"Pushing Small-Angle Neutron Scattering at OPAL to Smaller Q"

Report of a Workshop Held at the

Australian Nuclear Science and Technology Organisation

15-16 November 2007
"Pushing Small-Angle Neutron Scattering at OPAL to Smaller Q"

Report prepared by the participants of a workshop held at ANSTO, 15-16 November 2007

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6. Summary
EXECUTIVE SUMMARY

A workshop on “Pushing Small-Angle Neutron Scattering at OPAL to Smaller Q” was held at Lucas Heights, on 15-16 November 2007. Thirty-nine participants attended, from nine Australian universities, three ANSTO Institutes, the Australian Synchrotron, industry, CSIRO, and three leading overseas neutron scattering laboratories.

The purpose of the workshop was to discuss current Australian neutron scattering research on large scale structures having real space sizes ranging from 0.1 to 50 µm. Examples are polymers, precipitates, grain structures, viruses, and bacteria. The length scales of such materials exceed the measurement capabilities of conventional pinhole small-angle neutron scattering, as already implemented at OPAL in the Quokka instrument [1], by two orders of magnitude, and therefore require a different experimental approach.

The options for extending into the Ultra Small-Angle Neutron Scattering range come down to two: (1) the classical Crystal-USANS method, using perfect silicon crystals in Bragg reflection as collimators; and (2) the newer Spin-Echo-SANS method in which precessions of a polarised beam of neutrons are used to encode the scattering angle to very high precision. Initial estimates are that either can be achieved for $2-3M including labour, and that both would ideally be located on a cold-neutron guide at OPAL. Potentially, Option 2 can achieve huge $(10^3$ or more) gains in performance, especially for strong scatterers, and that this might include the majority of the science of interest to the present Australian community. Option 1 is lower in risk and, unlike Option 2, can handle weak scatterers, like solution scattering from biological molecules and defect structures in metals. There are differences between the two methods, but the choice ultimately comes down to the cross-over point (in % scattering) at which spin-echo SANS is uncompetitive – the workshop had a sense that this would likely be at ~3% - and a judgement regarding the scientific importance of the research dependent on weak-scattering samples.

In the months following the workshop, it is essential that (1) ANSTO calculate the relative intensities for samples with various scattering powers, for the two experimental approaches, and that (2) some physical samples, covering the range of scattering powers of interest, be measured using both methods at NIST and Delft respectively. Once this is done, ANSTO should be in a position to make a well-informed decision regarding which approach to take.

In summary, the workshop came to the following major conclusions:

1. There is a strong scientific case, with broad scientific application and a pre-existing user community in Australia for a USANS instrument of some description;
2. Two possible approaches exist, and the final choice between them depends on both calculations of performance and experiments at leading facilities overseas, in addition to a judgement about the relative importance and usage levels for weak and strong scattering samples;
3. Both approaches are best pursued on a cold-neutron guide, and in the short-term the best location would be on CG3, upstream of the Platypus reflectometer.
1. Introduction

A workshop on “Pushing Small-Angle Neutron Scattering at OPAL to Smaller Q” was held at Lucas Heights, on 15-16 November 2007, under the auspices of ANSTO and AINSE. Thirty-nine participants attended, from nine Australian universities, three ANSTO Institutes, the Australian Synchrotron, industry, CSIRO and three leading overseas neutron scattering laboratories.

The purpose of the workshop was to discuss current Australian neutron scattering research on large scale structures having real space sizes ranging from 0.1 to 50 μm. Examples are polymers, precipitates, grain structures, viruses, and bacteria. The length scales of such materials exceed the measurement capabilities of conventional pinhole small-angle neutron scattering (SANS), as already implemented at OPAL in the Quokka instrument [1], by two orders of magnitude, and therefore require a different experimental approach. Extending the range in this manner\(^1\) is usually termed *Ultra Small-Angle Neutron Scattering* or USANS.

A major point of this workshop was to make a start on defining specifications for a new purpose-built USANS instrument at the newly built research reactor OPAL.

Invited talks were given on two different USANS techniques: classical Crystal-USANS (or Bonse-Hart USANS, [2]) and spin-echo small-angle neutron scattering (SESANS) with emphasis on their scientific importance and potential. John Barker from NIST, USA, discussed scientific studies of polymer blends and hydrogels performed at BT-5, the currently best reactor-based crystal-USANS instrument. Markus Strobl from Hahn-Meitner Institute Berlin, Germany, presented the capabilities of the V12 double-crystal diffractometer in the area of bio- and engineering materials. Wim Bouwman from the Technical University of Delft, The Netherlands, emphasized real space resolution capabilities obtained by applying the spin-echo technique to small-angle neutron scattering, and highlighted science applications in dairy research.

In addition, all workshop participants had the opportunity to give their vision for science using low-Q neutron scattering techniques, and their interests covered a wide range of subjects from colloids, coals, complex fluids, porous materials, nanocomposites, cement, steel, to various areas in biology.

Eventually the workshop discussed the advantages and disadvantages of SESANS and conventional USANS techniques with regard to the users' fields of interest.

The workshop group photo is shown in Fig. 1 on the next page. A detailed list of attendees and affiliations is given in Appendix A.

\(^1\) There was also some discussion about using slit, rather than pinhole, apertures in conjunction with a higher resolution detector in Quokka to get part of the way to smaller Q, a method sometimes called *Very Small-angle Neutron Scattering* (VSANS).
Figure 1  Attendees of the ANSTO Workshop "Pushing Small-Angle Neutron Scattering at OPAL to Smaller Q"
2. Scientific Case for USANS Instrumentation at OPAL

2.1 Life and biomedical sciences: Structural biology and biotechnology

Biology and medical sciences have a direct interest in the study of molecular assemblies in larger length scales than are currently accessible using SANS. Contemporary research areas in biology and the life sciences include membrane biophysics, drug-delivery systems and pharmacology, dental and medical composites, biomaterials, fillings and implants. In each of these areas large length scale measurements become necessary as model biological systems begin to approach the complexity of natural systems. A particular strength of neutron scattering is that the use of selective bio-deuteration allows the investigation of the roles of individual components in complex large-scale mixtures.

Some active areas of research in Australia and New Zealand include:

- Organisation and structure of cellular components, such as nucleosomes, ribosomes, chaperonins, inclusions
- Large-scale conformational changes in regulatory proteins; DNA binding proteins, cell framework proteins etc.
- Phase transitions and supramolecular assemblies in and on membranes
- Structure of functional tissues complexes, such as neurons, synapses etc.
- Development and optimisation of drug-delivery systems, and their transport in cellular systems
- Virus structures and interactions with host cells
- Protein aggregation states, including fibril formation and growth in disease states such as amyloid plaque formations in Alzheimer's sufferers
- Biomineralization (i.e., naturally occurring mineral structures, such as bone, gall stones or kidney stones) particularly as a model for the production of new materials that mimic those naturally produced (biomimetic materials) and the investigation of diseases such as bone degeneration
- Whole tissue specimens from both animal models and human tissue can be used to unravel the relationship between the physical structure and organisation of particles and their biological function
- Use of time-resolved bent Crystal-USANS to study metabolic and toxin-induced shape changes in simple cellular systems (mammalian red blood cells)

2.2 Cement and clays

For many materials their properties at dimensions around the micron level are of great scientific and technological interest. Porosity (void structure) and particle size need to be understood so that the processes of agglomeration and water transport can be quantified in materials such as cements, oil bearing rocks and paint pigments. In particular, the properties of clays and cements are important if they are to be used as barriers to nuclear wastes and the long term ability of these barriers to stop water transport must be demonstrated. Nowadays, the techniques of SANS/USANS are now used routinely to obtain such micro-structural information. USANS measures pores and particles typically between 0.01 to 10 microns. Both gathering the data and the interpretation of the data are now fairly standard procedures.
There is considerable scientific interest in developments as illustrated by the fact that earlier this year Allen et al. published a Nature paper using USANS data to elucidate the water transport and properties of cement paste [4]. At ANSTO a team has been able to measure the growth of particles of hydrating cement as hydration proceeds and contrast this information with physical parameters such as heat of hydration. These preliminary results have generated considerable interest and have demonstrated the utility of the technique to obtain information on particle growth that could not have been measured in any other way. The potential of this technique is in monitoring the variations to growth by admixtures. For example by about 1980 the by-product silica fume (of about 0.1 micron) was used to produce high strength concrete with strength of over 100 MPa - resulting in a strength gain of at least a factor of 2. In the 1990's silica fume from Western Australia was used in NSW but more recently there have been a number of attempts to produce more cost-effective materials which can be used as substitutes. USANS not only models particle growth but also the infilling of the pores in the paste - information needed to understand the durability of concrete. Similarly USANS can monitor pore size difference in concrete caused by additives such as ground granulated blast furnace slag, which controls durability without the modifications caused by the drying of concrete. In concrete these capillary pores (sized below 10 microns) control properties such as durability, water transport, and shrinkage.

