours. The two-step crystallisation process is captured in extraordinary detail, especially with reference to the structural characters of the individual crystallographic phases.





S Kilcoyne, University of St. Andrews

frequency dynamics at surfaces and interfaces, and will require a development phase.

Diffraction

The large time frame on the Second Target Station will provide a wide dynamic range and excellent resolution for diffractometers, and the following instruments are proposed: **HRPD-II**, **hr-SXD** and **hr-ENGIN**. These will all utilise the high resolution, combined with improvements in background to extend the scientific programme in the study of advanced materials. The relocation of the current HRPD to the second target station will be combined with upgrades to the instrument, providing significant improvements in both flux and resolution and enhancing its position as the world's highest resolution neutron powder diffractometer. **LMX** has been designed for the study of large molecules such as proteins. Focusing incident beam optics will give a high intensity beam for small samples, and a curved guide will reduce background. **LMX** will provide a real opportunity for biological crystallography on a pulsed neutron source.

The high flux of cold neutrons on the second target station will extend the range of diffraction measurements out to longer d-spacings. **WISH** is optimised for rapid data collection over a broad d-spacing range of up to 20 Å with a good resolution of 1×10^{-3} $\Delta d/d$.

Many modern materials exhibit short-range structure with longer-range inhomogeneities. Understanding such inhomogeneities is often crucial to establish why particular materials behave the way they do, and it is often important to determine both types of structure in a single experiment. **NIMROD**, the near and intermediate range order diffractometer, is designed to measure both short range

structure with excellent spatial resolution (<0.1 Å) and intermediate range order (concentration fluctuations, density fluctuations) out to 100 Å in crystalline solids and amorphous solids, liquid mixtures, large molecule solutions, intercalation compounds, critical fluids, and engineering and polymeric materials. The aim will be to measure both short-range and intermediate-range structure so that an accurate large-scale model of the local structure can be established.

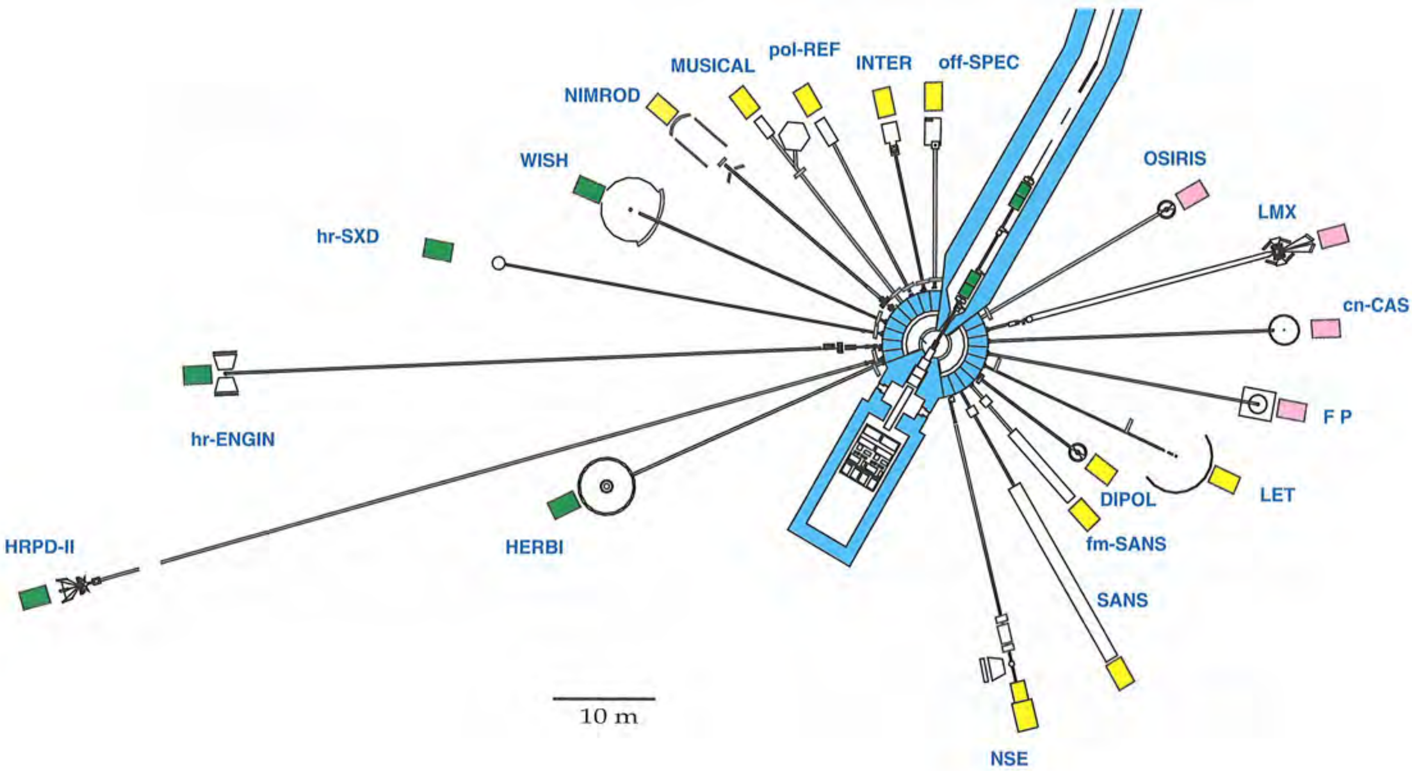
DIPOL is optimised for the measurement of diffuse scattering, in particular examining the interplay between atomic and magnetic short-range order. The emphasis is on providing high counting rates and low background in order to measure weak magnetic scatterers and small single crystals. Polarisation analysis is performed using spin-polarised ^3He and will be invaluable for studying magnetic systems.

Spin polarised neutron beams are an essential feature of instrumentation for magnetism studies. The provision of efficient 'white beam' polarisers and analysers, and spin turners is a key development issue.

Spectroscopy

Spectrometers on the Second Target Station will offer high resolution (<μeV) and a wide dynamic range of energy transfer (from neV to tens of meV). The proposed suite contains one direct geometry spectrometer, **LET**, which will allow a continuous choice of incident energy from 1meV up to 50meV with a resolution of less than 10μeV at an incident energy of 1meV. This high degree of flexibility combined with a wide detector bank will provide simultaneous access to a broad region of (Q,ω) space. The indirect geometry spectrometers, **OSIRIS**, **HERBI**, **cn-CAS**, and **MUSICAL** will offer a broad dynamic range. **HERBI** and **MUSICAL** are both optimised for sub-meV resolution. **OSIRIS** will offer good resolution accompanied by high flux and the option of full polarisation analysis. **cn-CAS** is a development of PRISMA which will offer a dramatic improvement in count rate, but also a huge increase in flexibility by using position sensitive detectors, and a more modular approach to the secondary spectrometer.

