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Neutron Scattering Science at the Australian Nuclear Science and Technology Organisation (ANSTO)

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ABSTRACT

Neutron scattering science at ANSTO is integrated into a number of fields in the Australian scientific and industrial research communities. The unique properties of the neutron are being used to investigate problems in chemistry, materials science, physics, engineering and biology. The reactor HIFAR at the Australian Nuclear Science and Technology Organisation research laboratories is the only neutron source in Australia suitable for neutron scattering science. A suite of instruments provides a range of opportunities for the neutron scattering community that extends throughout universities, government and industrial research laboratories.

Plans to replace the present research reactor with a modern multi-purpose research reactor are well advanced. The experimental and analysis equipment associated with a modern research reactor will permit the establishment of a national centre for world class neutron science research focussed on the structure and functioning of materials, industrial irradiations and analyses in support of Australian manufacturing, minerals, petrochemical, pharmaceuticals and information science industries.

A brief overview will be presented of all the instruments presently available at ANSTO with emphasis on the SANS instrument. This will be followed by a description of the replacement research reactor and its instruments.

THE NEUTRON SOURCE HIFAR

The High Flux Australian Reactor (HIFAR) is a DIDO-class 10MW(th) research reactor of modest performance (maximum thermal neutron flux $1\times10^{14}cm^{-2}sec^{-1}$) and provides the only neutron source in the country suitable for neutron scattering. HIFAR is

operated by the Australian Nuclear Science and Technology Organisation (ANSTO) (http://www.ansto.gov.au), a government research organisation with the mission to 'ensure that its research, technology transfer, commercial and training activities in nuclear science and associated technologies will advance Australia's innovation, international competitiveness and environmental and health management'.

HIFAR was designed and built in the 1950's primarily as a materials testing reactor and as such has limited facilities for neutron beam studies. Apart from the neutron beam quality discussed below, the reactor building is small (20 metre diameter) and does not provide the space required for modern neutron scattering instruments. In addition, there is no cold source installed. Figure 1 is a photograph of the interior of the HIFAR reactor building. The limitations of HIFAR as a neutron source have been recognised and in September 1997 the Australian government made the decision to replace HIFAR with a multi-purpose pool-type research reactor.

NEUTRON SCATTERING INSTRUMENTS ON HIFAR

The present supported instruments located on the HIFAR reactor include two single crystal diffractometers (2tanA, 2tanB), a polarised beam facility (LONGPOL), two powder diffractometers (HRPD, MRPD) and a small angle neutron scattering (SANS) instrument. A reflectometer is under construction. Except for 2tanA and 2tanB, instruments are located on radial beam tubes. The beam tubes have a diameter up to 25cm, and terminate either in the D_2O moderator or the graphite reflector regions of the reactor core. This has an impact on beam quality and has influenced instrument design. Table 1 is a summary of instrument characteristics and Figure 2 illustrates their disposition on the experimental floor.

The Single Crystal Diffractometers

The two single crystal diffractometers share one end of a 'tangential' beamline that passes under the reactor core. The High Resolution Single Crystal Diffractometer (2tanA) has a standard goniometer and a small 2D position sensitive detector (PSD) with single reflection capability has been installed. A copper crystal at a take off angle of 58° produces the 1.235Å neutron beam. The 10mm diameter neutron beam has a flux of ~5 × $10^5 cm^{-2} sec^{-1}$ at the sample position with good signal to background characteristics. The maximum 2θ is ~118° and over this range the reflection widths are typically ~0.4° (fwhm) (2θ) at 5° (2θ) increasing to ~0.75° at 105°. The major purpose of the instrument is high-resolution structure determination.

The Medium Resolution Single Crystal Diffractometer (2tanB) is a four circle instrument with a single BF_3 detector. A pyrolytic graphite monochromator provides a 10mm diameter 1.239Å neutron beam at a take off angle of 21°, with a flux of ~$10^6 cm^{-2} sec^{-1}$ at the sample position. The reflection widths are typically ~0.4° (fwhm) (2θ) at 5° (2θ) increasing to about 2° at 90° with a maximum 2θ of ~120°. The major purpose of the instrument is structure determination particularly where diffraction intensity is limited and/or where a small 2θ range provides the required information eg magnetic and larger molecule structures. The instrument can also be used to investigate texture and diffuse scattering from disorder in metals, alloys, ceramics, polymers etc.

The Long Wavelength Polarised Neutron Spectrometer

The Long Wavelength Polarised Neutron Spectrometer (LONGPOL) (Figure 3), the most novel instrument on HIFAR, is a diffractometer/spectrometer for diffuse and inelastic scattering measurements incorporating both neutron polarisation analysis and energy analysis[1]. The instrument operates at a wavelength of 3.6Å and is equipped with 8 ^{3}He detectors. Installation of high efficiency polarising supermirrors developed in collaboration with the Hahn-Meitner Institute is now complete. The incident polarisation of the neutron beam is now 96% - improved from 40% by the installation of a supermirror bender, made to ANSTO's design at the Hahn-Meitner Institute (Berlin), and supported by Australian Research Council Research Infrastructure Funds. The supermirror benders on single crystal silicon substrates are the latest in neutron polarisation technology. The next stage in this development is to install the polarising supermirror benders that are required to analyse the neutron spin orientation after scattering from a sample. Possible configuration options for the analyser benders have recently been investigated and the favoured prototype mount has been manufactured. This stage will deliver a further factor of >4 in performance. These improved characteristics increase the quality and quantity of data available from this instrument, which has special applications in the areas of magnetic order, flux dynamics in high temperature superconductors and crystal field studies. Polarisation analysis allows separation of magnetic scattering events from nuclear scattering events, and also allows isolation of nuclear-spin-incoherent scattering, which can be used to determine hydrogen concentration, for example. The neutron flux at the sample position is ~3 × 10^4 $cm^{-2}sec^{-1}$ in a low resolution configuration that makes the instrument ideally suited to neutron diffuse scattering studies. Main applications include determination of atomic and magnetic distributions in magnetic materials, measurement of hydrogen concentration in bulk materials, measurements of paramagnetic scattering, magnon measurements, neutron depolarisation studies, and flux creep in superconductors[2].