2.3 Porosity
In the case of zeolites, catalysts, cement, geopolymers (see below), carbon aerogels, voids in metals and alloys, USANS has the capability of studying porosity as well as particle size but differs from conventional techniques in measuring both closed and open pores. The smaller pore systems are synthesized in tens-of-thousands of tons, as supports for catalysts, with unique properties. Aluminium/silicon based inorganic polymers are being investigated as a replacement for structural materials such as cement. As with cement, the effect of microstructure on the mechanical properties of inorganic polymers is not well understood. The influence of the size of aggregates > several 100 nm in the inorganic polymer structure needs to be investigated.

Figure 2 Cracks initiated by water transport causing loss of durability in what should be high quality concrete [3]
2.4 Geopolymers
The term "geopolymer" is a name given to a broad class of materials. A typical synthesis involved mixing metakaolin (calcined kaolinite) with a highly alkaline potassium or sodium silicate solution, followed by curing at a temperature below 100°C for several hours. Geopolymers can be synthesized to have the following physical properties: high compressive strength, little or no shrinkage during curing and heat resistance. The compressive strength is strongly dependent on processing condition changes and is the most commonly reported physical property. Potentially, they are suitable for many applications such as an alternative to Ordinary Portland Cement, high temperature composites, radioactive waste encapsulation, castable ceramics and others. Fly-ash based geopolymer concrete as an Ordinary Portland Cement concrete alternative has been manufactured on a commercial scale and is attractive environmentally from a CO₂ emissions perspective. Geopolymers contain porosity and inhomogeneities on a length scale from nm's to several microns in size and USANS will give information on the micron size structure, which can be used to optimise the physical properties of the geopolymers.

2.5 Hydrogen storage and fuel cells
The most important factor to be considered regarding hydrogen being used as an energy carrier in various applications is its safe and efficient storage. The current technologies for hydrogen storage are either unsafe or expensive as in the case of compressed gas storage or liquefaction respectively. Therefore, alternative methods of storing hydrogen in the solid state are being investigated by combination with such materials as metal hydrides, high-surface-area materials such as metal-organic frameworks and carbon aerogels, carbon-based materials, nanoparticulate metals and complex hydrides. The fractal structure of carbon aerogels occurs on the nm to micron scale and USANS is an ideal technique to study the micron-scale structure. The clustering of nanoparticles into micron size clusters can also be studied using USANS. In addition as hydrogen is introduced into the material, USANS will be able to map the phase changes and inhomogeneities that manifest themselves on the micron scale in-situ. The micron structure of materials used in fuel cells such as Nafion can be determined using the USANS technique.

2.6 Metallurgy
Some areas of interest from a metallurgical perspective where SANS/USANS may have advantages over other analytical techniques in relation to metals technology would be:

2.6.1 In-situ study of dissolution and precipitation phenomena
The precipitation of non-metallic and intermetallic compounds for solid solution in metals is of widespread interest due to the importance of such particles on the microstructure and properties of metals, industrial alloys and coatings. In the case of steels, the precipitation of various particles from solid solution in austenite during thermo-mechanical processing can have very significant influences on the microstructure and mechanical properties of the steel. Precipitates of interest include MnS, AlN, microalloy carbonitrides (Nb, V, Ti) (C, N) and boron nitrides and iron borocarbides. These particles may range in size from several nm to several microns. All of these particles or precipitates have varying solubility in austenite and will precipitate at different temperatures both during isothermal or continuous cooling and in response to mechanical strain (rolling deformation). It has often been difficult to interpret precipitation behaviour in ex-situ experiments since the transformation of austenite to
ferrite greatly complicates the picture. The ability to study in-situ precipitation behaviour in the austenite of various steels may be possible using high-temperature SANS experiments. In addition traditional microscopic techniques have the drawback of requiring large numbers of sections or fields to obtain representative population data. SANS would enable rather large and more representative volumes of metal to be characterised.

2.6.2 In-situ recrystallization behaviour and phase transformations in thermo-mechanical processing
For many years there has been ongoing debate about the fundamental mechanisms of recrystallization and phase transformation in steels and other alloys. Many of the difficulties and confusion arising from the research has been due to the limitations of the experimental techniques used to study the phenomenon and the inability to obtain real time in-situ information under the conditions of interest in relation to temperature and deformation parameters. USANS could potentially provide information about the progress of recrystallization and may be able to distinguish between the varying recrystallization mechanisms such as static, dynamic and metadynamic recrystallization. The grain sizes of interest would be in the range of about a micron to 100 microns. In the case of steels, it may not be possible to use SESANS for such studies since it appears not to be applicable for magnetic materials. However, analogous materials such as Ti alloys could possibly be studied as means of unravelling the mechanisms at play in steels and other ferrous alloys.

2.6.3 Microstructures of Al-Zn metallic coatings
The microstructure of the ZINCALUME® Steel metallic coating consists of an aluminium matrix, with cored zinc-rich interdendritic regions. These zinc-rich regions will vary, depending on the composition and cooling rate, from a zinc-rich aluminium phase to actual zinc phases. Phase sizes vary from nanometres to about 1 um. A study of these phase formations, at room temperature and during cooling from 600°C, at cooling rates of about 10°C/sec, would increase the understanding of the corrosion behaviour. This work could be done with Crystal-USANS or SESANS, although the magnetic properties of the steel substrate may interfere with SESANS.

2.6.4 Microstructures of thin films
Studies of the microstructures, crystallography and porosity of thin metallic (or organic) films (10-80 nm) are required to understand their "in service" behaviour.

Types of films would be
- Metallic-Plasma Vapour Deposition
- Metallic-Vacuum Vapour Deposition
- Inorganic crystalline passivation films
- Organic films

2.6.5 Analysis of ores and sinters
The opportunity to conduct analyses of ore bodies (coke, sinter, iron sands, accretions etc.), looking at phase compositions, distributions and porosity may be possible with SANS/Crystal-USANS/SESANS, and some preliminary investigations would be of interest.
2.7 Oil and gas

CSIRO and ANSTO have an existing collaboration through CSIRO Wealth from Oceans Flagship Project to look at new technologies to enable Australia to access stranded gas reserves off the North West Shelf. There are many technologies currently under development by CSIRO within the Platform-Free Fields Project and one key issue is flow assurance in long pipelines.

![Diagram of pipeline design](image)

**Figure 3** Improved pipeline design will help realize the vision of platform free fields [5]

The major issue for flow assurance (particularly in the case of wet gas, found off the North West Shelf) will be the formation of gas hydrates. Gas hydrates are ice-like structures that form around the gas molecules to form "cages" which then agglomerate to form large plugs which block the pipelines. They are thermodynamically favoured by high pressure and low temperature, so the risk of hydrate formation is increased in deeper water and with longer pipelines. Formation of a hydrate plug leads to loss of production and they can be expensive and dangerous to remove.
Existing methods of hydrate mitigation include the use of thermodynamic inhibitors or polymeric based materials known as kinetic inhibitors. Both have problems associated with their use in a "platform free" environment, so improved inhibitors or new methods of hydrate mitigation must be identified.

The process of hydrate nucleation and growth is poorly understood, so fundamental work is required to accurately characterise these processes in order to design better inhibitors and then characterise the processes that make the inhibitors more effective.

Several other opportunities have also been identified within the area of materials chemistry for the oil and gas industry. These include the development of permeability modifiers to improve the efficiency of production from oil-bearing rock (thereby extending the useful life of a well-bore and making it more viable financially) and development of materials to improve the strength of a well bore (which will reduce the set up and operational expense of a well-bore).
2.8 Nanotechnologies, magnetic materials

Nanomagnetic materials: The physical properties and magnetic behaviour of magnetic materials are dramatically influenced by the particle size, shape and spatial distribution. Studies on structured magnetic materials, necessary to understand how to overcome the superparamagnetic limit are of paramount importance for the development of new magnetic devices. Magnetic particles in the nanoscale have shown tantalising evidence of macroscopic quantum tunnelling of magnetization, however the particles distribution, their ability to form ordered structures, and their interactions are not yet well studied. Developments in this direction are extremely important for new developments in spintronics. The ability to inject a current with spin up (down) on a semiconductor will open the way for the development of quantum computing. On the other hand, aside from possible new technology, the study of phenomena happening at the nanoscale, at the border between the solid state and physics of the single atom, will shed light on the quantum mechanical phenomena. The combination of the magnetic moment of neutrons, with the ability of scattering techniques to probe nanoscale phenomena makes SANS and spectroscopic techniques ideal tools for the study of single nanoparticles, however, the ability to study also their assemblage remains an important and fundamental task. Although, in the field of magnetic nanoparticles we observe a race toward smaller structures it remains of paramount importance to study the ability of nanoparticles and organic magnets to create ordered self-assembled structures in the micrometric range. USANS coupled with SANS seems to be the tool of choice to investigate such self assembly over large size ranges, the ability to create ordered superstructures and the presence of defects and their effects on the magnetic properties. The application of nanomagnets goes from the electronic industry (for example CoPt or FePt nanoparticles) to the biomedical/biotechnological field including magnetically targeted drug delivery, magnetic hyperthermia treatment, and novel biosensors. New engineered complex materials are currently under study: supported magnetic nanoparticles in the range of 1 to 10 nm coated with proteins or polymers offer tantalizing new technological possibilities only if such novel materials will deliver the ability to self structure over micrometric ranges. Furthermore, the study of magnetic molecules would take advantage of the possibilities offered by USANS, in particular the determination of the growth mode and their influence on the magnetic moments. In combination with spectroscopic techniques at the nanometric scale (e.g. X-ray magnetic dichroism) it offers the possibility to determine quantitatively the total magnetic moments as well as the spin and orbital moments in connection with the structural landscape. In the future the combination of organic and organic assembled magnets could open the way to a new class of self-assembled magnetic structures. The study of such materials requires strict environmental control, including very low temperatures, high magnetic fields as well as controlled environmental conditions (ultra high vacuum or noble-gas atmosphere).