Neutron spin echo offers ultra high (neV) resolution for quasielastic and inelastic scattering measurements. The existing NSE instruments are all on steady state sources; however, pulsed sources and the time of flight technique offer inherent advantages, including potentially greater Q resolution and a larger dynamic range of Fourier time in a single measurement.

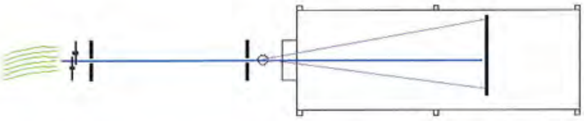


INSTRUMENTS

SANS

Small Angle Scattering Instrument

A world class SANS instrument with exceptionally broad simultaneous Q range.

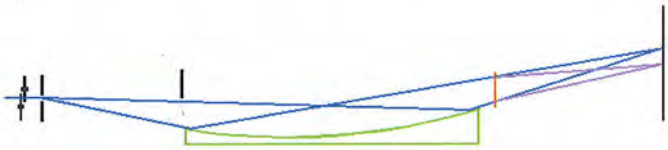


www.isis.rl.ac.uk/targetstation2/instruments/sans.htm

fm-SANS

Focusing Mirror Small Angle Scattering Instrument

A SANS equipped with a focusing mirror providing access to very low Q and hence extending the technique to longer length scales.

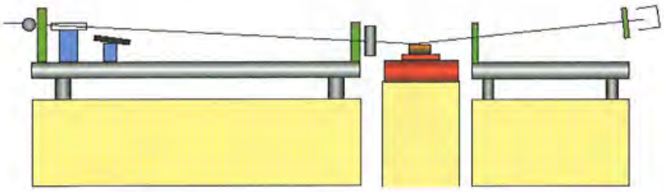


www.isis.rl.ac.uk/targetstation2/instruments/fmsans.htm

INTER

Chemical Interfaces Reflectometer

INTER is a reflectometer offering huge flexibility in the choice of resolution and flux. High flux and a wide wavelength band allow for short data acquisition times. This reflectometer will be ideal for time dependent studies on the time scale of seconds.

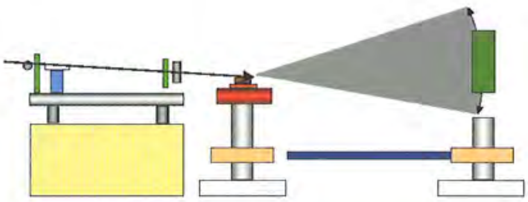


www.isis.rl.ac.uk/targetstation2/instruments/inter.htm

off-SPEC

Off-specular Reflectometer

off-SPEC is a reflectometer offering huge flexibility in the choice of resolution, and wave-vector range. Combined horizontal and vertical scattering geometries allow a wide range of systems to be studied.

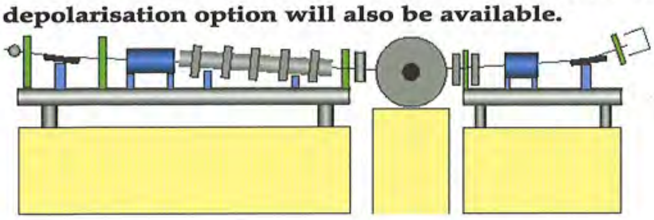


www.isis.rl.ac.uk/targetstation2/instruments/offspec.htm

pol-REF

Polarised Neutron Reflectometer

pol-REF is a direct geometry polarised neutron reflectometer. High flux and a wide wavelength band allow for short data acquisition times. A neutron depolarisation option will also be available.

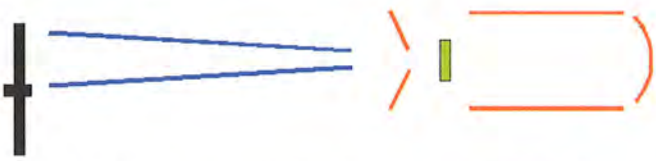


www.isis.rl.ac.uk/targetstation2/instruments/polref.htm

NIMROD

Near and InterMediate Range Order Diffractometer

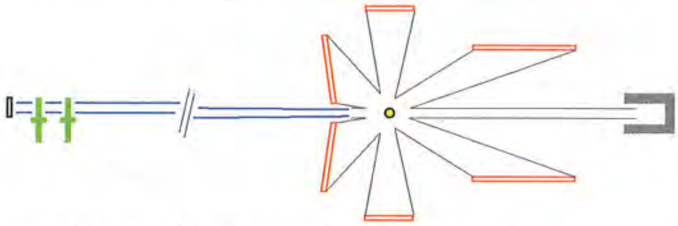
Combined low angle and wide-angle diffractometer for measuring local and intermediate range order in a wide range of crystalline and disordered materials.



www.isis.rl.ac.uk/targetstation2/instruments/nimrod.htm

HRPD-II
Ultra High Resolution Powder Diffractometer

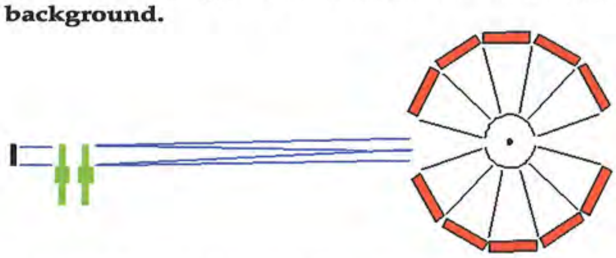
HRPD-II is designed to be the world's highest resolution powder neutron diffractometer with a constant Δd/d~0.03% over a wide range of d-spacing and offering unique possibilities for structural studies of complex materials.



www.isis.rl.ac.uk/targetstation2/instruments/hrpd.htm

hr-SXD
High Resolution Single Crystal Diffractometer

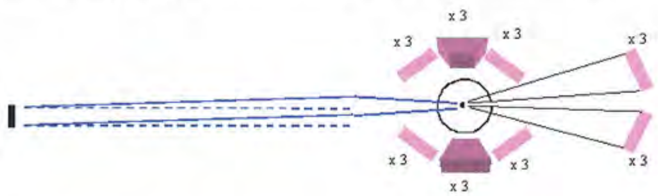
The large position sensitive detectors, principally situated in the equatorial plane, are designed for full reciprocal plane coverage whilst maintaining high spatial and temporal resolution and extremely low background.



