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Neutron Scattering with Low and Medium Flux Neutron Sources

Processes, Detection and Applications

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NEUTRON SCATTERING WITH LOW AND MEDIUM FLUX NEUTRON SOURCES

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NEUTRON SCATTERING WITH LOW AND MEDIUM FLUX NEUTRON SOURCES

PROCESSES, DETECTION AND APPLICATIONS

INTERNATIONAL ATOMIC ENERGY AGENCY
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FOREWORD

Neutron beam techniques, such as neutron scattering, are used in scientific and technological research and development, in education and training programmes and for the provision of commercial services. Neutron scattering is used worldwide to generate new knowledge and undertake fundamental research, and it helps to shape the researchers, scientists and engineers that constitute the future workforce in nuclear sciences and technologies. Neutron scattering is applied in health and life sciences, for example, to provide information on viruses, proteins and degenerative diseases, and to assist in the development of new drugs and therapies. Neutron scattering contributes to the understanding of processes relevant to production, pollution, purification, and conservation of food and water. It plays an important role in studying new energy sources to protect the environment and combat climate change, including hydrogen storage, fuel cells, batteries and solar cells. It is applied to unveil crucial information in cultural heritage, archaeometry and forensics, with applications to the conservation and restoration, dating, and authentication of artefacts and objects, and the understanding of weathering phenomena and past technologies and fabrication methods. Neutron scattering allows the study of electronic and magnetic nanosystems, spintronic systems, novel superconductors, molecular electronics and other systems, with applications to the development of new information and communication technologies. It is used in many industrial and engineering applications, including metallurgy, cement and concretes, aerospace and automotive components, and nuclear materials.

There are currently 236 operational research reactors worldwide. Of these, 44 report activity in neutron scattering in the IAEA's Research Reactor Database. However, only 17 are low or medium flux reactors, including facilities with a power up to 6 MW, with nearly half of them having a power of less than 1 MW. To date, few non-spallation accelerator based neutron sources have been applied to neutron scattering. However, several accelerator based neutron sources are being developed as national neutron facilities. While leading edge research is preferentially conducted at high flux facilities, an opportunity remains to expand the application of neutron scattering with medium and low neutron fluxes. This can be to develop techniques and instrument components, to perform test experiments prior to experiments at high performance facilities, to educate students and to train technical and scientific staff, and to perform research and development in many areas where medium flux neutron sources can provide competitive conditions.

The objectives of this publication are to provide up to date technical information on neutron scattering techniques and on instrumentation for neutron scattering; to present the main applications of neutron scattering that could be the object of research and development or education and training at low and medium flux neutron sources; and to encourage and assist research reactor and accelerator based low and medium flux neutron source facilities in developing neutron scattering techniques. This is complemented by national experience reported in the supplementary files available on-line. The publication is expected to be of interest to neutron scattering experts, research reactor and accelerator personnel and those considering implementing research and development or education and training programmes based on this technique.

The IAEA acknowledges the valuable contributions and support of the international experts who contributed to the drafting and review of this publication, particularly J. Dawidowski (Argentina), G. Török (Hungary), I. Sumirat (Indonesia) and A.M. Venter (South Africa). The IAEA officers responsible for this publication were N. Pessoa Barradas and I. Swainson of the Division of Physical and Chemical Sciences.

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CONTENTS

1. INTRODUCTION

1.1. BACKGROUND

Neutron beam techniques, such as neutron scattering and neutron radiography, are used in scientific and technological research and development (R&D), in education and training (E&T) programmes [1] and for the provision of commercial services [2]. In R&D, neutron scattering is well established in materials science to study the structure and properties of materials, including their crystalline and magnetic structure, condensed matter physics and chemistry, nanotechnology, polymer science, life science, sustainable energy research, sensors and smart materials, biotechnology, spintronics, engineering and archaeology [1]. This widespread use is due to their suite of properties:

- At the wavelengths used in many neutron scattering techniques, the neutron energies are in the low meV range and thus non-destructive, which is very important in the study of biology, cultural heritage and industrial devices;
- The neutron–matter interaction occurs mainly with the nuclei of atoms. There are two principal interactions:
  - Nuclear scattering driven by the strong nuclear force. The amplitude of such interaction, and thus the coherent scattering length, *b*, varies widely and non-monotonically from one atom to another along the periodic table of elements, and as it is a nuclear interaction, it is also different for isotopes of the same element. This amplitude can be either positive or negative. In comparison to X ray scattering, where the scattering factors are proportional to the atomic number *Z* and are positive, this gives neutron scattering a strong discrimination capability, particularly between some neighbouring elements;
  - Magnetic scattering driven by interaction with the magnetic field of unpaired outer electrons. The strength of the interaction is comparable to that in nuclear scattering. This plays an important role in case of scattering on magnetic systems, such as unpaired electrons in specific molecules, domain walls, magnetic structures, magnetic nanoparticles and others [3, 4];
- Hydrogen (^{1}H) has a negative neutron scattering length while deuterium (^{2}H) (and most other elements, such as C, O and N, commonly present in organic molecules) has a positive scattering length. This can be effectively used to do isotope contrast matching by labelling some molecules with deuterium. This is a very important advantage over X ray techniques. It is also possible to continuously tune the scattering ability (i.e. scattering length density) of a solvent in a complex system by mixing hydrogenated and deuterated solvents in order to match the scattering of a component, making it invisible in the measured scattering curve;
- Neutrons interact weakly with many elements. This enables in situ experiments in various sample environments, in particular within devices made of metallic components (pressure cells, etc.).

There are currently 236 research reactors worldwide in operational or temporary shutdown status, according to the IAEA Research Reactor Database [5]. Of these, 44 in 28 countries report activity in neutron scattering, and 69 in 37 countries report activity in neutron radiography. Neutron radiography is reported in 40 research reactors with medium flux (between 1×10^{12} and 1×10^{14} n/cm^2/s peak neutron flux in the core [5]) and in four with low flux (below 1×10^{12} n/cm^2/s) and can be considered as a well established technique in these facilities. However, only 16 research reactors with medium flux and one with low flux report activity in neutron scattering. These reactors include facilities with a power up to 6 MW, and

nearly half of the facilities have a power of less than 1 MW. To date, only a few non-spallation accelerator based neutron sources have been dedicated to neutron scattering[1]. This number, however, is expected to increase in the future, as several new non-spallation accelerator based neutron sources are being developed, as national neutron facilities.

While leading edge research is preferentially conducted at high flux facilities, an opportunity remains to expand the application of neutron scattering in medium and low neutron flux facilities. This can be for demonstration purposes, as well as for utilization in physics education programmes, for training of research and technical staff prior to experiments at a large user facility, for the development of techniques and experiments, and to perform R&D in many areas where medium flux neutron sources can provide competitive conditions.

Over 25 years ago, the IAEA summarized the state of the art in terms of the use of neutron beams in low and medium flux research reactors for R&D programmes in materials science [6] and for radiography and materials characterization [7]. In the intervening years, there has been significant technical and methodological development.

Accordingly, the IAEA organized in 2019 a Technical Meeting on Neutron Scattering and Spectroscopy with Low and Medium Flux Neutron Sources, in order to share experiences, challenges and good practices in the design and utilization of neutron scattering and neutron spectroscopy techniques with low or medium flux research reactor and accelerator based neutron sources. That meeting brought together practitioners, users and other stakeholders interested in neutron beam scattering and neutron spectroscopy techniques from both research reactor and accelerator based neutron sources in the low and medium neutron flux range, and to promote exchange of information on existing experience, good practices, lessons learned and the challenges related to those techniques.