Furthermore relevant areas of research include magnetism and superconductivity, spin glasses, vortex dynamics, textured materials, magnetoelectronic, magnetic semiconductors, and long-range spin correlations.
2.9 Colloids, emulsions, micellar systems
Complex fluids, containing structures and complexes in nanometer and much larger length scales, have widely varying physical properties and are extensively used in food (e.g. ice cream, mayonnaise, milk), cosmetic/personal care, pharmaceuticals and drug-delivery, and the mining industry. In these length-scales, which are inaccessible to standard pinhole-SANS/SAXS measurements, lie some of the organisational features that dictate the bulk rheological and stability properties of solutions. Therefore, a USANS technique in parallel with isotopic substitution enables the detailed study of these systems and their internal organisation, and the penetrating power of neutrons allow the application of industrially relevant external pressures and stresses to be followed in-situ.
Complex fluids can also be used as templates for the synthesis of new materials such as extremely low-density silicas or high-surface area catalyst structures. For the polymer industry, benefits include an improved understanding of the miscibility of polymers and solvents, an increased product range, improved moulding and improved recycling, plastic degradation and responsive materials.

2.10 Thermo-mechanical processes
Thermo-mechanical processes play an important role in the manufacture and shaping of metallic products in large industries like steel making and light metal production but also in very sophisticated niches like the gold wiring of semiconductor chips. On the other hand, work pieces may suffer from aging and fatigue which themselves are thermo-mechanical processes. Maybe more importantly than shaping the macroscopic form of the product is engineering its microstructure, which can influence the mechanical and physical properties by orders of magnitude. Everybody knows that a forged sword has much higher mechanical strength than a cast blade made out of the same composition but differing substantially in the finesse of the microstructure.
Microstructures are often arrangements off the thermodynamic equilibrium and thus must be engineered as a function of temperature and strain parameters but also evolve under those in their applications. They can be represented by a size and orientation distribution of just one material or complicated multi phase systems and precipitates. Therefore, they occur on all length scales from the atomic to the millimeter range.
Examples are crystallographic order and disorder; phase composition; dislocations and stacking faults; nano-grained materials; precipitates; ultrafine lamellae; segregates; grain growth and inclusions; pores and voids; arrangement and shape of grains; domain structures; texture; single crystalline materials. The physical aspects behind this are diffusion; phase transformation; nucleation and growth; segregation; strain and stress; misfit; orientation relationships; kinetics; crystallographic deformation systems; surface energy; rheology.

2.11 Equal Channel Angular Pressing, ECAP
ECAP is a method of Severe Plastic Deformation and can be used for grain refinement and the consolidation of powders, even non-mixable. Nano-crystalline solid blocks containing Al and $\gamma$-Al$_2$O$_3$ grains have been obtained after processing oxidized Al powder after 1 pass ECAP, 400°C, 0.2 mm/min and 200 MPa back pressure. There are numerous processing parameters to be investigated and rapid characterization is essential.

2.12 Ultrafine lamellar microstructures
Ultrafine lamellar microstructures in $\gamma$-based TiAl intermetallics develop upon heating in the non-equilibrium, quenched $\alpha$-phase and evolve in time into the micrometer range. This slow phase transformation changes the mechanical properties of the material and needs to be understood on all length scales in the bulk of the material.
2.13 Particles in cast magnesium

Particles in cast magnesium in form of pores, segregates and precipitates form during the solidification process and influence the mechanical properties which are important for further treatment. The length scales of these particles range from the nanometer to 100 um range and aspect ratios can be quite important.

2.14 Materials of the Future

Nature has the ability to design materials that are able to adapt to their environment. For example, our bodies' structural and biological functions are accomplished by the action of disordered, nanoporous materials from cell membranes to bone. Not only are these materials defect-tolerant, they are self-healing. Hence natural systems are often made up
of some fraction of the atoms are in a configuration to sense, and another fraction is earmarked to be mobile and ready to rush to the scene of a fracture or breach. The future generation of new materials will have a feedback loop between the material's capacity for response and a response that mitigates damage and renews the capacity to respond. Recent achievement of synthetic analogues of nacre ("mother of pearl") and bones represents a fundamental breakthrough in nanostructured materials design. The design of defect tolerant and self-healing material and strategies for their engineering are exciting questions that USANS could contribute significantly. The ability to understand the space available to molecular transport, interfaces, chain packing, and as well as gradient properties, of both ex-situ biological and synthetic samples are critical to the engineering of effective materials. The ability to monitor reaction kinetics and the adaptation of materials in various environments is key engineering the advanced materials both for structural applications and soft matter electronic materials of the future.

3. Low-Q Neutron Scattering: An Introduction

3.1 Various techniques to probe size scales
Figure 10 shows what experimental techniques are available to probe size scales ranging from the atomic scale up to 100 µm.

![Figure 10 Size scales probed by various techniques](image)

In the area of crystallography atomic structures can be studied by applying diffraction techniques using neutrons, X-rays, and electrons. Microstructures of e.g. proteins, micelles, and porous media show length scales ranging from 10 Å to 1000 Å (10⁻³ to 0.1...
μm) which can be probed by small-angle scattering (bulk samples) and reflectivity measurements (thin film samples) using neutrons (SANS) and X-rays (SAXS) as well as a different SANS technique based on spin-echo methods (SESANS). Ultra Small-Angle Scattering methods using neutrons (USANS) as well as X-rays (USAXS) allow investigating large scale structures of e.g. viruses, bacteria, and grains (a few tens of μm).

Note that due to the low penetration depth of X-rays, SAXS cannot (easily) be employed to study thick samples or samples requiring complex containers or other sample environment. Light scattering (optical microscopy) cannot be used to study optically opaque samples.

### 3.2 Why use neutrons as opposed to X-rays?

Neutrons provide a unique probe for the study of structure due to the following reasons:

- Neutrons scatter from nuclei, X-rays from the electron distribution in the atoms.

![Figure 11](image)

*Figure 11* Schematic scattering lengths of different elements as "seen" by X-rays and neutrons

Between elements, the scattering length of X-rays varies monotonically with increasing atomic number, while for neutrons it varies in a non-uniform fashion as well as between different isotopes of the same element.

⇒ Whereas X-rays are almost blind to hydrogen, neutrons "see" hydrogen atoms plus neutrons are sensitive to isotopic compositions of samples providing the ability to manipulate local scattering amplitudes via isotopic labelling or an appropriate choice of solvent (contrast variation).

- Neutrons cause minimal radiation damage.

- Neutrons possess a magnetic moment.
  ⇒ Neutrons can be used to study magnetic properties of the sample.

- Polarized neutrons can be used for studying magnetic materials.
  ⇒ Separation of nuclear and magnetic contributions to the scattering signal. Furthermore it allows a contrast variation analysis to study magnetization, density, and composition profiles at interfaces and surfaces in more detail.

- Neutrons possess a high bulk penetrating power.
  ⇒ Because of their weak interaction with and consequent deep penetration into matter, neutrons allow investigating properties of the bulk.
• Small absorption for most elements.
  ⇒ Strong neutron transparent materials, like aluminium quartz and sapphire, are available as sample-environment equipment.

3.3 Neutron techniques to probe low-Q regions
In neutron scattering experiments, the sample is exposed to the neutron beam, and one measures the intensity of the scattered beam as a function of the scattering angle θ and the wavelength λ of the probe, see illustration in Fig. 12.

![Figure 12 Basic set-up of a neutron scattering experiment [12]](image)

Q is the scattering vector (or: momentum transfer) and is related to the length scale D via

\[ Q = \frac{4\pi}{\lambda} \cdot \sin\theta = \frac{2\pi}{D} \]

Thus, investigating large scale structures requires experiments involving small values of the momentum transfer Q. Listed below in Tab. 1 are different low-Q techniques, their measurable size range D as well as the according Q-range.

<table>
<thead>
<tr>
<th>Low-Q Technique</th>
<th>Size Range D (µm)</th>
<th>Q-range (Å⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pinhole SANS</td>
<td>0.001 - 0.1</td>
<td>10⁻¹ - 10⁻³</td>
</tr>
<tr>
<td>VSANS</td>
<td>0.1 - 1</td>
<td>10⁻³ - 10⁻⁴</td>
</tr>
<tr>
<td>Crystal-USANS</td>
<td>0.1 - 10</td>
<td>10⁻³ - 10⁻⁵</td>
</tr>
<tr>
<td>SESANS</td>
<td>0.01 - 10</td>
<td>10⁻² - 10⁻⁵</td>
</tr>
</tbody>
</table>

*Table 1 Low-Q techniques and their accessible D and Q ranges, respectively*
A schematic intensity plot for small-angle scattering from disordered systems is illustrated in Fig. 13. The plot shows intensity I as a function of Q on a log-log scale. Data analysis performed in the different regions (as indicated by arrows) yields the following information on the sample: interparticle correlation functions (i.e. the probability that there is a particle at \( R \) if there is one in the origin); particle shapes and orientations (particular shapes and orientations result in different form factors \( F(Q) \)); radius of gyration, \( r_g \), which gives the size of the particle by fitting SANS data in the Guinier region; fractal dimension, and particle surface properties.