The Powder Diffractometers

The two powder diffractometers (Figure 4) were designed specifically to accommodate the conflicting requirements of neutron intensity and resolution[3]. The neutron flux at the sample position on the Medium Resolution Powder Diffractometer (MRPD) is typically five times that of the High Resolution Powder Diffractometer (HRPD) and the resolution is about half. This enables rapid phase transition type patterns to be collected on the MRPD and high resolution structure patterns on the HRPD. The HRPD has a selection of two Soller collimators before the monochromator (0.16 and 0.25°), a take-off angle of 120° and uses a germanium monochromator. Routine wavelengths are 1.371, 1.493 and 1.8834Å. The beam has dimensions 20mm wide by 50mm high and the neutron flux at 1.8834Å is approximately 8 × $10^4$$cm^{-2}sec^{-1}$. Patterns are collected from 5 - 156° (2θ) in a bank of 24 detectors (5° apart), with each detector having its own high efficiency collimator (0.17°). The minimum peak width is 0.25° and the width is less than 0.4° over most of the pattern. Data collected on this instrument is primarily for structure determination and quantitative phase analysis by multiphase Rietveld refinement of known components.

The collimation of the primary neutron beam on the MRPD can be either 0.25 or 0.5°. The monochromator consists of an array of 8 germanium single crystals with total

height 80mm and width of 50mm, with vertical focussing to increase the neutron flux at the sample position to ~10^6cm^{-2}sec^{-1}. Currently, 32 ^{3}He neutron detectors are mounted at 4° spacings at a distance of 0.7m on the 2θ drive. Each detector is mounted down-beam from a Soller collimator that has an acceptance angle of 0.35°. The instrument is designed for neutron powder diffractometry, magnetic structure determination, phase transition and residual stress measurements. *In-situ* kinetic studies of structure transformations, hydration mechanisms etc down to time resolution of ~15 minutes are also possible.

The Small Angle Neutron Scattering Instrument

The Small Angle Neutron Scattering (SANS) instrument (Figure 5) has a 5m collimation length and sample-to-detector distance range of 1.5 - 5m. The monochromator and first section of the collimator are located within the reactor containment building, and the second section of the collimator, the sample position and the detector system are located in an external laboratory. The monochromator is a double multilayer system based on a design developed at the Brookhaven National Laboratory (BNL)[4,5]. The multilayer monochromators are single d-spacing, planar geometry used in reflection mode. The large 64 × 64cm^2 active area PSD has been designed in collaboration with BNL and the ILL, and constructed at ANSTO[6]. The event readout system is based on the wire-by-wire method and the Proportional Chamber Operating System (PCOS) (LeCroy Inc USA) is used for event encoding. The detector is mounted in a vacuum tank that can be rotated +5 to -30° in 2θ to expand the accessible *q* range. The sample position provides a range of environments including a computer controlled sample changer that can operate *in vacuo*, or in the laboratory atmosphere. Since the instrument is located on a thermal neutron source, the flux is modest particularly at longer wavelengths. Nevertheless, considerable useful work can be undertaken on samples with high contrast and large physical dimensions.

The Reflectometer

A reflectometer has been designed and is presently being constructed. The instrument will be suitable for both solid and liquid surface reflectivity measurements, and polarised neutron facilities will be added in a later development phase. The neutron wavelength will be either 1.75Å (Cu) or 4.0Å (pyrolytic graphite) produced by the dog-leg monochromator. The resultant *q* range (liquids) will be 0.005 - 0.17Å^{-1} and the *q* resolution (Δq/q) will be 6%. Accessible reflectivities will be ~10^{-5}. Figure 6 is a schematic of the reflectometer. When complete, the instrument will facilitate a number of useful experiments in surface structure (monolayer surfactants, for example) and enable capability development.

NEUTRON SCATTERING SCIENCE

International scientific assessments rate the neutrons produced by research reactors as a unique and broadly applicable scientific tool for leading edge, basic and applied investigations across a wide range of scientific and technological disciplines in physics, chemistry, biology and medicine. Because neutrons probe in a non-destructive way, they are particularly suited for investigating the microstructure and properties of existing solid and liquid materials and of emerging advanced materials in the

aerospace, automotive, biotechnology, petrochemical and telecommunications fields. As each new class of materials (eg, high-temperature superconductors, carbon-cage "fullerene" molecules) has been developed, neutrons have been the primary tool for studying the properties and understanding the behaviour.