www.isis.rl.ac.uk/targetstation2/instruments/hrsxd.htm

LMX
Large Molecule Crystallography Diffractometer

LMX is optimised for high resolution studies of large unit cell structures, for example in protein crystallography, also offering the capability for studying larger assemblies at lower resolution.



www.isis.rl.ac.uk/targetstation2/instruments/lmx.htm

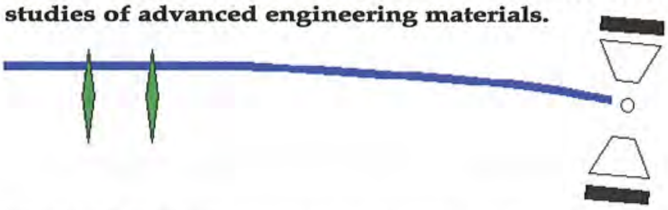
WISH
Wide Angle In a Single Histogram

WISH is a powder diffractometer with a large solid-angle detector that can be focused to a single histogram, covering a wide range of reciprocal space.

www.isis.rl.ac.uk/targetstation2/instruments/wish.htm

hr-ENGIN
High Resolution Engineering Instrument

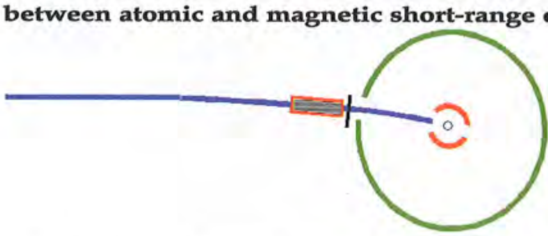
hr-Engin is an engineering instrument designed to make maximum use of the low repetition rate of the Second Target Station, for high resolution diffraction studies of advanced engineering materials.



www.isis.rl.ac.uk/targetstation2/instruments/hrengin.htm

DIPOL
Diffuse Scattering with Polarisation Analysis

DIPOL is optimised for measurements of diffuse scattering, in particular examining the interplay between atomic and magnetic short-range order.



www.isis.rl.ac.uk/targetstation2/instruments/dipol.htm

OSIRIS

Backscattering Spectrometer with Polarisation Analysis

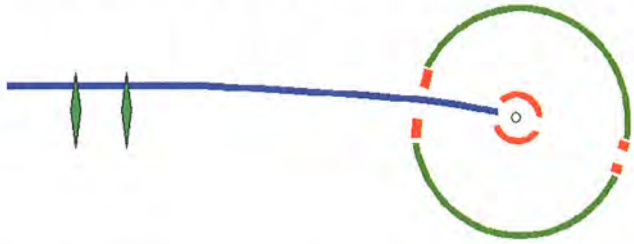
OSIRIS is an indirect geometry crystal analyser spectrometer, which provides excellent energy resolution and high counting rate with full polarisation analysis.

www.isis.rl.ac.uk/targetstation2/instruments/osiris.htm

HERBI

High Energy Resolution Backscattering Instrument

HERBI is an indirect geometry crystal analyser spectrometer, which provides very high-energy resolution over a large dynamic range.

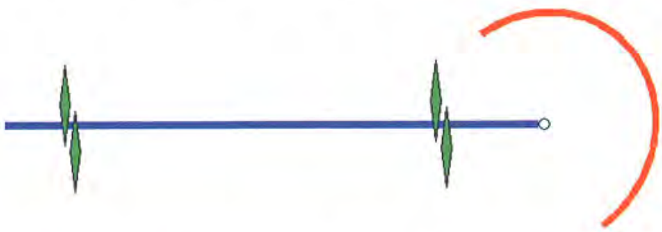


www.isis.rl.ac.uk/targetstation2/instruments/herbi.htm

LET

Low Energy Transfer Chopper Spectrometer

LET is a direct geometry spectrometer offering huge flexibility in the choice of resolution and flux with the capability of using quasi time focusing to optimise conditions for individual experiments.



www.isis.rl.ac.uk/targetstation2/instruments/let.htm

cn-CAS

Cold Neutron Single Crystal Crystal-Analyser Spectrometer

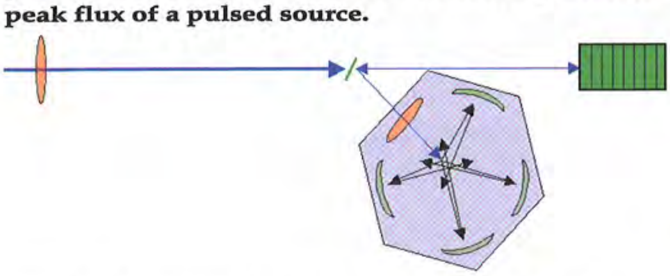
An indirect geometry crystal-analyser spectrometer optimised to exploit the potential for high resolution cold neutron single crystal spectroscopy with the intrinsic advantages of the time-of-flight technique.

www.isis.rl.ac.uk/targetstation2/instruments/cncas.htm

MUSICAL

Multi Silicon Monochromator Crystal

MUSICAL is a backscattering instrument that provides high resolution whilst benefiting from the peak flux of a pulsed source.



www.isis.rl.ac.uk/targetstation2/instruments/misical.htm

NSE

Neutron Spin Echo

A neutron spin echo spectrometer offering neV resolution and a large dynamic range.

www.isis.rl.ac.uk/targetstation2/instruments/nse.htm

Applications of the Proposed Instrument Suite

Large Scale Structures

	Soft Condensed Matter	*Bio-molecular Sciences*	*Advanced Materials*
SANS	• Micelle, microemulsion, surfactant and polymer systems • Complex, mixed surfactant and polymer systems close to industrial applications • In-situ studies of polymers and complex fluids in processing environments • "triggered" changes in surfactant and polymer systems • Mesophases	• Macromolecular systems close to "in-vivo" conditions • Membranes and their interactions with other molecules • Vesicles, gels and drug delivery systems • Food science	• Multi-phase, block and novel architecture copolymers • Micro-porous systems • Flux lattice and magnetic domains in multilayers and superconductors • Precipitation and phase separation in metal alloys • Low concentration defects in semiconductors
fm-SANS	• Mesophase texture and self assembly • Polymer phase separation • Emulsions	• Large biological macromolecules • Food colloids • Liposomes, drug and pesticide delivery systems	• Phase separation and precipitation studies of polymers and metal alloys • Nucleation and growth • Studies of 'fractal' materials over wide length scales: porous materials, rocks etc. • Texture studies
INTER	• Chemical interfaces • Diffusion and reaction kinetics • Solid/liquid surface modification • Surfactants at the air/liquid, liquid/liquid and solid/liquid interfaces • Inorganic templating at the air/water interface • In-situ electrochemistry	• Protein adsorption • Biological membranes • Nature of membrane/protein interactions • Interfacial structure in drug delivery systems	• Polymers and liquid crystals • Engineering alloys • Biocompatibility • Sensors
off-SPEC	• Phase separation in polymer films • Dewetting/wetting phenomena • Colloidal systems, surfactant polymer systems • Critical phenomena in fluid systems	• Lateral correlations in biological systems	• Magnetic domain correlations • Magnetic roughness • Magnetic recording media