**Figure 13**  Schematic intensity plot for small-angle scattering from disordered systems

In the following, we will focus on the Crystal-USANS and SESANS technique, respectively. Brief discussions of Pinhole-SANS, Multi- (MSANS) and Very Small-Angle Neutron Scattering (VSANS) can be found in the appendices.

### 3.3.1 Crystal-USANS

For extending the Q-range of SANS to smaller Q values, a neutron beam with an extremely sharp angular profile is required. Such beam can be obtained by diffraction from a perfect crystal because the Bragg reflectivity function from such crystal is mostly confined in a very narrow, typically a few seconds of arcs, range of angles. [13,14]
Fig. 14 shows the reflectivity function $R(y)$ derived by Darwin [15] for an infinitely thick and transparent crystal with

\[
R_D(y) = \begin{cases} 
1 & |y| \leq 1 \\
[y] - (y^2 - 1)^{0.5} & |y| > 1 
\end{cases}
\]

where $y$ is a dimensionless angular parameter of the dynamical diffraction theory, $y = 0 - \theta_B/\theta_D$, and $\theta$ the angle of incidence.

\[2\delta\theta(n) = \frac{(|F(n)|/\pi V_0) \cdot [(\lambda(n)/n)^2/\sin 2\theta_B]}{\delta\theta_D}\]

where $|F(n)|$: Magnitude of structure factor
$V_0$: Unit cell volume
$\lambda$: Wavelength
$\theta_B$: Bragg angle
$n$: Order of reflection
The left part of the equation above is determined by the material of the crystal (typically Si), the right part by the experimental set-up.

The minimal value of the scattering vector $Q = 4\pi \sin \theta / \lambda$, $Q_{\text{min}}$, given by

$$Q_{\text{min}}(n) = (4F(n)/ V_0)[(\lambda(n)/n) \cdot \sin \theta]$$

determines the Q-resolution, $\Delta Q$, according to

$$\Delta Q(n) = Q_{\text{min}}(n) \approx 4\pi \cdot \delta \theta(n)/\lambda(n)$$

Example:

<table>
<thead>
<tr>
<th>Crystal ( \text{Si(111)} )</th>
<th>( \lambda = 4.43 \text{ Å} )</th>
<th>( \theta_B = 45^\circ )</th>
<th>( 2\delta \theta_B = 3.7727 \text{ arcsec} )</th>
<th>( Q_{\text{min}} = 2.59 \cdot 10^{-5} \text{ Å}^{-1} )</th>
</tr>
</thead>
</table>

Table 2
Example of Darwin Plateau width and according $Q_{\text{min}}$

Crystal-USANS instruments use single Bragg diffraction at two perfect crystals in a non-dispersive arrangement ("double-crystal diffractometer"). Figure 15 shows rocking curves of Crystal-USANS measured for single-single bounce and triple-triple-bounce schemes.

![Figure 15](image)

**Figure 15** Rocking curves of Crystal-USANS diffractometer, from top to bottom [16]: Single-single bounce scheme, i) Triple-triple bounce scheme with channel-cut crystals, iii) Triple-triple bounce scheme with modified channel-cut crystals containing Cd absorber, iv) same crystals as iii) with etched surfaces
As can be seen from Fig. 15, operating a Crystal-USANS with single-bounce crystals [cf. curve i), filled diamonds] is handicapped by the wings of the reflectivity function, resulting in a decreased signal-to-noise ratio. This wing problem can be partially overcome by using triple-bounce, channel-cut crystals [cf. curve ii), open diamonds]. Such crystals suppress the wings by one order of magnitude as compared to the single-bounce configuration while leaving the plateau unaffected (because there the reflectivity is essentially equal to unity). The multi-bounce configuration also suppresses thermal diffuse scattering.

A further decrease in wing intensity [cf. curve iii), open circles] is achieved by suppressing contaminations from first back-face reflections, see Fig. 16 [17]. Cd inserts placed in the middle of the long crystal wall block neutrons from exiting the crystal after undergoing only a single reflection at the back-face (of the long wall). Other background-producing effects include surface irregularities and surface strain which can be controlled by carefully polishing and etching of the crystals [cf. curve iv), filled circles].

![Figure 16](https://via.placeholder.com/150)

_Figure 16_ Contamination from first back-face reflections can be avoided by Cd inserts

Figure 17 shows the typical schematic layout of a Crystal-USANS ("Bonse-Hart Camera"). The central parts of such instrument are two perfect Si single crystals (labeled monochromator and analyzer) mounted on an optical bench. When a sample is inserted between these two components, small-angle scattering spreads the highly collimated beam and this broadening is exhibited in the difference between the two rocking curves of the analyzer, with and without sample (i.e. the second rocking curve needs to be subtracted from the first one). Thus, the profile of the rocking curve in the absence of a sample characterizes the sensitivity and ultimate resolution, and is the critical parameter of the Crystal-USANS.
Figure 17 Sketch of Bonse-Hart Camera with triple-bounce monochromator and analyzer crystals [18]

Figure 18 shows a rocking curve (shown in green) for the BT-5 instrument at NIST using a 44 mm diameter sample aperture, exhibiting six orders of magnitude in signal-to-noise (S/N). The intensity is in units of counts per second ($s^{-1}$) in the detector. The blue curve is the theoretical profile for a pair of triple-bounce crystals. [19]

Figure 18 Rocking curves of perfect crystal [19] 
Blue: Theoretical, Green: Experimental (see text)
Crystal-USANS Instruments around the world use two different types of crystals:

i) Triple-bounce or multi-bounce crystals
   In use at

- Atom Institute Vienna, Austria
  http://www.ati.ac.at/~neutropt/experiments/USANS/usans.html

- Geesthacht Neutron Facility, Germany
  http://www.gkss.de/pages.php?page=w_abt_genesys_dcd.html&language=d&version=g

- Hahn-Meitner Institute Berlin, Germany

- ILL Grenoble, France
  http://www.ill.fr/s18/home/

- JRR-3M Tokyo, Japan
  http://www.issp.u-tokyo.ac.jp/labs/neutron/inst/ULS/

- FZ Juelich (instrument no longer available), Germany
  http://www.fz-juelich.de/iff/pics_pdf/wns/exp_DKD.pdf

- NIST, USA
  http://www.ncnr.nist.gov/instruments/usans/

- ORNL, USA (instrument no longer existing)

- PSI Switzerland
  http://morpheus.web.psi.ch/echo.html

- Serpong, Indonesia
  http://www.informaworld.com/smpp/content?content=10.1080/10448630600978400

 Figure 19  Triple-bounce channel-cut crystal
Advantages
- High peak to background ratio
- Better resolution
- Superior Q range

Disadvantage
- Time required for a typical experiment is about 24 h

ii) Bent crystals
In use at
- Hahn-Meitner Institute Berlin, Germany
- NPI Prague, Czech Republic

In contrast to conventional double-crystal arrangements, the fully asymmetric diffraction geometry on the elastically bent Si analyzer is employed to transfer the angular distribution of the scattered neutrons to the spatial distribution and to analyze the whole scattering curve by a one-dimensional position sensitive detector.

![V12a multipurpose instrument at HMI using bent crystals](image)

**Figure 20** V12a multipurpose instrument at HMI using bent crystals [20]

Advantage
- Determination of typical USANS patterns in 10 min
Disadvantages
- High background
- Lower resolution
- Lower Q range

Example: Combined results obtained from Pinhole-SANS and Crystal-USANS data

The angular distribution of neutrons scattered by a multi-component Al alloy was measured at a Crystal-USANS and at a SANS instrument with a data overlap at Q = 2·10^{-2} nm^{-1}. The differential scattering cross section (scattered neutron intensity) is shown as a function of the scattering vector. The analysis of the scattering profile yields the volume distribution function of the precipitates as shown in the inset. The full line is the theoretical scattering curve calculated from this volume distribution. The pinhole-SANS data allow to determine the size distribution of precipitates having radii ≤ 300 nm = 0.3 µm (the size distribution function shows a maximum at R = 50 nm = 0.05 µm) while the Crystal-USANS data probe larger length scales (≥ 300 nm = 0.3 µm). As can be seen, the USANS analysis indicates that large precipitates are formed having radii of up to 10 µm.
3.3.2 SESANS

SESANS is a novel method to determine the structure of materials in real space [22]. The use of divergent neutron beams (i.e. no collimation) results in a high beam intensity orders of magnitude higher than for conventional pinhole SANS or Crystal-USANS, and length scales up to 10 µm can be studied. The Q resolution range overlaps that obtained with USANS.

The SESANS experiment detects changes in neutron polarization after neutrons have passed through two equal and opposite Larmor precession fields located before (red diamond) and after the sample (blue diamond). A schematic presentation of the experimental set-up is shown in Fig. 22.