The participants of the Technical Meeting (May 2019) on "Neutron Scattering and Spectroscopy with Low and Medium Flux Neutron Sources" recommended that the IAEA prepare a draft document on “Neutron scattering with low and medium flux neutron sources”. The meeting participants agreed to be involved in the finalization of the document, contributing sections of chapters and individual country reports. A draft structure of the document and participant involvement was proposed. In the following months, contributions were received and collected. This was followed by a Consultancy Meeting (November 2019) on the "Development of a Publication on Neutron Scattering with Low and Medium Flux Neutron Sources" to review the status of the drafting of the publication, identify gaps in its structure and contents and establish an action plan for its finalization. This resulted in a first complete draft, which was subsequently reviewed by 10 experts not previously involved. The ensuing revision led to a second draft, which was discussed and finalized in a virtual Consultancy Meeting (October 2020) that brought together original drafters and reviewers.

1.2. OBJECTIVE

The objectives of this publication are:

- To provide up to date technical information on neutron scattering techniques and on instrumentation for neutron scattering;

[1] Spallation neutron sources require typically a proton beam with energy above 120 MeV, often in the GeV range. Non-spallation accelerator based sources function with lower ion or electron energies.

- To present the main applications of neutron scattering that could be the object of R&D or E&T at low and medium flux neutron sources;
- To encourage and assist research reactor and accelerator based low and medium flux neutron source facilities in developing and expanding their neutron scattering techniques and capabilities.

It is intended to serve as an authoritative reference on the application of neutron scattering at low and medium flux neutron sources, of interest to the neutron scattering experts, users, research reactor and accelerator personnel and those considering implementing R&D or E&T programmes based on this technique.

1.3. SCOPE

State of the art neutron scattering instruments are usually found at high flux neutron sources. These are high performance research reactors and accelerators, that often function as user facilities with many instruments dedicated to specific lines of research. Correspondingly, many of the applications and techniques mentioned in this publication are most often performed at high flux research reactors and accelerator based neutron sources.

Nevertheless, opportunities remain for low and medium flux sources to find relevant niches of application, in particular when specialized sample environments are available. Low and medium flux neutron sources play an important role in performing test experiments for large facilities, to educate students and train technical and scientific staff, and in the development of instrument components. Testing new ideas in the development of instrumentation is a cost effective method before transferring new methods to the large user facilities. In some cases, even demanding applications and techniques, including inelastic neutron scattering, can be competitively installed at well designed, medium flux facilities, albeit not necessarily in low flux facilities. Therefore, this publication covers nearly all neutron scattering techniques. The main exceptions are high energy resolution backscattering and neutron spin echo spectroscopy, which are inelastic neutron scattering techniques that only exist in a limited number of high performance neutron sources.

Each technique is covered in a dedicated section, where the purpose and main applications of each technique are stated. A summary of the main requirements of the technique is given, including the minimum and recommended neutron flux needed, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique, and needed staff. A brief description of the typical instruments used in the technique is given, as well as of sample requirements and data formats and analysis procedures.

The chapter on instrumentation covers all main components and systems used in neutron scattering. Emphasis is given to those more commonly found in instruments installed at low and medium flux neutron sources. The chapter includes a section on instrument optimization, which provides a brief overview of methods intended to optimize the performance of neutron scattering instruments at low and medium flux neutron sources.

The chapter on applications of neutron scattering concentrates on the main applications that could be the object of R&D or E&T at low and medium flux neutron sources. The main purpose of each area of application is summarized. Then, the techniques commonly employed for the application are described, with emphasis on the information that can be gained with each technique. Some of the techniques discussed require a high flux neutron source and state

of the art instruments if the objective is to conduct leading edge R&D in all possible areas. Nevertheless, they can be implemented at lower flux neutron sources for R&D in specific classes of materials. For such cases, the scope for low and medium neutron sources is stated. Each section concludes with a description of the classes of users typically interested in the application.

Finally, this publication is dedicated to neutron scattering only. Other neutron beam techniques such as neutron imaging fall outside this scope. The IAEA has publications on the latter subject [7, 8], and recently developed an e-learning course on neutron imaging [9]. Analytical techniques such as neutron activation analysis (NAA) and prompt gamma analysis (PGA) are among the most important applications of low and medium flux research reactors and are also covered elsewhere [10–12].

1.4. STRUCTURE

The present report consists of this introduction followed by three technical sections describing the main neutron scattering techniques, instrumentation for neutron scattering and applications of neutron scattering, a list of references, and a list of individual paper contributors, together with their affiliations and individual paper titles. The emphasis of the technical sections is on those elements of neutron scattering most adequate for implementation at low and medium flux research reactor and accelerator based neutron sources. In addition, an Annex listing the individual paper contributions has been added to this manuscript. The individual reports are available on the on-line supplementary material to this publication.

2. NEUTRON SCATTERING TECHNIQUES

This chapter presents neutron scattering techniques that could be implemented at low and medium flux neutron sources. Some of the techniques discussed require a high performance neutron source to reach their full potential and capabilities in R&D and provision of services, but nevertheless can be implemented at lower flux neutron sources for demonstration, E&T purposes, to develop experiments, as well as perform preliminary experiments before implementation at high flux sources. Neutron spin echo spectroscopy is not included in this category, as it is considered not to be appropriately implemented using low or medium neutron fluxes.

Each technique is the object of a dedicated section, following a common structure. The purpose of the technique is first stated, mentioning the most common and important applications, and also the minimum requirements for the implementation of a typical set-up. It should be noted that the time required to develop and install each technique and the cost of installation are for typical setups and are indicative only. They can vary widely and be much higher depending on which additional optical components and ancillary equipment, including development of particular sample environments, are considered. The given time periods assume experienced labs with access to significant human, budget and technical resources, facing only technical constraints. Even then, delays are common, for instance in procurement of materials and components or in obtaining regulatory licenses, and less experienced labs may require a considerable amount of time to acquire the necessary knowledge and expertise. Also, accounting practices and cost of materials vary widely from country to country, and the costs mentioned do not include salaries.

In practice, the duration and cost of some projects may far exceed the numbers given here. Likewise, the given human resources needed for operation represents the minimum required for operation. Full time user service, provided under an established user programme, requires more human resources, up to four (or more) staff per instrument [1]. As users often are not experts in the experimental techniques and in data analysis, extensive support needs to be provided in both areas to attract and ensure that more scientists use the opportunities offered by neutron scattering. In that case, additional human resources are needed. Additionally, user facilities often have a software group as well as staff specialized in sample environments that assist the different instruments and user groups.

The description of these minimum requirements is followed by a brief description of a typical instrument, commonly associated equipment, and of the fundamentals of the technique. Then any requirements for the samples that can be analysed are listed, including phase, dimensions, and non-ambient conditions that can be employed such as temperature, pressure and atmosphere. Each section concludes with a discussion of data analysis methods for the given technique.

As mentioned above, the minimum requirements and costs given in this publication do not include additional equipment, such as that dedicated to generating non-ambient sample environments, e.g. for control of vacuum, temperature, pressure, humidity, magnetic field and other variables. We cannot, however, overemphasize the importance of such capabilities to enhance the competitiveness of neutron scattering in medium neutron flux sources. Indeed, specialized sample environments are often a main condition for success of an instrument.

2.1. POWDER DIFFRACTION

2.1.1. Purpose

Neutron powder diffraction (NPD) is a unique method to refine and, in some cases, contribute to the determination of the structure of materials, including magnetic order, along with other structural properties. It is the simplest and most widely used neutron scattering technique. Low and medium fluxes are very often sufficient for standard use. The nomenclature powder diffraction is commonly used, but nevertheless the experimental technique can be applied to the study of polycrystalline materials that can be in powder or compact solid form. Similar instruments, with lower resolution, can be applied to liquids that have short range order [6].