	Soft Condensed Matter	Bio-molecular Sciences	Advanced Materials
pol-REF			• Magnetic superstructures magnetisation depth profiling • Absolute vector magnetometry • Magnetic disorder • Superconducting systems • Magnetisation kinetics • Magnetic nano-structures • Neutron depolarisation • Magnetic recording media • Spin valves • GMR materials
Diffraction			
HRPD-II		• Pharmaceutical systems - ab-initio structural determination and refinement of drug molecules.	• Mineralogical specimens. • Multiphase composites. • Microporous compounds (zeolites). • inhomogeneous systems. • Thermal expansion, compressibility, strain. • Phase transitions and critical scattering. • Non-stoichiometric solids • Structural changes in-situ under variable temperatures, pressures and applied fields.
hr-SXD			• Defect structures • Phase transitions. • Incommensurate structures, domain structures, quasicrystals. • Texture measurements within engineering components.
LMX		• Protein crystallography. • Pharmaceuticals. • Biological molecules	• Large molecule chemical crystallography, supramolecular chemistry, organometallic materials (including catalysts). • Organic structures – studies of charge density and hydrogen bonding.

WISH	• Coexistence of amorphous and crystalline structures	• Small organic molecule crystallography	• Large unit-cell structure determination • Magnetic structures – magneto-structural problems • Zeolites – host-guest problems • In-situ chemistry • High pressure • Batteries, • Catalytic materials • Clathrates • Molecular sieves
hr-ENGIN			• In-situ investigations - process optimisation • Microstress measurements - deconvolution of peak width • Phase transformations sensitive to stress • Intergranular stress measurements, multiphase & polycrystal • Stress measurements in cracks, coatings, buried interfaces, multilayers
NIMROD	• Intercalation compounds to study liquids at surfaces • Polymeric materials: short range and longer range order	• Liquid mixtures and large molecule solutions. • Protein folding. • Hydration.	• Crystalline solids: total scattering (pair distribution function analysis) of crystals to determine extent and nature of diffuse scattering and longer range lattice imperfections • Amorphous solids: determine extent of longer range structural inhomogeneities • Critical fluids • Engineering applications: superalloy coarsening, displacive transformations in ceramics, sintering of ceramics, hydration of cements, nanophase catalysts, zeolite crystallisation

	Soft Condensed Matter	*Bio-molecular Sciences*	*Advanced Materials*
DIPOL			• Elimination of incoherent scattering from S(Q) in amorphous materials • Absolute intensity scale and determination of hydrogen content • Interplay between atomic and magnetic short range order • Magnetically disordered systems
Spectroscopy			
OSIRIS	• Polymer dynamics		• High temperature superconductors • Colossal magnetoresistance materials
HERBI	• Diffusional processes • QENS • Quantum tunneling	• Vibrations in model systems e.g. peptide bonds • Dynamics of DNA – base pair breaking • Protein folding	• Quantum fluids • Hydrogen diffusion in fuel cells, batteries • Fluid diffusion in zeolites and porous media
LET	• Polymer dynamics • Chemical spectroscopy	• Protein folding • Water dynamics • Solute/solvent interactions	• High T_c superconductivity • Colossal magenetoresistance • Crystal field measurements in optically active materials • Non-Fermi liquid systems • Quantum fluids • Parametric density-of-states as a function of temperature, composition etc.
cn-CAS	• Tunnelling excitations in disordered and amorphous systems	• Dynamics of pharmaceutical molecules	• Low energy spin waves and phonons in small/complex samples • High pressure studies of single crystals
MUSICAL	• Diffusional processes • Molecular dynamics • Quasielastic scattering	• Low energy dynamics of biological molecules	• Super-paramagnets • Magnetic clusters
NSE	• Polymer dynamics • Macromolecular dynamics (colloids, emulsions, mesophases)	• Very low energy dynamics of biological molecules	• Spin glasses • Clusters

User Support Facilities

The gains in performance offered by the Second Target Station will open up new fields of experimental science. From the scientific case presented here it is clear that the demand for more complex and challenging sample environment will increase substantially. Examples include combined extreme conditions like high pressure and high temperature or very low temperature studies in strong magnetic fields, *in-situ* and time dependent measurements, and studies of flow and processing under realistic conditions. Higher throughput and parametric studies will require a greater degree of automation.

The focus of the ISIS User Support Group with respect to the Second Target Station is to continue the high levels of support provided at present. At the same time we will expand the current suite of sample environment equipment to enable the user community to exploit fully the potential of the new facility.

The tremendous amount of experience gained by ISIS staff with operating such a complex and demanding user programme will feed into the Second Target Station layout and will allow us to build in greater efficiency from day one. For example, installing liquid nitrogen supply systems and recycling helium gas will provide a much more effective management of cryogenic liquids. The greater emphasis on both Soft Condensed Matter and Bio-molecular Science will require an expansion of dedicated support facilities in these areas. Smooth and level access around the hall and support buildings will ensure an efficient delivery of equipment. The relative location of support laboratories with respect to the instruments is also essential.

The suite of new instruments identified through the science case has highlighted the need for a vigorous instrumentation R&D programme. In particular demands for a new generation of choppers, such as counter rotating disk choppers and for sophisticated neutron beam optics will require the provision of dedicated laboratory space, funds and manpower. A clean assembly area, for example for the neutron guides, detector banks, analyser arrays and similar delicate equipment, will be needed.

Doubling the capacity for user access to ISIS is an opportunity to review the non-scientific support provided for the user community. In particular with the decision to locate the New Synchrotron at the site of the Rutherford Appleton Laboratory there is now an opportunity to develop some first class user services, including a modern accommodation block on site and restaurant facilities which offer a more flexible and varied service to the visitors. A joint neutron and synchrotron User Office and Reception Centre should be envisaged as a single point of contact for both facilities.