![Figure 22 Schematic SESANS arrangement [23]](image)

In the absence of the scattering sample, the neutron polarization vector will precess in opposite directions by an equal amount within each of the two precession fields. At the exit of the second Larmor device, the net precession becomes zero and the neutron polarization vector is restored to its original state from before the precession had started. In the presence of a scattering sample, the scattered neutrons will have different path lengths within the second Larmor device. The opposing precessions of these scattered neutrons within the two Larmor fields will no longer cancel each other. The final polarization of these scattered neutrons will be different from that of the transmitted ones. Thus, the neutron scattering angles are encoded into the neutron polarization. The detection of the final neutron polarization at the analyzer yields a real space correlation function of the sample, the spin-echo length \( z \) (= length between two scattering volumes in the sample) with

\[
z = \frac{c \cdot \lambda^2 \cdot B \cdot L \cdot \cot \theta_0}{2\pi}
\]

where

- \( c \): Larmor precession constant
- \( \lambda \): Wavelength
- \( B \): Applied magnetic field
- \( L \): Length of precession arms

The spin-echo correlation function \( z \) can be probed by varying the magnetic field \( B \) or the length \( L \) of one of the two precession fields or by probing with different neutron wavelengths \( \lambda \).
Figure 23 shows a SESANS measurement on deuterated cyclohexane containing sterically stabilized silica particles [24]. The normalized polarization of the neutron beam is plotted as a function of the spin-echo length $z$. A saturation level of the depolarization is reached at $z = 298$ nm corresponding to a particle size of $d = 298$ nm.

![Figure 23](image)

**Figure 23** Sterically stabilized silica particles $d=298$ nm in deuterated cyclohexane [24]
4. A USANS Instrument for OPAL

While the atomic and the larger micrometer to millimeter structures can be easily analyzed by diffraction and microscopic/tomographic methods, bulk information of mesoscopic scale needs small-angle neutron scattering. In particular, \textit{in-situ} studies over a good volume average are necessary, which can profit from the penetration and the intensities of neutron beams.

Conventional pinhole SANS accessing size limits up to a few 1000 Å, in the form of Quokka, will already be available at the OPAL facility and will develop as one of the working horses for \textit{in-situ} studies.

Under discussion are Crystal-USANS or SESANS either of which can bridge the gap between the nanometer and sub-millimeter range.

4.1 General options

Figure 24 shows the layout of neutron scattering instruments at the OPAL facility.

Options for the next OPAL instrument are:

- **Instrument type**
  - Conventional Bonse-Hart Crystal-USANS
  - Bent Crystal-USANS
  - MSANS/VSANS
  - Spin-Echo SANS

- **Location (cf. Fig. 24)**
  - 1 - Reactor-face
  - 2 - Cold neutron guide
  - 3 - Thermal neutron guide

The expected neutron wavelength spectra can be found in Appendix I.
Figure 24 Neutron scattering instruments at OPAL facility and possible locations for next instrument:
1 - Reactor face, 2 - Cold neutron guide, 3 - Thermal neutron guide
4.2 Instrument Requirements
One critical aspect for the utility of a USANS instrument would be the acquisition time and sample environment, especially for in-situ studies.

Desired requirements for these kinds of studies include

- Object size range from overlapping pinhole SANS (up to 0.1µm) extending up to 50-100 µm
- Fast acquisition times in the minutes time range for in-situ studies
- Space, possibility and machine stability for heavy and bulky sample environment such as high temperature furnaces (1800 K) and load frames (500 kg weight)

4.2.1 Sample Environment
A consideration was made of the sample environments required for the USANS instrument. A guide to future use could be considered from the ancillaries currently being developed for use on SANS (as it is the complementary technique). However, here is a caveat in that some of the ancillaries may produce anisotropic signals, which may have an effect on the type of instrument being built. For SESANS e.g. it would be advantageous to rotate the field, not the sample environment.

One extra consideration is that some environments may not be conducive to the type of instrument, particularly with respect to the homogenous fields required for SESANS.

i) Sample environments currently unavailable and unplanned at OPAL that would be useful for USANS

- Shear cell capable of coping with high stress slurries (anisotropic scattering)
- A smaller sample changer e.g. 5 positions
- Gas mixing with high-precision control.
- (Multi-axial) tensile and compression stage materials, capable of coping with high T (anisotropic scattering)
- Polymer extruder (anisotropic scattering)
- Temperature gradients across a sample
- Pressure cells (40 MPa) - hydrostatic and head gas pressure
- Stopped-flow cell
- Active samples - the ability to handle radioactive materials (just a big Pb box)
- A possible vertical-field cryomagnet

ii) Sample environments in the pipeline at OPAL, but not yet available:

- ISRC furnace with SANS windows (approved), but have to provide inserts, such as high T insert.
- AC Impedance spectrometer
- High pressure cell
- Humidity cell
- CSIRO hydrothermal cell (250°C - 40 bar)

iv) Sample Environments that may be difficult for Crystal-USANS

- Rheometer can only be used in a radial configuration.
v) Sample Environments that may be difficult for SESANS
- Horizontal cryomagnet is definitely useless, as this has a huge stray field.
- Various environments may be restricted if they contain motors, or if they contain a lot of iron.
- These could include the rheometer or cryostat (although a pulsed tube cooler could be used), ISRC (contains a turbo pump, cables are also quite thick).

4.2.2 Performance
- As fast as possible, which may point to SESANS?
- USANS overlaps with SANS - This would be possible with changeable optics. Have the NIST and HMI set-ups that are interchangeable. Having both set-ups would get the best of both worlds. HMI works faster, but has lower resolution. NIST works slower, but has better resolution.
  ⇒ Combination of Bonse-Hart multi-bounce and bent crystals?
- SESANS size should overlap with SANS at the lower size range.
- Length scales 100nm - 20microns
- USANS background to be low enough at high Q to make sure to overlap with SANS is good.
- What affects the background in SESANS?
- SESANS: How do you control $z_{\text{min}}$ and $z_{\text{max}}$ for a given sample?
- 10% length resolution, but this will be controlled by choice of instrument. With USANS resolution is controlled by slit characteristics. Don't know how SESANS resolution is affected, but wouldn't like it worse than SANS.

4.2.3 Data acquisition and performance
One of the major problems is that new SESANS users are unfamiliar with the technique. The comfort factor is less than with the established Bonse-Hart USANS technique. All components should be under computer control, including SESANS magnets, if chosen.

- Software to be reduced to “standard form” at institute, with easily distributed, open source, software.
- The instrument to be controlled with a GUI based system, to make it accessible to new users.
- An active approach should be made for routine SESANS data analysis, as this is currently unfamiliar with the majority of our users.
5. A Comparison of Crystal-USANS & SESANS

5.1 Advantages and disadvantages

<table>
<thead>
<tr>
<th></th>
<th>Crystal-USANS</th>
<th>SESANS</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Advantages</strong></td>
<td>Low technical risk</td>
<td>High intensity</td>
</tr>
<tr>
<td></td>
<td>Good for weak scatterers</td>
<td>Short experiment times</td>
</tr>
<tr>
<td></td>
<td>Good for magnetic samples</td>
<td>Short times for whole scan (secs)</td>
</tr>
<tr>
<td></td>
<td>Reduction software available (NIST)</td>
<td>Wider Q-range</td>
</tr>
<tr>
<td></td>
<td>Potential for multiple set-ups with different resolutions</td>
<td>Direct space method (liquid structure factor much more easy to derive)</td>
</tr>
<tr>
<td></td>
<td>Insensitive to stray fields and bad neighbours</td>
<td>No de-smearing required</td>
</tr>
<tr>
<td></td>
<td>Crystals give sharp peaks</td>
<td>Tolerant of multiple scattering</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Greater potential for improvements</td>
</tr>
<tr>
<td><strong>Disadvantages</strong></td>
<td>Slower</td>
<td>No good for weak scatterers (&lt;3%; 10% is fine)</td>
</tr>
<tr>
<td></td>
<td>Data are slit-smeared</td>
<td>Experience base is lower</td>
</tr>
<tr>
<td></td>
<td>Space limitations for sample-environment</td>
<td>Sensitive to stray magnetic fields</td>
</tr>
<tr>
<td></td>
<td>Multiple scattering difficult for strong scatterers (esp. with cold neutrons)</td>
<td>Magnetic samples may be difficult</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Engineering is more complicated</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cannot buy components “off the shelf”</td>
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<td></td>
<td></td>
<td>Detector may not be able to handle main beam in the instrument</td>
</tr>
<tr>
<td></td>
<td></td>
<td>More complicated to operate</td>
</tr>
<tr>
<td></td>
<td></td>
<td>No direct overlap with pinhole-SANS</td>
</tr>
</tbody>
</table>