The most common applications of NPD include Rietveld analysis to refine crystal and magnetic structure, phase transition studies, multiphase materials, stress measurements, and E&T. Further possible research fields that can be addressed with this technique are functional materials, spintronics, hydrogen storage materials, high temperature superconductors, residual stress and texture in alloys, amorphous systems, order–disorder phase transitions and phase transitions in metal compounds. In situ and operando analysis is also possible. Possible industrial applications include the study of composite materials, catalytic agents, residual stress, creep and fatigue, materials in extreme conditions of temperature, pressure and/or magnetic field, concrete and cement, battery electrodes and geological samples.

2.1.2. Requirements

NPD usually employs thermal or cold neutrons. Table 1 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate a neutron powder diffraction instrument. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments. High and medium resolution is considered to correspond to $\Delta d/d < 10^{-4}$ and $\Delta d/d < 10^{-3}$, respectively.

2.1.3. Instrument description

There are two main types of diffractometers, according to their mode of operation: constant wavelength (CW) [13] and time of flight (TOF) [14]. The first one can be installed at research reactors and at steady state (not pulsed) accelerator based neutrons sources, whereas the latter methodology can be applied at both reactors and pulsed accelerator sources.

2.1.3.1. Constant wavelength diffractometers

In CW diffractometers a monochromator device consisting of one or more single crystals selects and directs a beam of specific wavelength from the neutron spectrum. This is achieved from constructive interference of the neutron beam from specific atomic crystal planes in the monochromator. The acquired neutron wavelength λ is related with the crystal plane spacing d and reflected beam direction 2θ (relative to the incident beam), by the Bragg equation:

$$\lambda = 2d \sin \theta \quad (1)$$

Thus, by appropriate crystal plane and beam direction selection, a monochromatic beam is selectively extracted with a narrow wavelength band to the sample position. In order to achieve higher flux at the sample position, vertical focusing of the monochromator can be made by appropriate vertical alignment of segmented inclined crystals and/or by changing the horizontally focusing geometry (see section 3.2.5). The monochromator materials most widely used are Si, Ge, Cu and Highly Oriented Pyrolytic Graphite (HOPG). The monochromator material and geometry should be chosen carefully to match other diffractometer components. The general features of a CW diffractometer are displayed in Fig. 1.

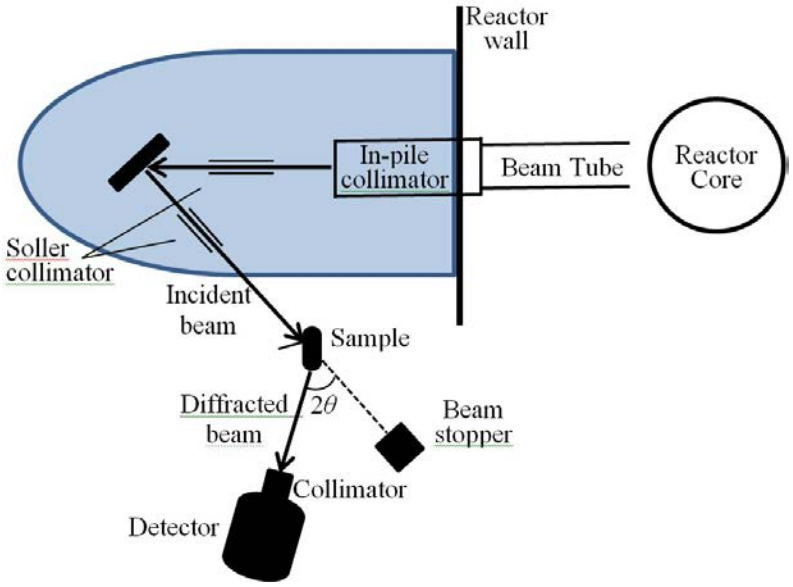


FIG. 1. Layout of the constant wavelength type neutron powder diffractometer (courtesy of Pakistan Institute of Nuclear Science and Technology, Pakistan).

TABLE 1. REQUIREMENTS FOR A TYPICAL NEUTRON POWDER DIFFRACTOMETER

Instrument	Powder diffractometer
Neutron beam spectrum	Thermal or cold
Minimum neutron flux (n/cm^2/s)	10^7 at beam port exit 10^4 at sample
Recommended neutron flux (n/cm^2/s)	$\geq 10^8$ at beam port exit $\geq 10^5$ at sample
Beam size (cm^2)	10 (typically 5 cm height, 2 cm width)
Required space (m × m)	3 × 3
Minimum time to develop/install technique	2–4 years
Minimum cost of installation (million USD)	0.5–2 (depends on detection system and on CW/TOF options)
Human resources needed for operation	1–2 instrument scientists 0.5 technician. Technicians can attend more than one instrument Additional instrument scientists and/or software engineer for support to users

2.1.3.2. Time of flight diffractometers

A TOF diffractometer simultaneously utilises all the available neutron wavelengths in the incident beam. In this geometry, the detector angle is fixed with the wavelength being the variable parameter. Since each distinct wavelength corresponds to a specific energy and thereby a velocity, the TOF method measures the flight times of the neutrons from the source to the detector. Data collection by the detector is made with electronic systems that have the capability to encode the flight times of the incoming neutrons in each pulse with reference to a starting trigger. Typically, the minimum time resolution needed is about 1 microsecond per channel when fast neutrons are used. For thermal neutrons 10 microsecond time resolution is sufficient.

Therefore, in order to apply this technique, discrete bursts of neutrons are required to enter the neutron powder diffractometer. At steady state sources, bunches of neutrons are produced with a beam chopper, whereas at pulsed sources the neutron beam is naturally generated in bunches. The neutron wavelength depends on the flight path L (see Fig. 2) and time t measured by an electronic device. These relations can be expressed as:

$$\lambda = ht/m\,L \tag{2}$$

where h is Planck's constant and m is the neutron mass. This gives a modified version of Bragg's law in which

$$d = ht/(2m\,L\,\sin\theta) \tag{3}$$

For a given detector situated at angle θ, there is a simple linear relationship between t and d.

The actual design of a diffractometer depends on the neutron source and the magnitude of the available flux. Additional equipment and devices required for NPD instruments in the two modes of operation are provided in Table 2, as a rough guide.

The main difference in equipment at steady state reactor is that CW diffractometers employ a monochromator crystal device, while TOF diffractometers use a system of choppers (see section 3.2.7), which in practice makes the TOF systems slightly more expensive. The latter, however, enables higher instrumental resolution (sharper Bragg peaks). The detector system of CW diffractometers must cover an angular range. This is achievable with horizontal ^{3}He or BF_3 position sensitive detector tubes, multiwire or microstrip detectors. These can additionally be configured into pseudo area detectors by stacking units vertically to increase the area coverage. In the TOF case, a single detector can measure the time spectra at a chosen angle according to the desired Q range. In practice, 2D detectors or groups of detectors are employed to improve the data capturing efficiency.

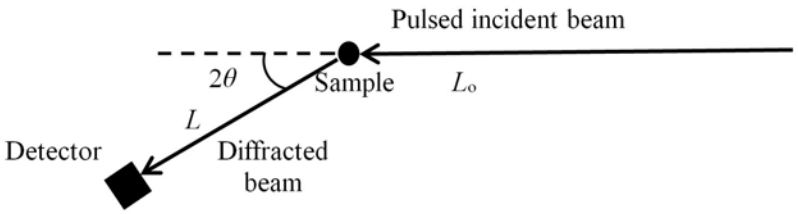


FIG. 2. Schematic view of a TOF diffractometer. The pulsed incident beam comes either from a pulsed source or from choppers as discussed in chapter 3 (courtesy of Pakistan Institute of Nuclear Science and Technology, Pakistan).