Whilst opportunities exist to pursue scientific ideas of a fundamental nature, in general, the neutron scattering science at ANSTO is problem driven. Consistent with ANSTO's mission to contribute to the vitality and competitiveness of Australian industrial research and development, the neutron scattering effort has a focus on science that support this mission. Recent and/or ongoing studies of practical significance include the following:

- To estimate the pore structure of rocks intact. Porosity in rock controls the flow of oil to the well head. An ideal bore should have a porosity that allows the oil to flow but does not allow the drill lubricant to penetrate. If possible, therefore, the location of the oil well should take this into account hence porosity needs to be determined. A problem is that conventional methods usually underestimate porosity and require the rock sample to be crushed. SANS is a means to overcome these limitations and at the same time measure the smaller pores that have the major influence on oil flow. SANS measurements are being coordinated with conventional methods.

- The interstitial distribution and site occupancy of hydrogen/deuterium in metals and alloys is important in the understanding of the application as a hydrogen storage media, and in rechargeable battery construction. For example, time resolved powder patterns of the phase transition induced in $LaNi_5$ by the passive diffusion of D_2 gas (at a pressure of 1MPa) into the lattice structure, have been obtained. The analysis has provided information on anisotropic strains in the lattice structure[7]. Extensive studies on palladium hydride have also been undertaken[8].

- To estimate the pore structure of metals and contaminated metals during the refining process. Metal extraction from an ore in a blast furnace obviously requires the addition of a firing agent. Separation of the residual firing agent from the molten metal is the key to the efficiency of the blast furnace. This separation is a wetting problem. Pore size determination of the residual is needed to determine if pore size and distribution is a factor. Pore size determination on the nano-scale must be combined with techniques that determine the interfacial structure on the atomic scale.

- To investigate template/framework interactions during the synthesis of oxides with ordered mesoporosity, controlled wall thickness and domain sizes exceeding 100nm (particularly titania) by surfactant templating. SANS contrast variation will be used for in-situ studies of the evolution of the ordered oxide framework, surfactant self-assembly processes, and surfactant/metal oxide precursor interactions in "isolation".

- Neutron diffraction has been used to study, at atomic resolution, the incorporation of various radioactive waste elements into the crystal structures of the components of the synthetic rock, synroc[9]. Synroc could

have a major impact on the storage of high level radioactive waste on a global scale[10]. In another study, SANS contrast-variation techniques were used to investigate the local oxide ultrastructure in TiO_2/ZrO_2 synroc precursor sols[11].

• Production of mesoporous titanate gel microspheres from sheared sols. To investigate the effect of shearing precursor sols on the microstructure and porosity of spray-dried gel microspheres. Simultaneous SANS/rheology studies on sheared silica sols have indicated that the application of shear during the sol-gel transition profoundly alters the properties of the resulting gels. This work is being extended to titanate-based sols, and the effect of such shearing on the properties of titanate gel microspheres (e.g. porosity) is being determined.

• Crystal growth in contaminated environments. The goal of the study is to measure crystal growth in aqueous solutions contaminated with organic impurities and propose methods to remove the contaminants. Metal refining requires the treatment of complex contaminated ores. Metals are extracted from the appropriate crystals that must be isolated and grown in this contaminated, organic environment. How these organics affect crystal growth is a major concern to industry.

• Nanocomposites. To prepare and characterise nano-structure organo-clay minerals and organo-colloid composites. The potential of using clay as the filler in a polymeric organic/inorganic composite is well understood and appreciated. The properties, however, of a composite are most influenced when the largest possible clay surface area is presented to the polymer matrix. Ideally, the clay should be fully dispersed into its constituents and bonded to the matrix using an appropriate organic intermediary or the polymeric material itself. Unfortunately, clay platelets tend to form large aggregates in the presence of organics, so normally will not disperse. The problem is to prevent this aggregation. The solution will not only allow the manufacture of nanomaterials, but will impact significantly such divers areas as pollution prevention and remediation, enhanced oil recovery and the treatment of petroleum liquids and the manufacture of cosmetics and pharmaceuticals.

• Deposition in petroleum liquids. The aims of the study are to monitor and measure asphaltenes and waxes in petroleum fluids and propose possible growth arrestors to prevent their formation. Deposition is a major economic problem in oil extraction, transport and refining particularly in Australian oil fields. Deposition can occur any time when the ambient conditions are changed eg changes in temperature and chemical composition of the petroleum fluid itself. There is an urgent need for growth arrestors to prevent the formation of asphaltenes and waxes. It is necessary to understand the physical and chemical nature of asphaltenes before any growth arrestors can be used effectively.

• Inorganic materials with ordered mesoporosity. The synthesis and structure of inorganic materials with ordered mesoporosity and surface

areas exceeding 1000m^2/g, using self-assembled surfactant systems as supramolecular templates, is an important, emerging field in Materials Science. Such materials have applications in areas as diverse as ion exchangers, selective sorption of cations and anions, filtration, catalysis, biomolecule separations, formation of semiconductor nanostructures, etc. They could also form the fundamental building blocks for the development of a range of "Smart Materials" with surfaces functionalised for a range of specific environmental responses. Although there have been numerous reports of the preparation and characterisation of templated silica systems (eg MCM-41), there have been only limited reports of templated mesoporosity in alternative metal oxide systems such as titania, tin oxide, zirconia, niobium oxide, tantalum oxide, alumina, etc. In addition, many of the reported products are not pure metal oxides, and contain residues due to incomplete removal of the templating species during sample washing and/or pyrolysis. They also contain significant quantities of disordered and/or amorphous material.