Table 3  Advantages and disadvantages of Crystal-USANS and SESANS

5.2 Crystal-USANS Considerations

- Fixed wavelength
- Cold and thermal USANS for same cost as SESANS
- Could you combine wavelengths from cold and thermal sources? (Mezei)
- Cheaper to build 2 instruments or a combined Bonse-Hart multi-bounce & bent crystals
- USANS location – advantage of cold guide (increased divergence, double wavelength, 4 x improvement as gain in upstream and downstream divergence BUT disadvantage for systems which already suffer from multiple scattering become worse for cold neutron
- Thermal order magnitude lower flux than cold guide (cannot multiplex BT5 beamline)
- Need dedicated and cannot use end guide position (because divergence too small)
- Or cold neutron guide (cheaper because shielding and shutter mechanism)
• Copy back end of existing BT5
• Consider 7 bounce with prisms (modification of BT5)
• 3 bounce and 7 bounce - consider mounting of crystal arrays
• Si (111) for long wavelength, perhaps Si (220) for v. long wavelength but ideally want to be at $\theta = 45^\circ - 4.4 \text{ Å}$ wavelength = ca. $\theta = 45^\circ$ and likely graphite premonochromator or bent Si (111) but riskier
• Or dedicated thermal beam port - because main detector blocks through beam - would need to insert hole into shielding and place instrument behind
  o Therefore cold guide-based USANS
  o Issue of multiple scattering for some users
  o But assists by factor of 3 for non-multiple scattered samples
  o Bent crystal single reflection to improve speed
  o A bent Crystal-USANS can be run in tandem with a triple-bounce Crystal-USANS. The asymmetric cut monochromator crystal on bent Crystal-USANS views the same beam off of premonochromator after transmitting through triple-bounce monochromator. - The triple-bounce monochromator only takes a small slice (a couple of arcsec) out of the beam coming off the premonochromator. The total beam divergence is about a degree. The transmission should be greater than 99%.
  o Run one in high flux mode and one in high resolution mode
  o Darwin width grows with wavelength; divergence gain with wavelength; 4.4 Å almost perfect compared to 2.4 Å (John Barker - $\lambda^3$ benefit - see his paper)
  o Be filter to remove $\lambda$ on 3 reflections from Si(111) no $\lambda/2$ (Debye-Waller factor weak 3rd order reflection anyway). Line-of-sight not an issue because premonochromated beam
  o Tall guides (50 mm x 200 mm) - could put premonochromator in CG3 or guide-cut post reflectometer - closer to reactor - fewer reflections and gain in divergence - locate on opposite side from CG2 towards cabin
  o Flexible location - guide cuts were done at NIST
  o Crystals - HMI - retired; but synchrotrons require crystals
  o For small samples - is it possible to compress w/ asymmetric bounce rather than slitting down?
  o $^3$He 5 x 1" diameter x 6" tall (2" beam at sample when focussed) - minimizing dead time corrections - 3 at back and 2 in front
  o bent crystal - 1D - need position sensitive detector
  o 1D detector USS30K w/ electronics
  o Crystals $50K$ / pair
  o Stages: Huber at HMI, Physical Instrument (PI) at NIST
  o Mirror + autocollimator, possibly

5.3 SESANS Considerations

• Magnets (w/shop need to build, electromagnets, take good care of horseshoes, extremely flat and parallel) - designed at Delft
• ISIS magnets built "in steel companies"
• Films - electrochemically deposited permalloy on silicon (30 micron thick for 2 Å neutrons); half thickness for 4 Å; for TOF need different coils and foils; NO, electrochemically deposited and 3 microns thick
• Wavelength determines distance in foil and determine precision required on film thickness (few hundred revolutions)
• Say 1% precision, 100 turns, precision ca. 10 microns in the bending of the film; (Wim can provide calculation)
• If variations in film thickness then can change tilt of foils and tune with adjustment coils
• How precise does foil thickness need to be (within single foil and between foils)?
• Control of magnetic field (continuously monitored), during measurement have to adjust especially with higher fields, heating generated field drifts - need active control and water cooling
• \( \pi/2 \) flippers
• Polariser (Gatchina)
• Analyser (Gatchina)
• Field stepper in middle of set-up
• Correction coils to correct for mis-setting of coils
• Larger distance between coils, greater precision in terms of polarization of beam but reduced flux
• Films advantageous over coils as can have a much finer angle
• Cold beamline with velocity selector and sets of different flipping foils of different frequencies so can change wavelength
• Helmholtz coils
• Mechanisms to facilitate interaction with Delft
• Instrument at least 8 m long
• Wavelength and resolution variation - bandpass filters - XENOCS and also at Saclay VSANS instrument (imaging facility at HMI uses double bounce graphite monochromator)
• Resolution influence in terms of wavelength resolution
• Bandpass filter means do not need to use an end of guide position
• Bigger beams, bigger magnets, bigger budget (currently 8 mm high, 18 mm wide beam dimension at Delft) - max. 30 mm high - therefore take small part of guide but could use focussing optics?
• At Delft, vertical direction limited by foils, horizontal direction limited by size of polarizer and analyzer, could optimize by rotating instrument by 90°
• Width 2.5 cm, 10 cm long - horse shoes wider - optimizer foil, polarizer and analyzer in single plane
• See J. Appl. Cryst. article on Saclay instrument (Desert)
• Reflectometer option if buy slits (but could do on USANS also)
• Bigger magnets (more homogeneous fields, better polarization) different sets of foils - to get to different wavelengths (too much scattering could use shorter wavelength / too little (magic number of 20-50%) use longer wavelength - wavelength variation can be achieved by using different foils)
6. Summary

The workshop participants came to the following conclusions:

- There is a strong scientific case with broad scientific application and a pre-existing user community in Australia for a USANS instrument at the OPAL reactor. Such instrument will extend the Q-range of the already existing SANS instrument Quokka by two orders of magnitude.

- There have been identified two options for extending into the USANS range:
  
  o **Classical Crystal-USANS method**
    This method uses perfect Si crystals in Bragg reflection as collimators. It is regarded a low risk and can handle weak scatterers.
  
  o **Spin-Echo SANS**
    This newer method uses precessions of a polarized beam of neutrons to encode the scattering angle to very high precision. Potentially, huge gains in performance can be achieved, especially for strong scatterers (which might include the majority of the science of interest to the present Australian community).

- Initial estimates are that either can be achieved for $2-3\text{M}$ including labor, and that both would ideally be located on a cold neutron guide.

- The final choice between these two options depends on both calculations of performance and experiments at leading facilities overseas in addition to a judgement about the relative importance and usage levels for weak and strong scattering samples.
APPENDIX A - Workshop Attendees and Interested Persons

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FAX: +61 (0)7 3365-4299  
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Phone: +61 (0)2 9717-3691  
FAX: +61 (0)2 9717-9260  
Email: robert.russell@ansto.gov.au

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FAX: +49 (0)30 8062-2523  
Email: strobl@hmi.de

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Phone: +61 (0)2 4275-7401  
FAX: +61 (0)2 4275-3489  
Email: jim.williams@bluescopesteel.com
A.2 Interested Persons Unable to Attend Workshop

**Burnet, Stephen**  
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Kwinana WA 6167  
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FAX: +61 (0)8 8302-3683  
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Email: andrzej.radlinski@ga.gov.au

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FAX: +39 (0)91 590015  
Email: triolo@unipa.it

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FAX: +61 (0)2 6125-4903  
Email: jww@rsc.anu.edu.au

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Australia  
Phone: +61 (0)2 4275-7436  
FAX:  
Email: paton.wilson@bluescopesteel.com
APPENDIX B - Workshop Attendees' Wish List & Survey