TABLE 2. ADDITIONAL EQUIPMENT TO BE CONSIDERED FOR NEUTRON POWDER DIFFRACTION INSTRUMENTS

Equipment and devices	
Constant wavelength	**Time of flight**
Monochromator	Choppers
1D or 2D detector system	1D or 2D detector system (usually 2D)
Radial collimators	Radial collimators
Beam flux monitor	
Soller collimators before and after monochromator	
Sample table	
Sample environment	
Filter for fast neutron	
Filter to remove second order harmonics	

One variant of a TOF spectrometer, less commonly used, is the Fourier chopper diffractometer. This is a high resolution diffractometer used in long pulse neutron sources [15].

With the use of an optimised focused monochromator, a greater neutron flux can be delivered at the sample position and still achieve medium resolution capability. A coarse radial collimator between the sample and the detector is used to reduce the background level in the detector. The highest resolution is achieved with the implementation of very tight Soller collimators (parallel beam) in both the incident and diffracted beam paths, at the expense of reduced transmission. Filters of unwanted fast neutrons in the primary beam, usually sapphire or silicon monocrystals of 10 to 15 cm length, must be employed to reduce the background in both CW and TOF cases (section 3.2.3).

For both CW and TOF diffractometers, there is an advantage in providing detectors to measure at high 2θ, as this highest Q resolution (precision in measuring lattice parameters) comes from measuring diffraction at high θ, as differentiating eq. (2) yields $\Delta d/d = -\cot\theta\Delta\theta$, and $\cot\theta \to 0$ as $\theta \to 90°$. The resolution between overlapping peaks can be increased in TOF diffractometers by reducing $\Delta t/t$ ($\Delta d/d$) which usually involves increasing the length L of the diffractometer (Eq. 1), although this is gained at the expense of count rate.

2.1.4. Sample requirements

The ideal samples for NPD are fine powders (grain sizes larger than 10 microns) with no preferred orientation or morphology. The diameter of the grains of polycrystalline aggregate should be about two orders of magnitude smaller than the probed dimensions of the sample volume, which is geometrically defined by optical devices such as slits and collimators. Polycrystalline solid samples can be investigated to obtain representative diffraction patterns, but results may be influenced by phenomena such as particle size, morphology, preferred orientation, degree of agglomeration and strain. Investigations of amorphous, liquid and gas phase samples are possible, though not very often performed [16, 17].

Compared to conventional X ray beam intensities, neutron beams are considerably weaker, which is compensated for by using larger sample volumes, up to several cubic centimetres.

However, it should be noted that large diameter samples increase the width of the diffracted beam and may increase the background levels due to multiple scattering. On the positive side, they decrease the sensitivity of neutron powder diffraction measurements to preferred orientation, which is a common issue with X rays.

Powder samples are easily contained in thin walled vanadium cylinders and setup for ambient condition diffraction experiments. Vanadium in addition is a good choice as a sample container for experiments under vacuum and temperatures from the mK range up to 1200 K. ZrTi alloys that render null coherent scattering length, or SiO_2 glass, can be used as sample holders for investigations in air above 500 K. It is possible to make relatively inexpensive cylinders from thin vanadium plate using laser welding or Ni arc welding. In the simplest case an aluminium foil can be used as a sample holder, because it does not have incoherent scattering and causes well known, easy to identify, peaks with relatively small intensity due to the small amount of material added to the sample.

Studies at non-ambient conditions are extensively performed. In these cases, the addition of an environmental chamber to the sample generates parasitic contributions to the diffraction signal, and special care is required to reduce them. These parasitic contributions are minimised by masking unwanted scattering contributors with neutron absorbing materials such as cadmium sheets and foils, B_4C sheet, borated polymers or gadolinium paint, or minimised with the use of radial collimators between the sample and the detectors.

Compact devices such as high pressure cells limit the maximum possible sample size to a few cubic millimetres. This subsequently leads to low scattered intensity conditions and low signal to noise ratios. The Paris-Edinburgh cell enables pressure experiments with neutrons up to 9 GPa and beyond [18, 19].

2.1.5. Data

The raw data measured with CW diffractometers, either equipped with single or position sensitive detectors, produce a diffraction pattern as function of 2θ which is analysed in the same fashion as conventional X ray diffraction (XRD) data. To improve the instrument efficiency, most instruments are equipped with multiple detectors to increase the counting rate and sample throughput. Data from such arrangements need to be appropriately treated and calibrated for efficiency (along detector length and between individual detectors), normalisation of angular values, etc. Efficiency corrections are done by measuring the scattered intensity from an isotropic scattering material such as vanadium or non-crystalline plastic (e.g. polyethylene, poly(methyl methacrylate), etc) over the full detector view. Normalisation is done by measuring with each detector the diffraction pattern from a standard sample that has a large number of diffraction peaks. One detector is chosen as a reference against which the diffraction peak intensities and positions of the other detectors are normalised. Measured diffraction patterns are converted to d spacing by using the exactly calibrated neutron wavelength.

In TOF instruments, diffracted and background intensities vary extensively across the diffraction pattern due to the incident neutron spectrum wavelength dependence. Standard samples of well known d spacing are used to calculate the θ value of each detector. Diffraction data of all the detectors are combined, corrected for efficiency (as for CW diffractometers) and converted from TOF to d spacing (Eq. (2)).

A variety of computer codes and fitting packages are available, including a number of open source or free packages. A non-exhaustive list includes FULLPROF [20, 21], GSAS [22], DWBS [23, 24]. Note that, for analysis of TOF diffraction data, it is important to take into account the neutron pulse time shape. This is particularly true for small and medium flux sources, where the moderator design tends to be based on the optimization of total flux instead of pulse shape. Hence, the profile functions available in the different software packages should be considered carefully.

2.2. SINGLE CRYSTAL DIFFRACTION

2.2.1. Purpose

Single Crystal Neutron Diffraction (SCND) [25, 26] is applied to study single crystals to provide information on their crystal and magnetic structures [27], as well as their behaviour as a function of temperature, pressure and electric and magnetic fields [28]. It provides the same kind of information as powder diffraction on the crystal structure from the atomic arrangement that includes lattice parameters, space group, atomic positions, bond angles, mean square displacement, lattice site occupancies, etc., as well as equivalent information on the magnetic structure (when present). However, the data from SCND are superior as the intensities from individual reflections are typically individually resolvable. This technique is capable of determining the localization of light elements and studying light element bonding, such as hydrogen in metals and other structures (using deuterium substitution).

Two approaches are possible, i.e. using monochromatic radiation (angular dispersive) or white beam (quasi Laue or TOF) modes (see section 2.1.3). The aim is to cover the full reciprocal space. The TOF mode is not commonly used in low or medium flux neutron sources. The other approaches are commonly found at medium flux neutron sources. With monochromatic radiation, single crystal diffraction can also be done at low flux sources if large crystals and Laue techniques (white beam) are implemented. Even then, only a limited number of lines is measured and counting times are long.

2.2.2. Requirements

Table 3 shows the minimum and recommended neutron flux, the time and cost typically needed to develop and install the technique and the required staff to operate a single crystal diffraction instrument. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments.

2.2.3. Instrument description

Schematic representations of typical instrumental layouts are shown in Fig. 3. The procedure of measurements can be summarized as follows: a single crystal under investigation is placed in a neutron beam; the intensities measured at different sample orientations (spatial conditions) are analysed to determine the crystal structure or the magnetic moment structure of the material.