THE AUSTRALIAN NEUTRON SCATTERING COMMUNITY

The neutron scattering community in Australia is distributed throughout universities and government research organisations. Within this context, neutron scattering is identified as a national facility and this environment influence funding for its continued use and expansion. The neutron scattering team at ANSTO undertakes the development of instruments and techniques for neutron scattering and provides assistance with instrument operation and data analysis.

ANSTO's capability in neutron scattering science brings direct benefit to a number of internal programs as well as to programs driven by external partners. The internal programs are focussed either on ANSTO's broader core business (materials development and environment, for example), or problem solving for industry. The externally driven science programs are accomplished, in part, by collaborating with ANSTO scientists and, as a result of a competitive review process, are allocated beamtime on a particular instrument. The competitive review process for beamtime involves representatives of all users groups including the Australian Institute for Nuclear Science and Engineering (AINSE) (http://www.ainse.gov.au). AINSE is a consortium of 36 Australian and New Zealand universities in partnership with ANSTO, and was established by the Australian Government in 1958 to provide a mechanism for access to all the special facilities at ANSTO by universities and other tertiary institutions.

REPLACEMENT RESEARCH REACTOR PROJECT

The Australian Government has decided that HIFAR will be replaced to ensure that Australia retains the capabilities to produce its own medical and industrial radioisotopes, to conduct nuclear based research and to maintain the first hand ability to remain abreast of international and regional nuclear developments and regulation. The decision to replace the HIFAR research reactor by the year 2005 will opened up exciting new opportunities for Australia's capabilities in nuclear medicine, neutron scattering science, environmental science, education and industrial support. The modern neutron source will facilitate research and development relating to, for

example, polymers, ceramics and other new materials, life sciences and biotechnology, understanding complex industrial processes, advanced therapeutic treatment strategies with radiopharmaceuticals, and advanced environmental management processes.

Therefore, Australia's requirement is for a multipurpose neutron source with adequate facilities for radioisotope and materials irradiation plus the ability to efficiently illuminate a range of neutron beam tubes and sources. A pool-type reactor with its open architecture, adaptable design, and inherent safety and reliability features would most likely meet all the design criteria. Table 2 illustrates the difference between HIFAR and the replacement research reactor and an outline of the project strategy is presented in Figure 7.

The total project will including reactor island, all associated infrastructure and buildings. It will incorporate modern instrumentation and enhanced experimental access, high intensity neutron beams, cold and hot neutron sources. Since cold neutron sources provide the basis for many of the current advances in neutron science and technology, the replacement reactor with the most advanced cold neutron source will enable Australia's basic and applied research scientists to enter new areas of endeavour. An example is the field of nanotechnology, which requires multidisciplinary application of knowledge in physics, chemistry, mathematics, biology and electronics and where science and engineering converge at the level of individual atoms. The developments in nanotechnology will require access to intense sources of neutrons to probe the most minute structures of materials.

In order to ensure that the neutron scattering instruments will be the most appropriate for the Australian scientific and industrial research community, a consultative group was formed to identify present and future research priorities. As a consequence of extensive deliberations a suite of instruments was formulated. Table 3 lists the range of instruments considered most likely to facilitate the priority research science areas, and Figure 8 is a schematic layout of the suite of neutron scattering instruments located on a replacement research reactor. The neutron scattering facilities will be built up from the existing expertise and equipment base. When the replacement reactor commences operation in 2005 it is anticipated that four newly developed instruments will be relocated from HIFAR and four new instruments will have been built. Some of these instruments will be located on cold neutron beam guides. Three more instruments will be built in the first 5 years' of operation.

SUMMARY

Neutron scattering science is an important, integral component of the scientific and industrial research community in Australia. The present neutron source, HIFAR, and the suite of neutron scattering instruments facilitate research programs of a very high standard. The replacement research reactor due to be commissioned in 2005 will build on the strengths developed on HIFAR as well as offer new opportunities particularly in cold neutron scattering science. The combination of four decades of experience and a modern research reactor will assure ANSTO's place as a national and regional centre for neutron scattering science.

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Table 1:

Characteristics of neutron scattering instruments located on HIFAR

Instrument		Monochromator	Detectors	Neutron Wavelength (Å)	Maximum Neutron Flux ($cm^{-2}sec^{-1}$)	Resolution	Beam size (mm)
2tanA	High Resolution Single Crystal Diffractometer	Cu single crystal	1 (BF_3) (2D (16^2 elements) ^3He)	1.235	6×10^5	0.4° at 5° (2θ) 0.2° at 50° (2θ) 0.75° at 105° (2θ)	10 diameter
2tanB	Medium Resolution Single Crystal Diffractometer	pyrolytic graphite	1 (BF_3)	1.239	10^6	0.4° at 5° (2θ) 2° at 90° (2θ)	10 diameter
LONGPOL	Long Wavelength Polarised Neutron Spectrometer	pyrolytic graphite	8 (^3He)	3.6	3×10^4	$1 \leq \Delta E \leq 10meV$ $0.3 \leq q \leq 3.0Å^{-1}$	30(H)×20(V)
MRPD	Medium Resolution Powder Diffractometer	Ge multiple single crystals (focussing)	32 (^3He)	1.06 -5.0	3.8×10^5	0.4° - 0.8° (2θ)	20(H)×50(V)
HRPD	High Resolution Powder Diffractometer	Ge single crystal	24 (^3He)	1.2 - 2.96	8×10^4	0.25° - 0.4° (2θ)	20(H)×50(V)
SANS	Small Angle Neutron Scattering Instrument	multilayer	2D (128^2 elements) (^3He)	2.0 - 8.0	$\sim 10^4$	$0.08 \leq q \leq 0.1Å^{-1}$	40(H)×50(V)

Table 2.
Comparison of relevant features for the HIFAR reactor and the replacement research reactor.