Future Opportunities

New Instruments for the High Power Target Station

In addition to opportunities on the Second Target Station for the creation of entirely new instruments, instruments whose performance is currently compromised on the existing High Power Target will be moved to the Second Target Station. This will provide the space for new instruments to be developed on the high power target, building on the range of neutron instrumentation that has been created over the past decade (see the instrument development plan on page 10). Some possible examples are given below.

The high flux offered by the 50 Hz target is well suited to parametric or time dependent studies in either diffraction or spectroscopy. One difractometer proposed for the High Power Target Station is **IN-SITU**, which will be designed for the study of chemical reactions as they proceed. Scientific applications would include time-resolved studies of chemical/electrochemical processes performed in-situ on the diffractometer and studies of the kinematics of phase formation. This clearly has relevance to the understanding of, for example, catalysis processes and the development of new materials for applications such as batteries.

Some spectroscopic studies are also well suited to an instrument that offers very high intensity. The high intensity spectrometer, **HIS**, would offer modest resolution, but high count rates by virtue of a large detector solid angle and a focusing mirror monochromator. Detailed parametric studies would be possible, by either rapidly measuring a range of samples of varying composition or measuring the dynamics as a function of parameters such as pressure, temperature or magnetic field. Applications span from studying crystal field excitations in optically active glasses to molecular spectroscopy.

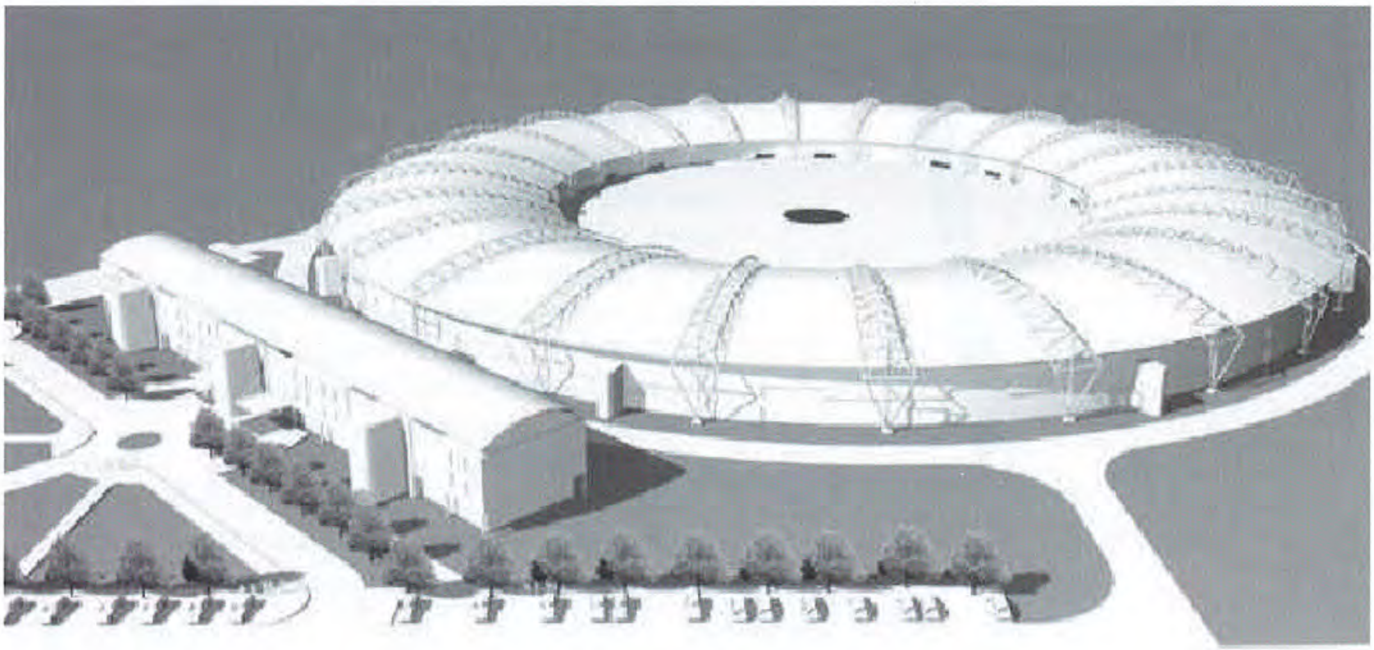
Neutron Brillouin scattering is a powerful tool to monitor the long-range dynamical processes in a disordered material. **BRIL** would be a direct geometry spectrometer specifically designed for neutron Brillouin scattering offering access to very low Q and a wide incident energy range. In addition to providing valuable information for the study of disordered materials, **BRIL** would also be able to probe coherent phenomena in many large-scale systems such as polymers and biological molecules.

An engineering instrument optimised for high flux, **HIED**, will be ideally suited for rapid monitoring of phase changes and texture measurements. Access to a high flux of short wavelength neutrons will also allow deeper penetration for stress measurement.

Applying the technological developments used on the GEM detectors to an upgraded SANDALS would provide significantly enhanced performance. The demand on MARI for inelastic studies of non-crystalline materials cannot currently be satisfied, and additional capacity in this area is much needed.