B.1 Wish List

<table>
<thead>
<tr>
<th>Research Facility</th>
<th>Areas of Interest</th>
<th>Size Range of Interest</th>
<th>Sample Environment</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSIRO</td>
<td>• Nucleation growth and ripening</td>
<td>Sample entire range and overlap size ranges (single nucleation to nanometers to microns)</td>
<td>• In-situ process and mineralization</td>
<td>• Dynamics: Fast, seconds if possible to control time scale</td>
</tr>
<tr>
<td></td>
<td>• Reversible reactions and irreversible reactions</td>
<td></td>
<td>• Small sample volumes, 1.0 ml</td>
<td>• High throughput</td>
</tr>
<tr>
<td></td>
<td>• Materials such as clathrate</td>
<td></td>
<td>• Temperature: -150°C to 150°C</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Self assembling systems, polymeric materials</td>
<td></td>
<td>• Pressure: 10-20 MPa</td>
<td></td>
</tr>
<tr>
<td>U. South Australia, Ian Wark</td>
<td>• Gels and polymers</td>
<td>Meso- to micro scale</td>
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<tr>
<td></td>
<td>• Organic and inorganic hybrids</td>
<td></td>
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<tr>
<td></td>
<td>• Colloids</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>• Mineral processing</td>
<td></td>
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<tr>
<td>BlueScope Steel</td>
<td>• Solution and precipitation behaviour</td>
<td></td>
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<tr>
<td></td>
<td>• Steels, Mn sulphides, microalloy carbonitrides</td>
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<td></td>
<td>• High mechanical stress and strains</td>
<td></td>
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<tr>
<td></td>
<td>• Transformation and crystallisation behaviour</td>
<td></td>
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<tr>
<td></td>
<td>• Pigmentations, particle counts and shapes, nanometers and microns</td>
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<tr>
<td></td>
<td>• Weathered products after exposure</td>
<td></td>
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<tr>
<td></td>
<td>• Polymer crosslinking interactions</td>
<td></td>
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<tr>
<td></td>
<td>• Metallic coating phase distributions</td>
<td></td>
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- Nanometers to microns
- High temperatures: 100°C - 1250°C
- Magnets required?
<table>
<thead>
<tr>
<th>Research Facility</th>
<th>Areas of Interest</th>
<th>Size Range of Interest</th>
<th>Sample Environment</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Curtin U.</td>
<td>• Geopolymers</td>
<td>5 nanometers to microns</td>
<td></td>
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<tr>
<td></td>
<td>• Carboaerogels, diffraction nature</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Clustering of nanoparticles</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Monash U.</td>
<td>• Biological systems</td>
<td></td>
<td>• Conducted under processing condition</td>
<td>All of the above</td>
</tr>
<tr>
<td></td>
<td>• Dairy</td>
<td></td>
<td>• Pressure: 200 MPa minimum</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Deep scattering concentrated systems</td>
<td></td>
<td>• Shear and heat</td>
<td></td>
</tr>
<tr>
<td>ANU Auckland</td>
<td>• Dairy</td>
<td></td>
<td>• Pressure: 400 MPa</td>
<td>• Ditto</td>
</tr>
<tr>
<td></td>
<td>• Complex fluids</td>
<td></td>
<td>• Shear and temperature control, viscous</td>
<td>• Fast throughout</td>
</tr>
<tr>
<td></td>
<td>• High internal phase emulsions</td>
<td></td>
<td>• short time scale for analysis</td>
<td></td>
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<tr>
<td></td>
<td>• Proteins</td>
<td></td>
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<tr>
<td></td>
<td>• Oil water surfactants</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>• Complex systems</td>
<td></td>
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</tr>
<tr>
<td>ANSTO</td>
<td>• Red blood cells</td>
<td></td>
<td>• Ability to make real time measurements</td>
<td>Mostly sufficient sample space and isolation from the delicate parts of the instrument to introduce new and interesting sample environments</td>
</tr>
<tr>
<td></td>
<td>• Wood</td>
<td></td>
<td>• Timescales of the order 10's of minutes</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Complex fluids</td>
<td></td>
<td>• Temperature control: 0-80°C</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Polymer Blends</td>
<td></td>
<td>• Couette</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Tensile deformation</td>
<td></td>
<td>• Shear</td>
<td></td>
</tr>
<tr>
<td>ANSTO</td>
<td>• Structural integrity</td>
<td>Low volume fractions</td>
<td>• Heating and cooling range</td>
<td>Ditto BlueScope</td>
</tr>
<tr>
<td></td>
<td>• Chromium ppt</td>
<td></td>
<td>• Temperature: 600°C to 700°C</td>
<td></td>
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<tr>
<td></td>
<td>• Voids creep or hydrogen build-up</td>
<td></td>
<td>• 1200°C</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Hydrides, zircoly (?)</td>
<td></td>
<td>• Gleeble instrument</td>
<td></td>
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<td></td>
<td>• Micro</td>
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<td></td>
<td>• P91</td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Nuclear materials</td>
<td></td>
<td></td>
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<tr>
<td>Research Facility</td>
<td>Areas of Interest</td>
<td>Size Range of Interest</td>
<td>Sample Environment</td>
<td>Other</td>
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<tr>
<td>-------------------</td>
<td>-------------------</td>
<td>------------------------</td>
<td>--------------------</td>
<td>-------</td>
</tr>
</tbody>
</table>
| UNSW              | • Cement paste   | Interconnected voids, up to 100 microns | • Humidity: 0-100 %  
• 10 min analysis  
• Sample changer with at least 20 sample holders  
• A way to correct for multiple scattering  
• Temperature: -10°C to 100°C, also 5 K | Everything |
|                   | • Clay            |                        |                    |       |
|                   | • Strong scatterers |                       |                    |       |
|                   |                   |                        |                    |       |
| UWS               | • Nucleation and growth in confined media  
• Inclusion in geochemical systems  
• Food systems |                        |                    | All of the above |
|                   |                   |                        |                    |       |
| BRAGG             | • Polymer phase separation with ability to follow kinetics  
• Complementary X-ray data  
• Light scattering data  
• SANS access same day?  
• Diffuse wave spectroscopy  
• Biological samples  
• Dilute solutions molar  
• Virus  
• Components of cells  
• Radiation damage  
• Ion exchange resins  
• Hydrogen storage  
• Explosives  
• Aerosol | • Time range: Seconds to minutes to hours  
• Small samples – 300 ul  
• Deuteration capability  
• Radioactive samples | • Software to correct from SANS to USANS  
• Reciprocal to real space? |
B.2 Survey

Real space size D of interest
What fraction of your experiments will study what size ranges?

![Real space size D chart]

Real space size D resolution
What fraction of your experiments need what resolutions in real space?

![Real space size D resolution chart]
Speed requirements
What fraction of your experiments require a full data set (say over a Q range of $10^{-3}$ to $10^{-5}$ Å$^{-1}$) to be collected in
APPENDIX C - Workshop Program

Program for Workshop on

"Pushing Small-Angle Neutron Scattering at OPAL to Smaller Q"

OPAL Conference Room, Building 83, ANSTO, New Illawarra Road, Menai, NSW 2234
15-16 November 2007

THURSDAY, 15 November 2007

<table>
<thead>
<tr>
<th>Time</th>
<th>Presentation</th>
<th>Presenter</th>
<th>Chair</th>
</tr>
</thead>
<tbody>
<tr>
<td>8:30</td>
<td>Arrival at ANSTO</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9:00</td>
<td>Opening and welcome</td>
<td>George Collins, ANSTO</td>
<td>Rob Robinson, ANSTO</td>
</tr>
<tr>
<td>9:05</td>
<td>Charge to the workshop</td>
<td>Rob Robinson, ANSTO</td>
<td>Craig Buckley, Curtin U.</td>
</tr>
<tr>
<td>9:10</td>
<td>Neutron scattering at OPAL</td>
<td>Shane Kennedy, ANSTO</td>
<td></td>
</tr>
<tr>
<td>9:25</td>
<td>Introduction to low-Q neutron scattering</td>
<td>Christine Rehm, ANSTO</td>
<td></td>
</tr>
<tr>
<td>9:55</td>
<td>Morning tea</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10:10</td>
<td>The science we can do at lower Q: Experience from NIST</td>
<td>John Barker, NIST</td>
<td>Greg Warr, U. of Sydney</td>
</tr>
<tr>
<td>11:10</td>
<td>The science we can do at lower Q: Experience from the V12 double crystal diffractometer at Hahn-Meitner Institute</td>
<td>Markus Strobl, HMI</td>
<td></td>
</tr>
<tr>
<td>12:10</td>
<td>Scientific and technical visions: 5 min. for each interested attendee (Contributions from biology, polymer, geology, materials science, industry)</td>
<td>Rob Robinson, ANSTO</td>
<td></td>
</tr>
<tr>
<td></td>
<td>USANS Experience and scientific needs</td>
<td>Craig Buckley, Curtin U.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Understanding and control of the self-organization in macromolecules and bio-macromolecules</td>
<td>Naba Dutta, U. of South Australia</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Kinetic studies of red blood cells</td>
<td>Chris Garvey, ANSTO</td>
<td></td>
</tr>
<tr>
<td></td>
<td>TBA</td>
<td>Lee Hoffman, Flinders U.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>The SAXS beam line at the Australian Synchrotron</td>
<td>Nigel Kirby, Australian Synchrotron</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Using USANS to probe the structure of polypeptide biomimetic microreactors</td>
<td>Jeremy Ruggles, U. of Queensland</td>
<td></td>
</tr>
<tr>
<td></td>
<td>More presentations to be added...</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13:00</td>
<td>Lunch</td>
<td></td>
<td></td>
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<tr>
<td>13:45</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>
13:45  The science we can do at larger R: Experience from Delft UT  
       Wim Bouwman, Delft  
       Patrick Hartley, CSIRO

14:45  Technical aspects of Bonse-Hart USANS as implemented at NIST  
       John Barker, NIST

15:15  Technical aspects of Bonse-Hart USANS as implemented at the V12 double crystal diffractometer at Hahn-Meitner Institute  
       Markus Strobl, HMI

15:45  Workshop photo

16:00  Afternoon tea

16:15  Technical aspects of Spin-Echo SANS as implemented at Delft UT  
       Wim Bouwman, Delft  
       John Bartlett, U. of Western Sydney

16:45  Discussion of scientific opportunities for "Pushing Small-Angle Neutron Scattering at OPAL to Smaller Q"  
       Christine Rehm, ANSTO

18:00  Move to dinner

18:30  Workshop dinner (venue see below)

---

**Thursday evening**

**Thai Peninsular Restaurant**

2 Prices Circuit  
Woronora, NSW 2232  
Ph: +61 (0)2 9545-4357  
http://www.thaipeninsular.com.au

- Exit bridge to left  
- Follow road underneath  
- Cross river again  
- Turn left into Prices Circuit
<table>
<thead>
<tr>
<th>Time</th>
<th>Presentation</th>
<th>Presenter</th>
<th>Chair</th>
</tr>
</thead>
<tbody>
<tr>
<td>9:00</td>
<td>Welcome back</td>
<td>Christine Rehm, ANSTO</td>
<td>Nigel Kirby, Australian Synchrotron</td>
</tr>
<tr>
<td>9:05</td>
<td>Characteristics of the neutron guide systems at OPAL</td>
<td>Shane Kennedy, ANSTO</td>
<td></td>
</tr>
<tr>
<td>9:25</td>
<td>Discussion of technical issues</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>What type of instrument should we build? What optics should we have? etc.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10:30</td>
<td>Coffee</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10:45</td>
<td>Definition of instrument parameters for &quot;Pushing Small-Angle Neutron Scattering at OPAL to Smaller Q&quot;</td>
<td>Christine Rehm, ANSTO</td>
<td></td>
</tr>
<tr>
<td>12:15</td>
<td>Charge for writing workshop report</td>
<td>Rob Robinson, ANSTO</td>
<td></td>
</tr>
<tr>
<td>12:30</td>
<td>Lunch</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13:30</td>
<td>Split into groups for report writing</td>
<td></td>
<td></td>
</tr>
<tr>
<td>16:30</td>
<td>Workshop summary and close</td>
<td>Christine Rehm, ANSTO</td>
<td></td>
</tr>
</tbody>
</table>
APPENDIX D - On Pinhole SANS

Shown below is the principle layout of a conventional pinhole SANS instrument which uses a narrowly collimated monochromatic neutron beam.