Feature	HIFAR reactor	Replacement research reactor
Reactor power heat output (MW)	10-15	14-20
Neutron Flux ($\times 10^{14}$ n cm^{-2} s^{-1})	1	At least 3
Number of fuel elements	25	#
Fuel enrichment (% uranium-235)	60%	20%
Fuel load (kg uranium-235)	7	#
Architecture	Tank	Pool
Core	Loose array of fuel elements	Compact array of fuel elements
Spent fuel elements a year	37	#
Coolant	D_2O	H_2O
Reflector	D_2O	D_2O
Experimental positions*	11	17 (max)
Neutron guide hall	No	Yes
Beamline geometry	Radial	Tangential
Cold source	No	Yes
Hot source	No	Yes

Notes:
* for neutron scattering instruments
# dependent upon design

Table 3.

List of neutron scattering instruments proposed for the replacement research reactor.

	Instrument
1	Small Angle Neutron Scattering (SANS) Instrument (30 metre)
2	Horizontal Neutron Reflectometer
3	High Intensity Powder Diffractometer
4	High Resolution Powder Diffractometer
5	Polarisation Analysis Spectrometer
6	4-Circle Diffractometer
7	Quasi Laue Diffractometer
8	3-Axis Spectrometer
9	High Resolution Backscattering Spectrometer
10	Amorphous Materials Diffractometer
11	Residual Stress Diffractometer
12	Radiography Station
13	Small Angle Neutron Scattering Instrument (6 metre)
14	Neutron Spin Echo Spectrometer
15	Vertical Neutron Reflectometer
16	4-Circle Diffractometer
17,18	Thermal and Cold Neutron Instrument Development Stations

Figure 1

Photograph of the interior of the HIFAR reactor building. The top plate of the reactor tank has been removed for routine maintenance. Note the limited space for neutron scattering instruments.