TABLE 3. REQUIREMENTS FOR A TYPICAL SINGLE CRYSTAL NEUTRON DIFFRACTOMETER

Instrument	Single crystal diffractometer
Neutron beam spectrum	Thermal
Minimum neutron flux (n/cm^2/s)	10^7 at beam port exit 10^4 at sample
Recommended neutron flux (n/cm^2/s)	$\geq 10^9$ at beam port exit $\geq 10^6$ at sample
Minimum time to develop/install technique	2–4 years
Minimum cost of installation (million USD)	1–2.5
Human resources needed for operation	2 instrument scientists 1 technician Additional instrument scientists and/or software engineer for support to users

Fig. 3a represents a schematic top view of a typical 4-circle SCND instrument as used with monochromatic neutrons. A white neutron beam coming from the reactor is first monochromatized by a single crystal. This monochromatic beam is diffracted by the sample towards a detector. The beam stop is set to catch the neutrons that are not diffracted and pass through the sample. Typically, the detector records the neutron intensity vs. spatial position of the sample. For a single crystal investigation, a special form of goniometer called a four circle goniometer is usually applied. Fig. 3b shows a layout of the four circle goniometer that has four independent axes, i.e. θ, 2θ, χ and φ. The combination of the angles corresponds to specific positions in the reciprocal space of the sample. Fig. 3c is an example of a schematic layout of a real single crystal facility, installed at the Neutron Scattering Laboratory at BATAN, Indonesia, using a half four circle goniometer. A typical TOF single crystal diffractometer instrument setup is schematically shown in Fig. 3d. In this case, the angle between the incident beam and the detector centre is 90º; the distance between the sample and the detector is around 30 cm; the detector area is around 30×30 cm^2, with around 35 crystal settings to cover a full hemisphere of reciprocal space; and the distance between the sample and the moderator is nearly 10 m, which enables good resolution in q space [22].

Normal beam geometry is employed when the crystal can only rotate around an axis, for instance in the presence of an applied magnetic field.

Table 4 summarises the technical aspects of typical single crystal neutron diffraction instruments [25–32].

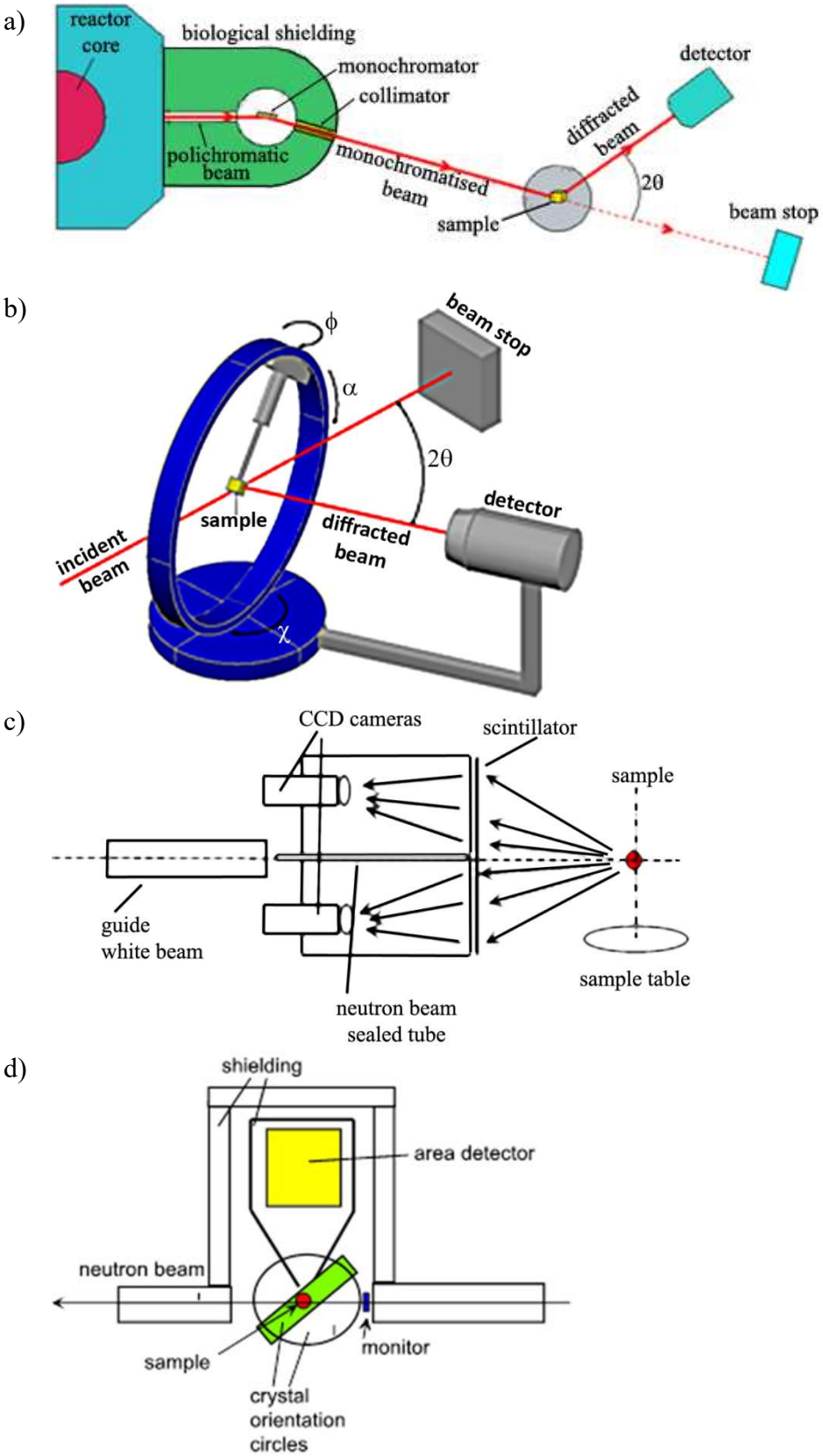


FIG. 3. (a) A schematic top view of a typical 4-circle Single Crystal Neutron Diffractometer as used with monochromatic neutrons; (b) Detail view of a typical 4-circle goniometer; (c) Schematic of a quasi Laue instrument located at the end of a beam line; (d) Schematic of a neutron TOF instrument (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary, and of BATAN, Indonesia).

TABLE 4. TYPICAL INSTRUMENTAL PARAMETERS

Beam spectrum	Constant wavelength (4-circle)	White beam (quasi Laue)	TOF
Space required	2 × 2 m^2 for the instrument 5 × 5 m^2 including the monochromator chamber	3 × 3 m^2. Instrument can be located at end of a beamline	Minimum 3–5 m depending on the pulse width
Monochromator	Bent Silicon (Si); Ge; HOPG; Cu	n/a	n/a
Incident wavelength	1.0–2.5 Å		
Crystal size requirement	1 mm^3	> 0.1 mm^3	1 mm^3
Maximum crystal size dimension	5 mm to minimize extinction effects		
Detector	BF_3, ^3He, scintillation detector, image plate In most cases single detector is adequate for CW; 2D detector provides higher data collection efficiency		
Resolution ($\Delta d/d$)	10^{-2}		

2.2.4. Sample requirements

Samples need to be high quality solid single crystals free of stacking faults and twinning (verified upfront using a polarized microscope), usually with a minimum volume of 1 mm^3 [25, 27, 31, 32] and maximum length 5 mm to limit extinction and absorption effects (corresponding to a maximum 125 mm^3 volume). The full sample volume needs to be illuminated with the neutron beam. The relative ease of making powder samples means that NPD is more prevalent than SCND. Investigations can be performed in extreme conditions such as high pressure, low and high temperatures, electric and magnetic fields with typical ranges:

- Temperature: 10–1000 K
- Magnetic field: 0–2 T
- Pressure: up to 2 GPa
- Electric field: Power supply up to 4000 V on sample

The values given above meet the requests for most experiments. In some cases, more extreme environments are implemented.