ISIS and the New Synchrotron Source

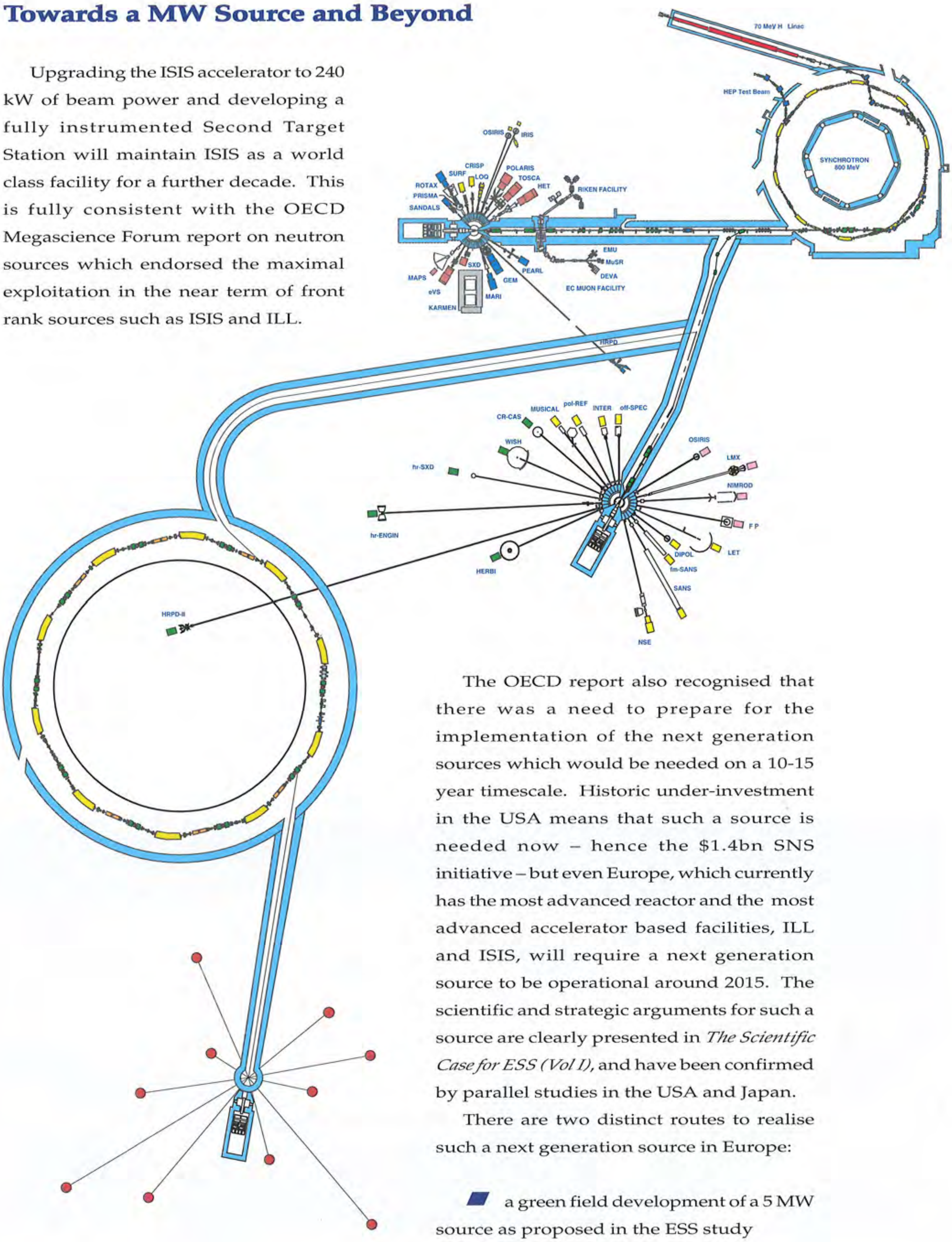
Synchrotron radiation and neutron scattering have together provided much of what we know at the microscopic level about the nature of the material world around us. Synchrotrons have enabled an unparalleled breadth of research from photoelectron spectroscopy to protein structure; neutron scattering has provided answers of unrivalled precision in research topics such as high temperature superconductivity and magnetism. Synchrotron radiation has been the diffraction tool of choice for large, complex systems such as protein structures, while neutron diffraction is the preferred tool for simpler systems such as superconductor oxides. There is, however, considerable overlap in the use of both techniques in Materials Science, Physics, Chemistry and Engineering. In high pressure studies, for example, the decision whether to use neutrons or synchrotron radiation is determined largely by the nature of the sample. Furthermore, over the past decade, particularly as a result of research at ISIS and the Institut Laue Langevin (ILL), Grenoble, significant advances have been made in the study of more complex systems such as polymers, surfactants and catalysts by neutron scattering. Indeed, ISIS and the ILL lead the world in the breadth of neutron scattering from Materials Physics to the study of Soft Condensed Matter. The key to this diversity of research is a wide range of neutron energies. The next step in the world of neutron scattering is the construction of the Second Target Station at ISIS that facilitates optimal epithermal and thermal neutron spectra on one target station and cold neutrons on a second. This will give an unparalleled breadth to new possibilities with neutron scattering that will complement the science performed at synchrotron sources. The fact that this key next step in neutron scattering is planned to take place on the same campus as the new UK synchrotron provides the UK scientific community with a once-in-a-generation opportunity to command a leading role in many aspects of condensed matter science in Physics, Chemistry, Earth Sciences, Engineering, Pharmaceutical Sciences and Structural Biology. With the emphasis in the science programme for the Second Target Station on complex systems, soft condensed matter, advanced materials and the biomolecular sciences, ISIS and the New Synchrotron will provide unique opportunities for complementary and combined research programmes that optimise the use of both facilities.



OPPORTUNITIES

Towards a MW Source and Beyond

Upgrading the ISIS accelerator to 240 kW of beam power and developing a fully instrumented Second Target Station will maintain ISIS as a world class facility for a further decade. This is fully consistent with the OECD Megascience Forum report on neutron sources which endorsed the maximal exploitation in the near term of front rank sources such as ISIS and ILL.



The OECD report also recognised that there was a need to prepare for the implementation of the next generation sources which would be needed on a 10-15 year timescale. Historic under-investment in the USA means that such a source is needed now – hence the $1.4bn SNS initiative – but even Europe, which currently has the most advanced reactor and the most advanced accelerator based facilities, ILL and ISIS, will require a next generation source to be operational around 2015. The scientific and strategic arguments for such a source are clearly presented in *The Scientific Case for ESS (Vol I)*, and have been confirmed by parallel studies in the USA and Japan.

There are two distinct routes to realise such a next generation source in Europe:

- a green field development of a 5 MW source as proposed in the ESS study

- an evolutionary development of the science complex at ISIS.

The first strategy is fully documented in the ESS case. The UK continues to play a major part in the activities of the ESS R&D Council in collaboration with our European partners, and RAL remains a potential site for such a facility.

There is, however, much to recommend an evolutionary approach, building on the substantial investment, both physical and intellectual, which already exists at ISIS.

This strategy also provides strong synergy with UK needs in Particle Physics (muon derived neutrino beams, muon collider R&D), potential interest in transmutation, isotope production and possible energy amplification, and radioactive nuclear beam developments.

The proposed upgrade path for ISIS exploits the fact that neutron production is essentially proportional to proton beam power, and that beam power is the product of beam current and beam energy.

The present 800 MeV ISIS synchrotron is limited in intensity to 200 µA (160 kW) due to the space charge limit at injection. In the short term, the intensity will be increased by the use of dual harmonic acceleration (4 additional rf cavities) which enables a larger area of RF phase space to be captured at the start of acceleration. The radio frequency quadrupole necessary to provide a higher pulsed current in the 70 MeV linac is already under test at ISIS. Together with the dual harmonic RF system (installation in 2002) this will allow 300 µA to be accelerated to 800 MeV in the present synchrotron giving 240 kW of beam power.