![Principle layout of pinhole SANS](image)

**Figure D1** Principle layout of pinhole SANS [25]

A velocity selector filters neutrons with a predetermined wavelength. In combination with a segmented neutron guide, the beam intensity can be enhanced with larger divergence. After passing two apertures (or: diaphragms) made of neutron-absorbing material (e.g. Cd), the beam irradiates the sample and the scattered neutrons are counted in a two-dimensional position-sensitive detector.

The resolution function \(<\delta Q>^2\) is described by

\[
<\delta Q>^2 = \frac{k^2}{12} \left[ \left( \frac{d_D}{L_D} \right)^2 + \left( \frac{d_E}{L_S} \right)^2 + d_s^2 \cdot \left( \frac{1}{L_S} + \frac{1}{L_D} \right)^2 + \Theta^2 \left( \frac{\delta\lambda}{<\lambda>} \right)^2 \right]
\]

where
- \(d_D\) (\(d_E\)): Diameter of first (second) aperture
- \(L_S\): Distance between the two apertures
- \(L_D\): Distance between sample and detector

For a given instrumental setting, neutrons can be detected in a limited angular interval; the setting is adjusted by the distance between sample and detector (between 1.25 m and 20 m) leading to possible neutron wavelengths between 5 and 15 Å to a Q interval of \(10^{-1}\) Å\(^{-1}\) to \(10^{-3}\) Å\(^{-1}\) (several overlapping runs).
Optimal conditions of such instrument are achieved with $L_S = L_D$ and $d_D = d_E$, i.e. if geometrical and wavelength contributions to the $Q$ resolution are matched with the resolution according to

$$\delta Q_{opt} \propto \frac{d_2}{L_D}$$

and the intensity at the sample position according to

$$\Delta I_0 \propto \left(\frac{\delta Q}{k}\right)^4 \cdot L_D^2$$

This shows that the intensity at the sample is proportional to the square of its length. That is why typical SANS instruments have lengths of a few ten meters.

Shown below is the SANS instrument Quokka built at OPAL's cold neutron guide CG1, as well as some instrument parameters.

**Figure D2** SANS Instrument Quokka [1]

- Wavelength range: $4.5 \, \text{Å} < \lambda < 43 \, \text{Å}$
- q-range: $8 \cdot 10^{-4} \, \text{Å}^{-1} < q < 1.0 \, \text{Å}^{-1}$
- Source to sample distance: 1, 2, 4, 6, 8, 10, 12, 14, 16, 20 m
- Sample to detector distance: 1-20 m
- Neutron guides: $^{58}\text{Ni}$-equivalent guides (Ni/Mo-Ti)
- Maximum beam cross-section: 50 mm x 50 mm
- Incident beam polarization: Fe/Si supermirror
- Detector area: 1m² with horizontal offset by up to 450 mm
- Optics: MgF₂ lens and prism focussing optics
- Large sample area (standard connectors for user-defined ancillaries)
- Provision for polarization analysis, further optics, chopper
APPENDIX E - On MSANS/VSANS

Purpose
Increase $Q$ resolution by at least one order of magnitude compared to SANS at equal intensity extending the measurement of correlations to a scale of several $\mu$m.

Principle
Same as for classical pinhole SANS but with very small apertures and a two-dimensional position-sensitive detector (spatial resolution 2-3 mm) to ensure measurements over a wide wavevector space.

More information about this technique can be obtained from the following neutron research facilities:

- HMI

![Figure E1](image.png)  
**Figure E1**  Multi-pinhole grid collimators for neutron selection, VSANS [26]
The following paper (to be published) will report on the first demonstration experiment of the MSANS technique at a 5.6 m long diffraction beam line, leading to a Q-resolution of $3 \times 10^{-4} \text{ Å}^{-1}$.

Multiple Small-Angle Neutron Scattering (MSANS): A New Two-Dimensional Ultra Small-Angle Neutron Scattering Technique
To be published

Figure E2 Overlay of multiple individual beams, MSANS [27]
• LLB
A detailed discussion of the design and characteristics of a "very small-angle neutron scattering" (VSANS) instrument at Laboratoire Léon Brillouin can be found in [28].

Figure E3 Scheme of the spectrometer TPA at LLB [28]

• NIST
The planned VSANS instrument which will cover the usual SANS range will also allow configuration to cover the range between $q \approx 10^4 \, \AA^{-1}$ to $10^3 \, \AA^{-1}$ with a sample beam current of $10^4$ neutrons/s.

Figure E4 VSANS instrument concepts considered at NIST [29]
4.2.4 TG3 Spectrum

The TG3 spectrum was measured at the end of the INVAP-supplied neutron guide. The disc chopper system was mounted ~200 mm from the guide end in the Kowari monochromator drum, behind a wall of thermal neutron shielding. Measurement parameters were as follows.

- TG3 TOF 17 December 2006
- OPAL power ~19 MW
- Path length (mm)3760
- Aperture 1 10 mm x 1 mm
- Chopper 8 slots (10 mm x 1 mm)
- Frequency (Hz) 176
- Filter none
- Aperture 2 none
- Detector 23Li
- Reduced data file: tg3tof17dec.dat

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<tr>
<th>Neutron counts</th>
<th>Al cut-off edge</th>
<th>Zr cut-off edge</th>
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<tr>
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</tr>
</tbody>
</table>

**Figure 4.2.4.1** Wavelength spectrum measured on TG3 (measurement parameters listed above). Overlays indicate Bragg cut-off for Aluminium metal and Zirconium metal.

Fast neutron background subtraction is not applied here for several reasons:

i. The detector used is low efficiency and therefore not sensitive to ambient radiation

ii. The measurements were taken ~5 metres beyond the line of sight position, so very few fast or epithermal neutrons were present
iii. The spectral range only extends to $\lambda \sim 5.73 \text{ Å}$, which is not long enough for the thermal spectrum to reduce to negligible contribution. This means that we were not able to use the long wavelength region to determine background level.

However, there is clearly a case to consider subtraction of the sharply deceasing background seen at short wavelengths. This may be created by errors in the $1/v$ correction applied to the raw data. Correction of the short wavelength region ($\lambda < 0.6 \text{ Å}$) was made by extrapolation of the spectrum between $\lambda = 0.6 \text{ Å}$ and $\lambda = 1 \text{ Å}$. The resultant spectrum is shown in Figure 4.2.4.2. This spectrum was used to determine the calibration factors for TG3.

![Graph showing neutron counts and background difference](image)

Figure 4.2.4.2  TG3 wavelength spectrum with background subtracted

Calibration factors extracted from TG3 data are

i. Peak wavelength = 1.33 Å

ii. Fraction of spectrum below energy of 100 meV = 0.909

iii. Mean wavelength = 1.67 Å
4.2.7 CG3 Spectrum

The CG3 spectrum was measured at the end of the INVAP-supplied neutron guide. The disc chopper system was mounted ~ 100 mm from the guide end, behind a wall of thermal neutron shielding. Measurement parameters were as follows:

CG3 TOF 24 June 2007
OPAL power ~19 MW
Path length (mm) 1978
Aperture 1 1 mm diameter
Chopper 2 slots (10 mm x 0.5 mm)
Frequency (Hz) 79
Filter none
Aperture 2 none
Detector 1He
Reduced data file: cg3tof25jun.dat

Background was low, indicating no significant leakage of neutrons – neither fast neutrons from the source, nor thermal neutrons circumventing the thermal neutron shielding. So no background corrections were required.

![Energy spectrum graph](image)

**Figure 4.2.7.1** Energy spectrum measured at the end of the guide on CG3 (measurement parameters listed above). Note that the energy scale is logarithmic and runs from 1 meV to 1 eV. Overlays indicate Bragg cut-off for Aluminium metal and Zirconium metal, up to energy of 40 meV.

**Calibration factors extracted from CG3 data**

i. Peak in energy spectrum = 3.05 meV.

ii. Fraction of energy spectrum below 10 meV = 0.617

iii. Mean wavelength = 3.15 Å
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Neutron Beam Instrument Project, ANSTO