2.2.5. Data

The measured intensities are usually the object of integration and data reduction. For instance, in white beam and time-of-flight geometries it is very important to normalize the measured intensities to the incoming spectrum. A variety of computer codes are available for the processing of integrated intensities versus sample orientation, that include a number of open source or free packages, such as those given in references [33–36].

2.3. RESIDUAL STRESS AND TEXTURE

2.3.1. Purpose

Neutron residual stress measurement is a special case of neutron powder diffraction that is specifically aimed at the accurate non-destructive measurement of diffraction peak positions from which the interatomic spacings, strains and stresses are sequentially calculated [37–41]. The positions of the atoms with respect to the basic crystalline structure can also be determined with spatial resolution up to sub millimetre precision. In conjunction with directional specific component measurement (along the diffraction scattering vector), this enables depth dependent triaxial stress determination inside components.

Residual or applied stresses displace atoms from their equilibrium positions in a crystalline material in direction and position dependent ways. The resulting change of lattice parameters is quantified in terms of the elastic strains from which the triaxial stresses are calculated by applying Hook's law with the incorporation of the elastic properties of the material.

Depth dependent measurements provide information about the strain and stress conditions that exist within the engineered components and structures. Due to the non-destructive nature of the technique, investigations can be performed on components in their as produced condition, as well as after in service conditions. The following are the most common applications of this technique:

- Strain and stress due to mechanical, thermal and phase processing determined directly from the microstructure;
- Stresses in welded components (metal arc, electron beam, laser beam, friction stir welding, explosive joining, etc.);
- Influence of structure changes, phase transitions, phase specific stresses in multiphase materials;
- Coated systems (thermal, cold, laser deposition processes, etc.) with special interest on average in-plane stresses in coatings, interfaces and depth resolved in substrates;
- Thermomechanical testing of newly developed materials to study deformation and transformation mechanisms, i.e. elastic–plastic behaviour of polycrystalline components under conditions of applied external load (tension, compression, torsion, temperature, etc.);
- Investigate resulting structure changes and stress fields in components after irradiation (radiation damage);
- E&T.

Texture refers to the distribution of crystallographic orientations from a random distribution in a polycrystalline material [42]. The resultant anisotropy is an intrinsic feature that influences many physical properties during natural and technological processing of metals, ceramics, polymers, rocks, etc.

By exploiting the penetrating capability of thermal neutrons, bulk texture information averaged over large volumes is obtained to complement localised and microtextural features obtained from complementary X ray and electron based techniques. Texture is qualitatively visualised as pole figures and quantitatively analysed as orientation distribution functions.

The following are general applications of this technique:

- Microstructural features influenced by recrystallization, grain growth and annealing phenomena due to the thermomechanical processing of materials and components.
- Deformation texture associated with mechanical processing and crystal plasticity that results from cold working, forming (including e.g. rolling, forging and extrusion), pressure, etc. A practical real world example is the deep cup drawing processes in the formation of aluminium beverage cans;
- Natural geological deformation of rocks and minerals;
- Archaeological and cultural heritage artefacts, museum objects and archaeological findings. The non-destructive nature of the investigations enables searching for hidden materials and structures. Cultural heritage topics of interest are the production of historic weapons such as swords, manufacture of coins and others;

This technique is commonly found at medium flux neutron sources where applications probe very small volumes, typically < 20 mm^3, to high spatial resolution. Low neutron flux sources, though not commonly used for residual stress measurements, may be utilised for studies where probed volumes are larger than 30 mm^3 in conjunction with advanced focusing neutron optics for the investigation of high coherent scattering materials. Low neutron flux sources are more commonly used for texture investigations, in particular if large samples are available.

2.3.2. Requirements

As the strains in materials are usually of the order of 10^{-4}, a sufficiently high resolution in $\Delta d/d$ (usually < 10^{-4}) of the instrument is required using thermal or cold neutrons. The preferred scattering geometry is approximately 90° (2θ), where the investigated gauge volume has a symmetrical rectangular form.

Table 5 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate a residual stress diffraction instrument. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments.

Texture investigations are performed with a similar instrumental geometry as for residual stress measurements, but the strict gauge volume definition and high resolution are not required are not required. Thus, the flux requirement can be at least one order of magnitude less that that outlined in Table 5 for a stress diffractometer. The resolution can also be lower, usually $\Delta d/d < 10^{-3}$. However, the instrument needs to be equipped with a vertical cradle stage (full circle or partial geometry such as ¼ cradle) or even a robotic system, to facilitate full coverage of all sample orientations for the compilation of the pole figure stereographic projections.

TABLE 5. REQUIREMENTS FOR A TYPICAL RESIDUAL STRESS DIFFRACTOMETER

Instrument	Residual stress and texture diffractometer	Texture diffractometer
Neutron beam spectrum	Thermal or cold. Access to >2.5 Å is beneficial for texture	Thermal or cold. Access to >2.5 Å is beneficial for texture
Minimum neutron flux ($n/cm^2/s$)	10^8 at beam port exit 10^5 at sample	10^7 at beam port exit 10^4 at sample
Recommended neutron flux ($n/cm^2/s$)	10^9 at beam port exit 10^6 at sample	$\geq 10^8$ at beam port exit $\geq 10^5$ at sample
Beam size (cm^2)	Typically 2 cm height and 2 cm width. Gauge volume configurations (> $1mm^3$) are selected in accordance with the required spatial resolution	Typically 2 cm height and 2 cm width with minimal collimation. Typical sample sizes for metals $0.6 \times 0.6 \times 0.6$ cm^3; mineralogical samples $2 \times 2 \times 2$ cm^3
Required space (m × m)	3 × 3	3 × 3
Minimum time to develop/install technique	1–3 years	1–2 years
Minimum cost of installation (million USD)	1–4 (depending on detection system and on CW/TOF options, as well as required in situ environments)	1–2
Human resources needed for operation	1–2 instrument scientists 1 technician Additional instrument scientists and/or software engineer for support to users	1–2 instrument scientists 1 technician Additional instrument scientist and/or software engineer for support to users

2.3.3. Instrument description

Residual stress instruments can be either CW or TOF configurations (see section 2.1.3), with each having their own characteristics and niches of application [37–41]. The general features of a CW diffractometer are displayed in Fig. 4, and those of a TOF instrument in Fig. 5. With CW instruments analyses are done from single peaks (enables d spacing determination), whereas TOF instruments record the entire diffraction pattern (enables determination of the lattice parameters from multipeak analyses). In long pulse neutron sources, a Fourier method is sometimes used to acquire the full spectrum [15]. Essential parameter and component considerations are:

- Wavelength: Range of 1.5–3.5 Å enables investigation of most materials;
- Resolution: Beam parameters such as divergence and $\Delta\lambda/\lambda$ should enable diffraction angle precision of at least 0.01° (which corresponds to a strain of 0.01% measured at $2\theta = 90°$);
- Gauge volume definition: The region of interest in a sample is established with a well defined small volume (dimensions and shape) fixed in space over the sample table vertical axis. This is achieved by the apertures that define the beam in the incident and diffracted beams, either in the form of slits, or radial collimators. The region of overlap between the incident and diffracted beams define the gauge volume;

- Sample positioning: If the objective is to measure the distribution of stresses at different locations and directions within specimens, the positioning accuracy should be at least 10% of the gauge volume dimension. This requires bringing different locations and sample orientations in precise alignment with the gauge volume using a specimen positioning table that is equipped with three mutually orthogonal linear positioning axes (x,y,z) and a vertical rotation stage;
- Sample setup aids: Laser beams (trackers) and high precision optical devices such as theodolites enable sample setup to accuracies of 100 microns. Higher accuracies are particularly important when measuring through steep strain gradients, interfaces, surfaces or when large translation scans are required. This is accomplished with surface entry scans of the sample through the gauge volume in conjunction with appropriate model fitting that incorporates the gauge volume shape, sample geometry and sample attenuation. Accuracies should be recorded and reported;
- Detector: A linear detector for CW measurements that covers at least double the width of a diffraction peak; with TOF, a bank of single detectors positioned at 90° geometry.