ISIS can be further upgraded by the addition of two rapid cycling synchrotrons with an energy of 3.3 GeV. These will have a repetition rate of 25 Hz but run exactly out of phase with each other. Built in the same tunnel, each 45 m radius synchrotron will receive beam from alternate pulses from the 800 MeV ISIS synchrotron. The new synchrotrons will use fast injection (1 turn of two 800 MeV beam bunches) and the beam accelerated to 3.3 GeV within the 40 ms period. The design of the rings can be made similar to those proposed for a 5 MW option for the ESS. Working in tandem, the two synchrotrons will deliver a beam of 1 MW at 50 Hz.

A further upgrade of ISIS to 5 MW is possible by replacing the fast injection from the 800 MeV synchrotron into the 3.3 GeV synchrotrons by multi-turn H^- injection from an 800 MeV linac. The beam intensity required in the linac is 1.5 mA. This may well prove to be a cost effective route to realise 5 MW power levels for European neutron scattering in the second decade of this century.

SIRIUS

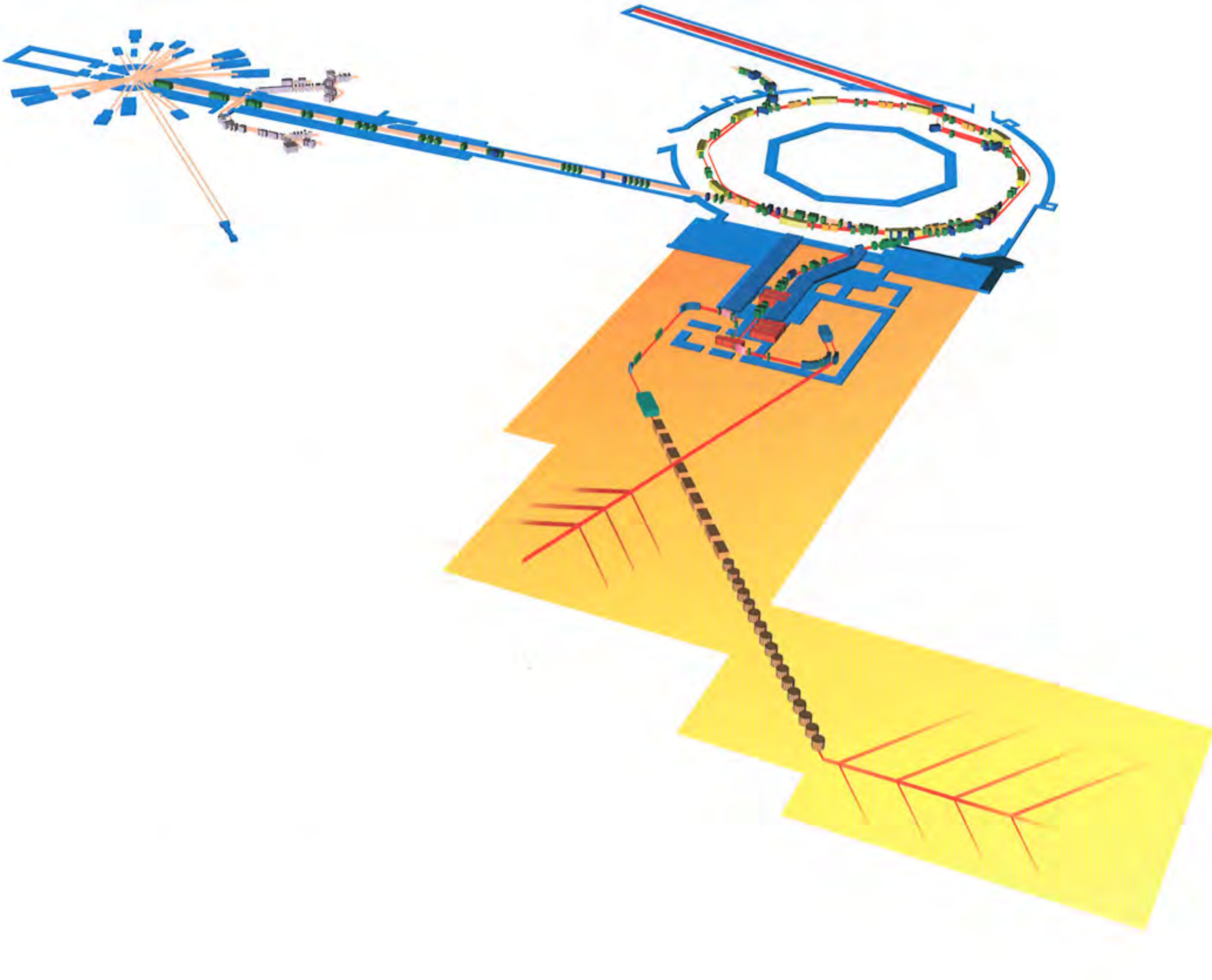
SIRIUS is a proposed next generation accelerator facility utilising a fraction of the ISIS proton beam. It is designed to produce and accelerate beams of radioactive nuclei for fundamental studies of nuclear physics and nuclear astrophysics processes, and for applications in materials, bio-medical and environmental science.

SIRIUS has been designed to use up to 100 μA of 800 MeV protons from the ISIS synchrotron. It will provide multiple beams of separated isotopes for parallel use at energies up to 200 keV, plus a high intensity beam of nuclei with masses up to 200, at energies upto 10 MeV/nucleon.

This is to be compared with the existing radioactive beam facility ISOLDE at CERN which uses 2 μA at 1 GeV and the new facility, ISAC, at TRIUMF which is under construction and has been designed for a proton beam current of up to 100 μA at 450 MeV.

The SIRIUS design is totally compatible with the operation of the Second Target Station for neutron scattering. It allows the proton beam to be shared on a pulse by pulse basis between the three facilities giving the required flexibility for operations.

SIRIUS is based on proven technology and could be fully implemented within four years of project approval.



User Consultation Meeting 19 May 2000

A User Consultation Meeting was held on Friday 19th May 2000 at RAL to discuss and refine the scientific case for a Second Target Station at ISIS. The meeting was attended by over 100 potential users, and by ISIS staff. The meeting expressed strong support for the draft proposal. The views of the user community represented at the meeting have been incorporated into this document.