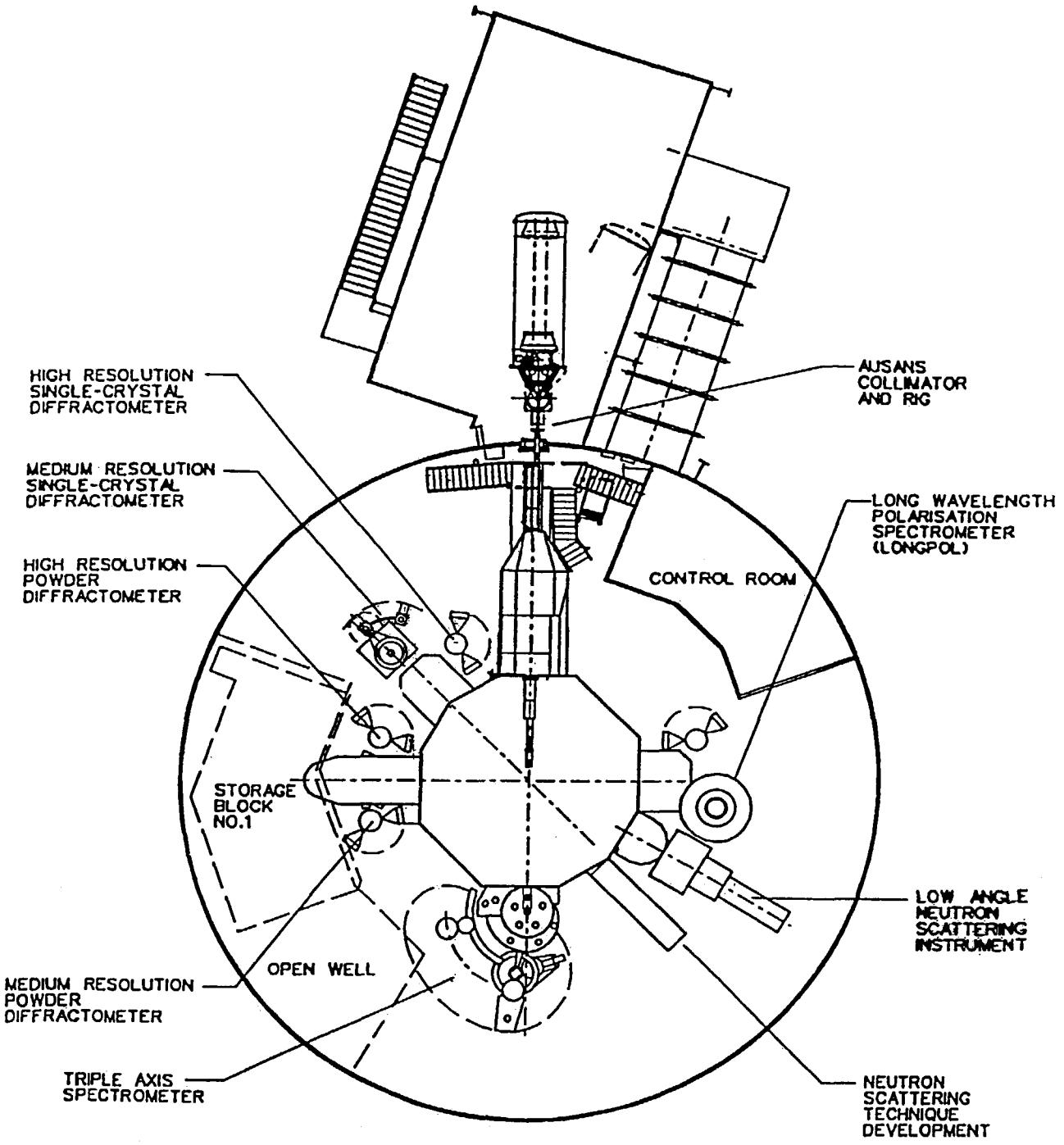

Figure 2.

Disposition of the neutron scattering instruments on HIFAR.

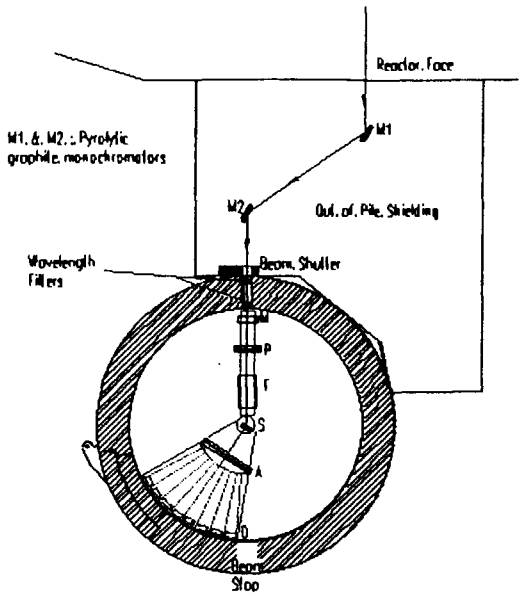

Figure 3.

Schematic of the Long Wavelength Polarised Neutron Spectrometer (LONGPOL) on HIFAR.

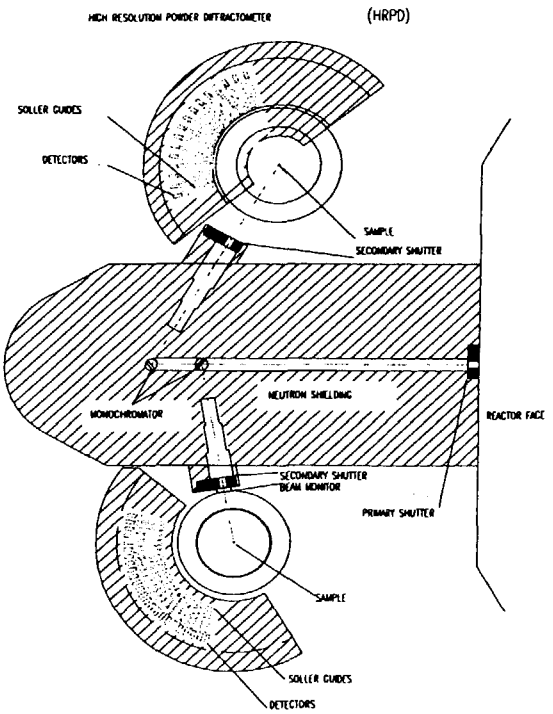

Figure 4.

Schematic of the two powder diffraction instruments (MRPD and HRPD) on HIFAR.