The instrument efficiencies can be substantially improved with the incorporation of area detectors for CW and multiple banks for TOF, i.e. at -90° and +90°. The latter enables simultaneous measurement of two orthogonal strain components.

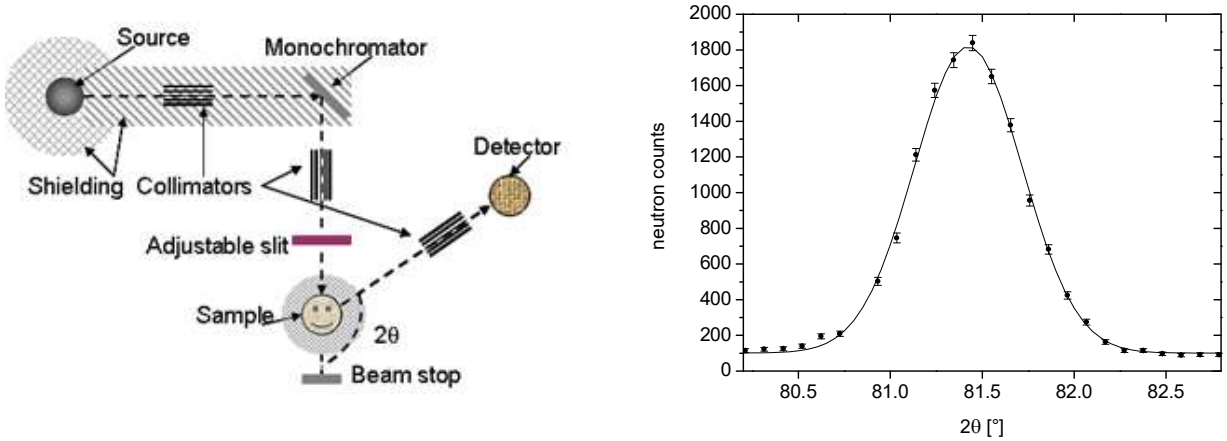


FIG. 4. Left: Schematic illustration of CW diffractometer for strain measurement. Right: Example of a single diffraction peak measurement [38].

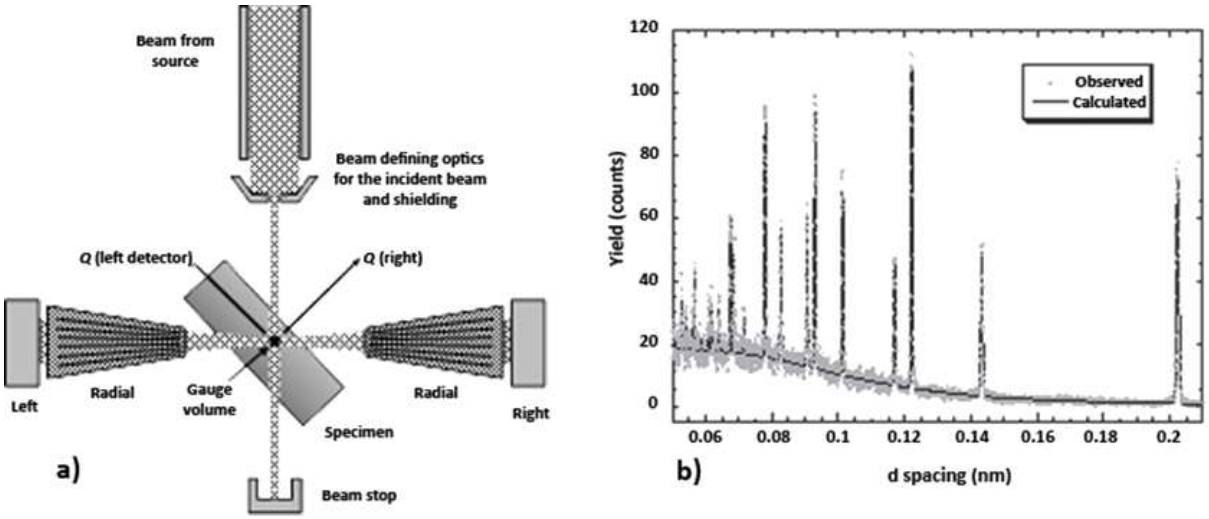


FIG. 5. Left: Schematic of a TOF diffractometer. Right: TOF diffraction pattern as measured in each of the two detectors [38].

In texture investigations, the sample needs to be rotated in space to ensure that all sample orientations are measured on a measurement grid that enables representation of data as a stereographic projection, pole figure, for each (*hkl*) reflection. Subsequently, in a CW instrument this requires the use of either a full or ¼ Eulerian cradle (χ positioning from 0° to 90°) and ϕ rotation at each χ setting that covers the range 0° to 355° (see Fig. 3b). The latest trends are to replace the goniometer with a robot since it does not obstruct the beam and can be used in conjunction with a sample changer for enhanced sample throughput. Typical metals can be investigated with wavelengths of 1–2 Å. Studies on mineralogical samples though require longer wavelengths typically > 2.5 Å to overcome extensive peak overlap.

2.3.4. Sample requirements

Neutron residual stress measurements can be carried out on most solid natural and industrial materials provided that they are crystalline and ideally fine grained. The most fundamental limitation is the total path length of the neutron beam through the material of the sample. For materials like steels that display strong diffraction, the depth of penetration is limited by depletion of the beam to path lengths in the order of 50 mm. In general, neutron diffractometers are large and robust with sample handling capabilities up to hundreds of kilograms.

Residual stress investigations are normally performed at ambient conditions. The reference sample — usually called the d_0 sample — is made of the same material as the investigated one but believed to be stress free. Usually a 2×2×2 or 3×3×3 mm^3 cube cut from the stress free region of the original sample is used [37–40].

Conditions representative of practical in service use, or materials science problems can be mimicked with in situ studies by incorporating equipment such as:

- Bending devices to study elastic properties, fatigue and others;
- Load frame (tension, compression, torsion) to study elastic properties, fatigue, the anisotropy of elastic and plastic properties and others;
- Eulerian Cradle to study texture (preferred orientation);
- Furnace to study stresses related to phase transformations, thermal stresses, stress relaxation, stress changes induced by temperature changes, etc [15, 40, 41];
- The ideal samples for texture investigations are spheres with typical diameters of 6 mm, since this offers uniform attenuation at all sample orientations. Alternatively, samples can be cubes with 6 mm edges, but corrections need to be applied to account for the different beam path lengths and thus attenuation;

Due to the weak scattering and attenuation of geological materials, sample sizes can be substantially increased to typically 20 mm edges to increase the scattered intensity. Full sample illumination though needs to be maintained throughout the investigations.

2.3.5. Data

Data files and formats vary considerably between institutes, but there are campaigns to standardise data formats, such as the NeXus format [43] that includes metadata in addition to the diffraction data.

The peak angular position and its associated statistical uncertainty is usually obtained by fitting the experimental peak profile with an appropriate function, e.g. a Gaussian, a pseudo Voigt or a known (measured) function of the TOF spectrum [44]. In the case that the diffraction pattern is acquired over a broad angular range that includes a number of peaks, different algorithms can be used to analyse the entire spectrum, such as the Rietveld, Pawley or Le Bail profile methods [45]. Strain and stress calculations from these fits are usually done using spreadsheets.