Programme

Welcome: *R Thomas (Oxford)*

Introduction: *A Taylor (ISIS)*
Technical Details: *J Penfold (ISIS)*

Science for the Millennium

Soft Condensed Matter: *T McLeish (Leeds)*
Bio-molecular Sciences: *C Dobson (Oxford)*
Nano-Scale Materials: *S Mann (Bristol)*
Advanced Materials: *J White (ANU Canberra)*

Discussion Sessions

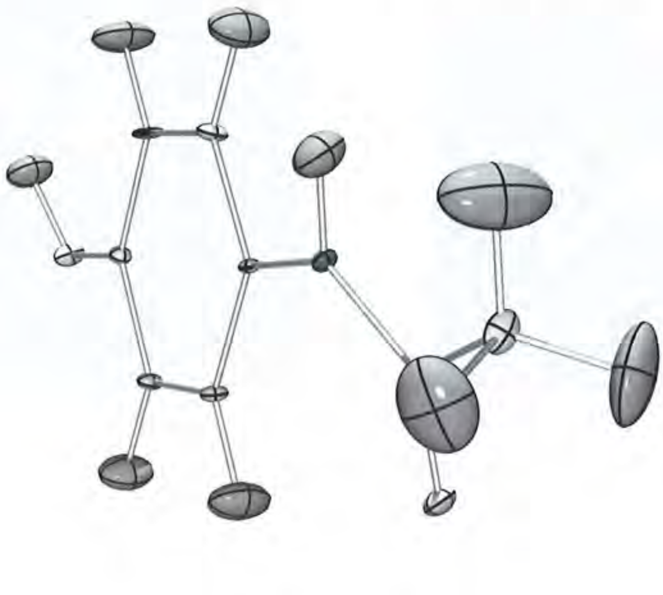
Soft Condensed Matter: *R Richards (Durham)*
Bio-molecular Sciences: *A Watts (Oxford)*
Advanced Materials
Solid State Chemistry: *A Harrison (Edinburgh)*
Magnetic Materials: *J Bland (Cambridge)*
Disordered Materials: *J Finney (UCL)*

Feedback from Discussion Sessions, and Summary

Right: Participants at the User Consultation Meeting listen to presentations of Science for the Millennium. Left: John White (ANU), Tom McLeish (Leeds) and Bob Thomas (Oxford) discuss future science prospects.

User Consultation Meeting Participants

Dr M Adams	Paul Scherrer Institut
Professor C Andreani	University of Rome
Dr V Arrighi	Heriot-Watt University
Dr J P Attfield	University of Cambridge
Professor J Baldwin	Liverpool John Moores University
Dr D Barlow	Kings College London
Dr J A C Bland	University of Cambridge
Dr A T Boothroyd	University of Oxford
Dr J Bowers	Queen Mary & Westfield College
Dr J P Bradshaw	University of Edinburgh
Mr P Buchanan	H H Wills Physics Laboratory
Dr D Bucknall	University of Oxford
Dr C J Carlile	Institut Laue-Langevin
Dr J Carpenter	Argonne National Laboratory
Dr N Clarke	UMIST
Dr S Clarke	University of Exeter
Dr S Clarke	University of Cambridge
Dr J Cole	University of Cambridge
Professor T Cosgrove	University of Bristol
Professor R Cywinski	University of St. Andrews
Professor P Day	Royal Institution
Professor C M Dobson	University of Oxford
Dr M T Dove	University of Cambridge
Dr K Elder	University of Bath
Dr M Fitzpatrick	Open University
Professor J L Finney	University College London
Dr R C Ford	UMIST
Dr C S Frampton	Roche Research Centre Welwyn
Dr B D Fulthorpe	University of Durham
Dr B Gabrys	Open University
Dr A M Gaspar	UMIST
Dr A Glidle	Glasgow University
Dr J P Goff	University of Liverpool
Mr J Gold	OSTD
Mr J Green	EPSRC
Dr P C Griffiths	Cardiff University
Dr Telling	University of Salford
Dr T Gutberlet	Hahn-Meitner-Institut
Dr M Guthrie	University of Edinburgh
Dr P J Hall	University of Strathclyde
Dr P Haris	De Montford University
Professor A Harrison	University of Edinburgh
Dr T A Harroun	University of Edinburgh
Dr T P A Hase	University of Durham
Professor J Helliwell	University of Machester
Professor C M B Henderson	University of Manchester
Dr S J Hibble	University of Reading
Professor A R Hillman	University of Leicester
Dr D Holland	University of Warwick
Professor J A K Howard	University of Durham
Dr M T Hutchings	Open University
Dr U A Jayasoorija	University of East Anglia
Professor R A L Jones	University of Sheffield
Dr K Jungmann	University of Heidelberg
Professor G J Kearley	University of Delft
Dr S Kilcoyne	University of St Andrews
Dr C A Kirk	University of Sheffield
Dr A Kolesnikov	UMIST
Dr J Lawrence	King's College London
Dr A Lennie	University of Manchester
Dr J C Li	UMIST
Dr S Lister	University of Oxford
Dr J R Lu	University of Surrey
Professor S Mann	University of Bristol
Professor K A McEwen	University College London
Professor T C B McLeish	University of Leeds
Dr I McLure	University of Sheffield
Dr I Michalazias	UMIST
Mr C C Miles	BBSRC
Dr P C H Mitchell	University of Reading
Professor R J Nelmes	University of Edinburgh
Professor S J Perkins	University College London
Professor S E V Phillips	University of Leeds
Dr A Powell	Heriot-Watt University
Professor G Zu Putlitz	University of Heidelberg
Dr A J Ramirez-Cuesta	University of Reading
Dr S Redfern	University of Cambridge
Dr A R Rennie	King's College London
Professor M A Ricci	University of Rome
Professor R W Richards	University of Durham
Dr J W Richardson	Argonne National Laboratory
Professor R M Richardson	University of Bristol
Dr S Roser	University of Bath
Professor K Ross	University of Salford
Dr P S Salmon	University of Bath
Dr J Santisteban	Open University
Professor H Schenk	University of Amsterdam
Dr N Skipper	University College London
Professor G L Squires	University of Cambridge
Dr R J Stewart	University of Reading
Dr G M Swallowe	Loughborough University
Dr A Tennant	University of Oxford
Dr R K Thomas	University of Oxford
Mr C Thompson	BBSRC
Dr H Tietze-Jaensch	Forschungszentrum Juelich
Dr D Timms	Portsmouth University
Professor A H M Verkooijen	University of Delft
Dr D Visser	NWO
Dr C Washington	University of Nottingham
Professor A Watts	University of Oxford
Professor J W White	Australian National University



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