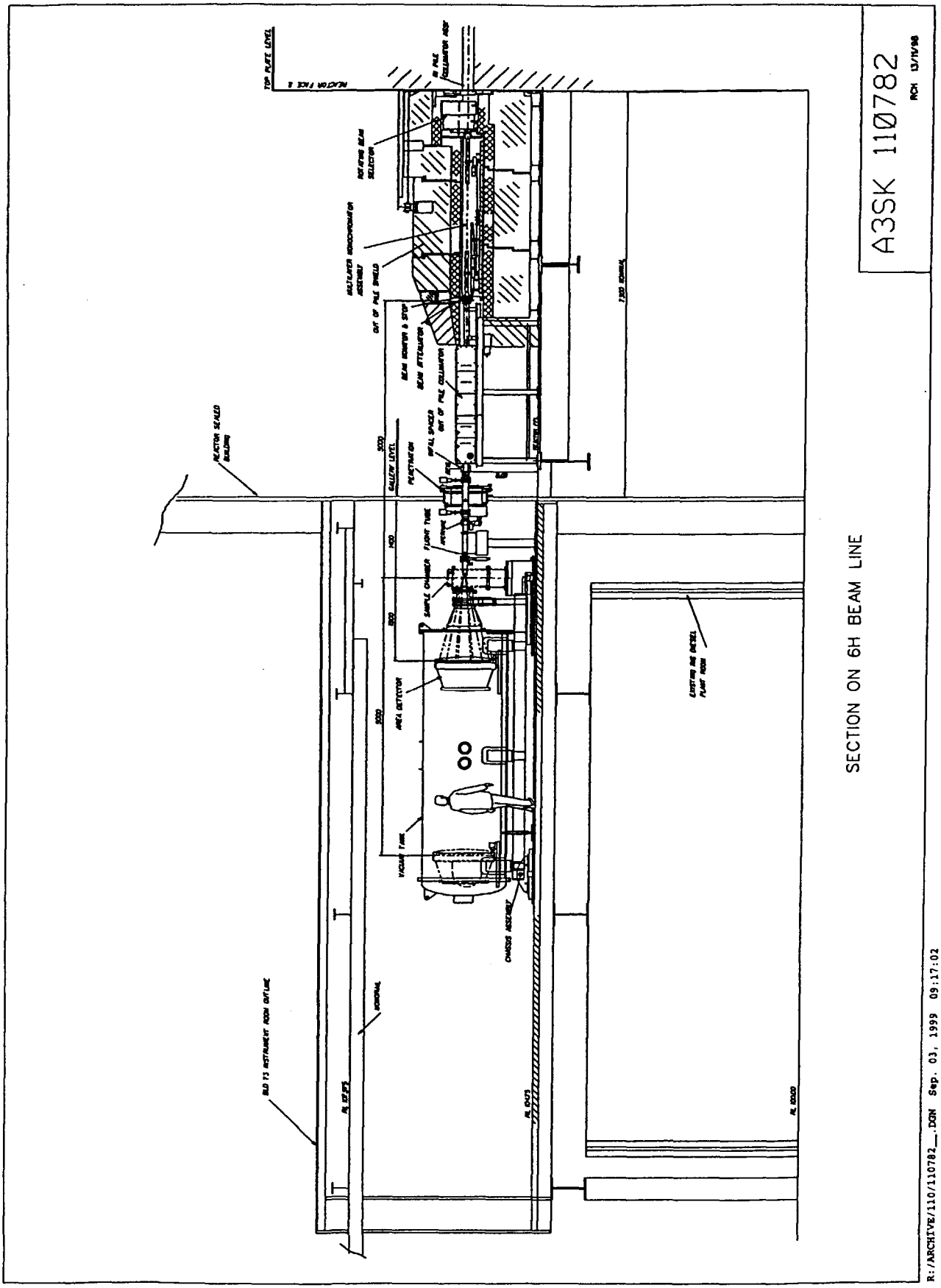

Figure 5.

Schematic of the small angle neutron scattering (SANS) instrument on HIFAR.

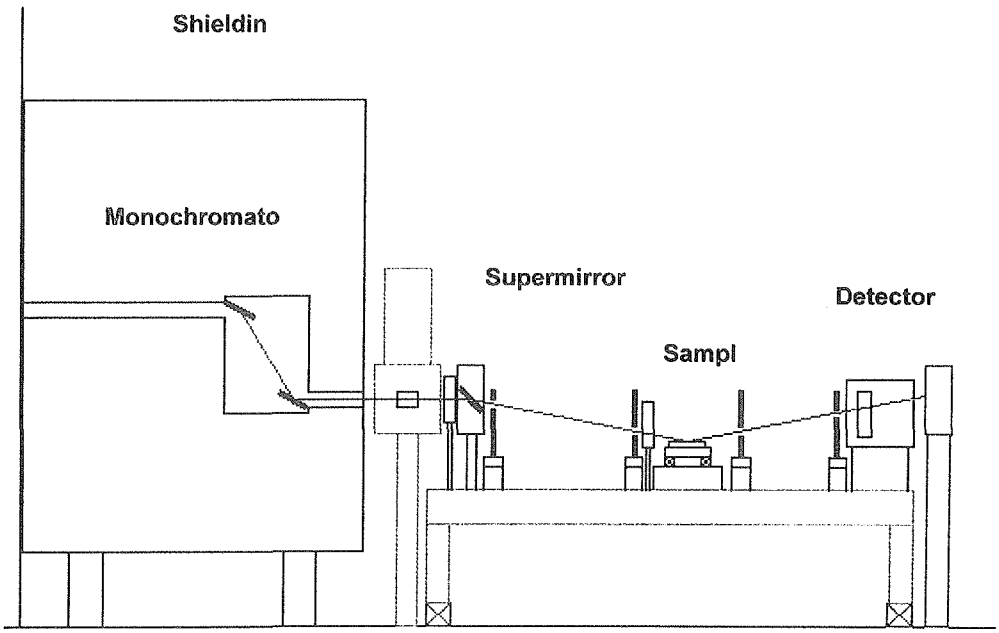

Figure 6

Schematic of the reflectometer presently under construction on HIFAR.

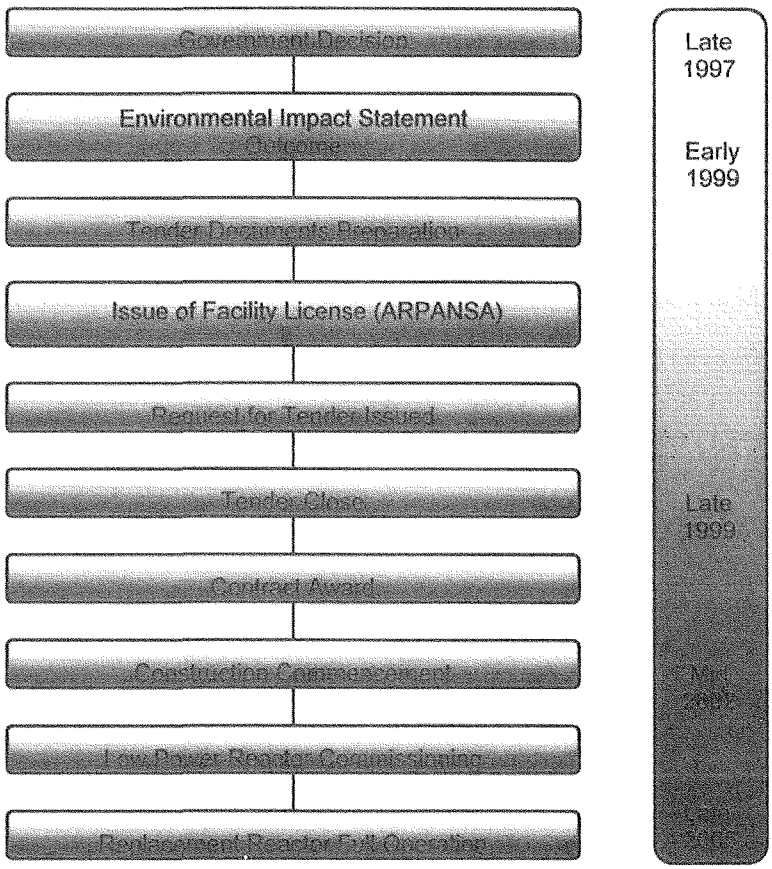

Figure 7.

Outline of the replacement research reactor project strategy.

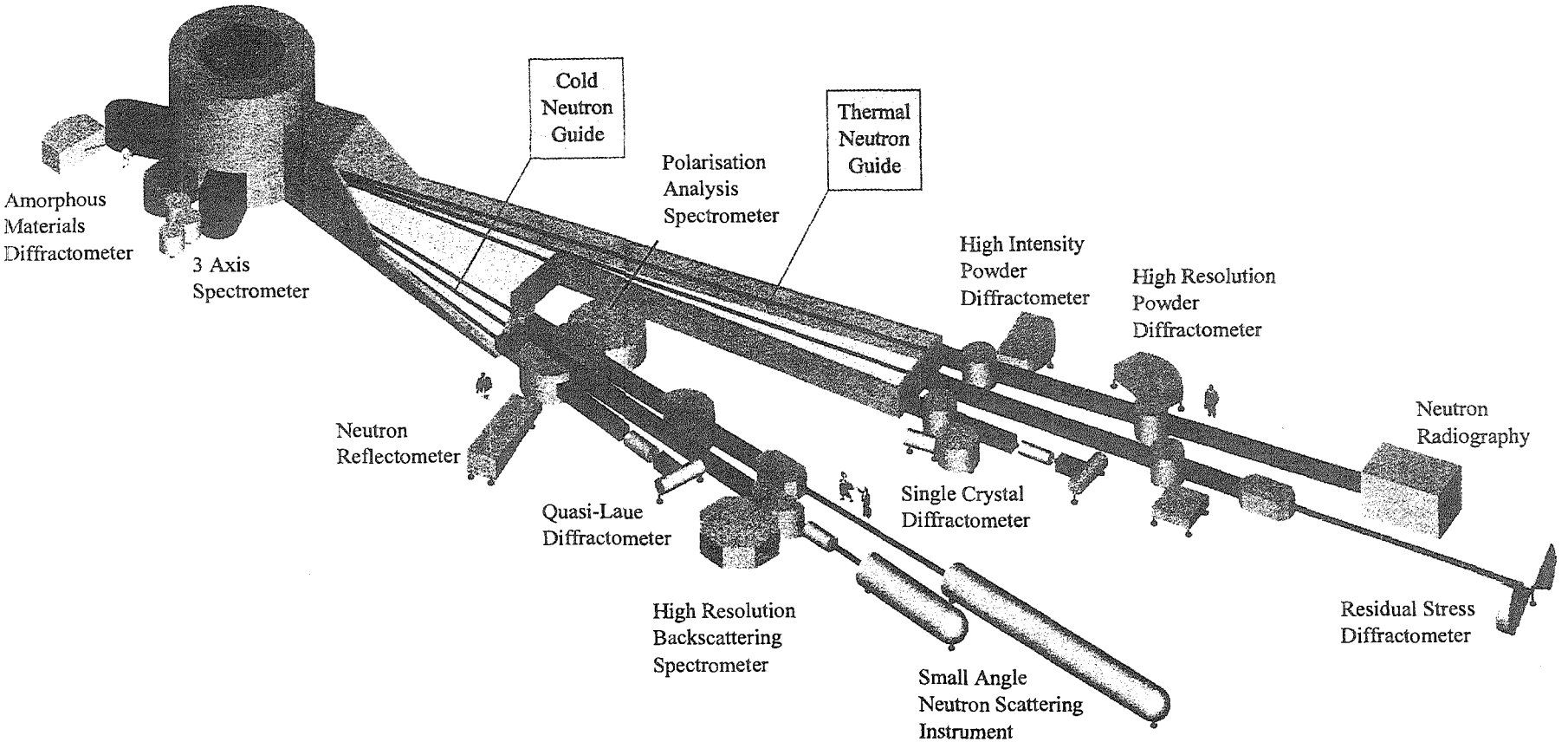

Figure 8.

Schematic layout of the neutron scattering instruments proposed to be located on the replacement research reactor (ANSTO).