Data treatment and processing for texture analysis is much more involved since geometrical corrections need to be applied to account for attenuation corrections. Data is represented as pole figures for each (*hkl*) reflection. A full texture analysis involves the establishment of orientation distribution functions. Limited open source software is available, such as:

- popLA (preferred orientation package – Los Alamos) [46];
- MAUD stands for Material Analysis Using Diffraction [47].

2.4. LIQUID AND AMORPHOUS MATERIALS DIFFRACTION

2.4.1. Purpose

This technique aims at studying the structure of liquids, glasses and other non-crystalline solids, i.e. the absence of long range order that characterizes crystalline materials. Liquids and amorphous solids do, however, possess a rich and diverse array of short to medium range order, which originates from chemical bonding and related interactions. Fig. 6 categorises the different degrees of disorder that occur in condensed matter systems [48]. Assuming that the elementary constituents are molecular units, we find that the disorder may be due to the lack of translation or orientation symmetry of those units.

Liquid and amorphous diffraction techniques are aimed at the study of the systems indicated in the coloured areas of Fig. 6, with emphasis in the top right quarter of the representation. The objective of the technique is to determine the structures of atoms and molecules in such systems. Experimentally, the structure factor $S(Q)$ must be determined, from which the spatial correlation functions $g(r)$ are obtained by Fourier transform. This requires measurements over a wide Q range, thus requiring the use of high incident neutron energies (~500 meV, 0.4 Å).

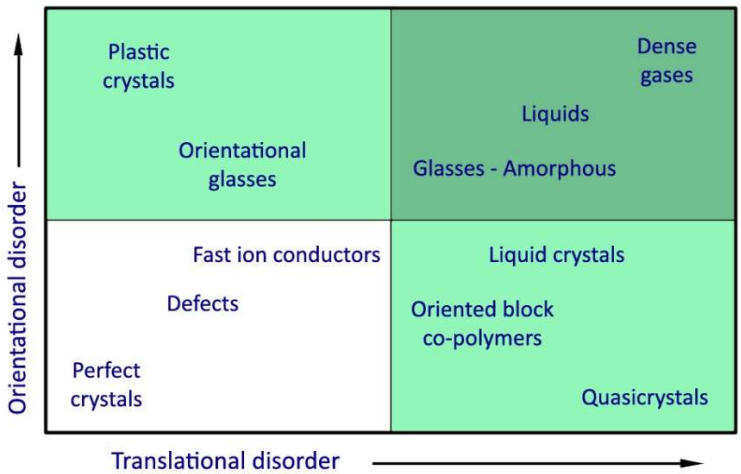


FIG. 6. Classification of materials according to their degree of orientational and translational disorder. The coloured areas indicate the potential application domain of diffraction techniques for liquids and amorphous systems. The main domain for such diffractometers is the top right quadrant.

Liquid and amorphous diffraction techniques can be implemented at medium flux neutron sources. They can also be implemented at low neutron flux sources, if large samples are investigated. Some research reactors, including TRIGA type ones [49], have a spectrum with a fairly high fast neutron component, which is a favourable condition. Compact accelerator based neutron sources (CANS) have a fast neutron spectrum, so even low flux CANS may be appropriate for liquid and amorphous diffraction techniques.

2.4.2. Requirements

Liquid and amorphous materials diffraction requires the use of epithermal neutrons. In reactor based neutron sources this is not easily achievable, since the typical thermal spectra do not provide sufficient flux in the required energies and high values of Q mentioned above. For that reason, a hot source must be implemented (section 3.2.2). This consists of a graphite block that can be heated to well over 2000 K by the gamma radiation emanating from the uranium fission originated in the fuel element. The insertion of a hot source in a reactor that is already in operation requires redesign and modification of the core environment. Therefore, its implementation is usually done as a design parameter during the development phase and is not common in low to medium flux reactors. In some research reactor designs (such as TRIGA [49]) the neutron spectrum extends to the near epithermal energy part, which can be used for such experiments. In contrast, in accelerator based sources, the neutron energies naturally extend to the epithermal region, requiring only an adequate design of the moderators.

Table 6 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate a liquid and amorphous materials diffraction instrument. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments. A resolution $\Delta d/d \approx 10^{-2}$ is usually required.

2.4.3. Instrument description

Fig. 7 shows a typical setup for a liquid and amorphous diffractometer installed on a small accelerator based neutron source.

The following parts of the system are distinguished for application on an accelerator based neutron source:

- Fast neutrons produced in a target. For electron accelerators, the target is a heavy element such as lead, tungsten, etc., while for proton accelerators lithium and beryllium targets can be employed. Typically, accelerators are pulsed at repetition rates of 12.5, 25, 50 and 100 pulses per second;
- Moderator: normally a hydrogenous material is used that provides a spectrum ranging from thermal to epithermal energies, usually corresponding to under moderated neutrons;
- Collimation systems inside a vacuum tube, made of a material that takes the required neutron spectrum into consideration, such as boron containing hydrogenated plastic.

Typically, a beam diameter of 5 cm is suitable. Using smaller beams, the reduced neutron count rate needs to be corrected for by increasing the sample size;

- Beam monitor placed before the sample, used to sense the beam stability and to normalize the measurements;
- Detector systems. In Fig. 7, three detector banks are displayed for the most efficient case (selection of the most appropriate detector system is made considering desired performance and available budget):
  - Backscattering. It provides the best Q resolution but has lower count rate. It is recommended only if the instrument will be used also for crystallography;
  - Scattering at 90º. Better suited for studies of amorphous and liquid systems;
  - Forward scattering. Has a lower Q range, but better resolution at low Q.

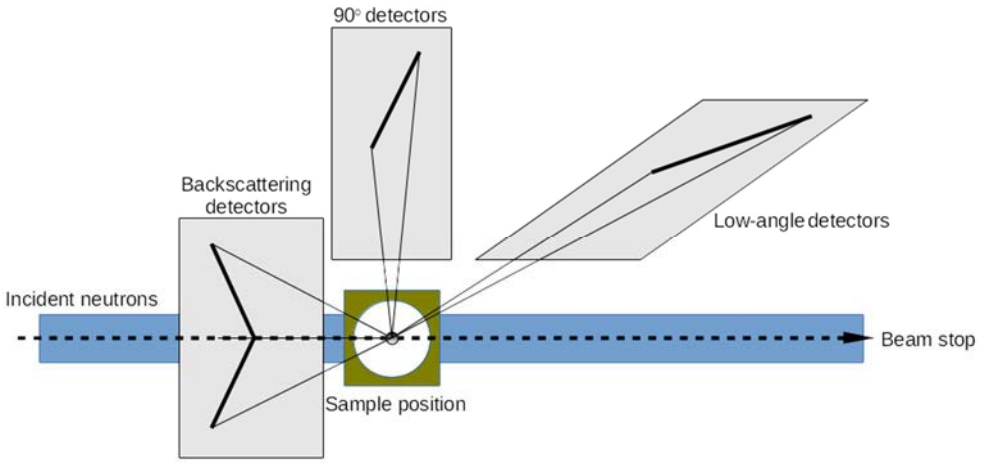


FIG. 7. Proposed experimental setup for a liquid and amorphous diffractometer based on an accelerator source (courtesy of CNEA, CONICET, Centro Atómico Bariloche, Argentina).

TABLE 6. REQUIREMENTS FOR A TYPICAL LIQUID AND AMORPHOUS MATERIALS DIFFRACTOMETER

Instrument	Liquid and amorphous materials diffractometer
Neutron beam spectrum	Epithermal
Minimum neutron flux (n/cm^2/s)	10^7 at beam port exit 10^4 at sample
Monochromator Cu, Si, Ge	Focussed
Recommended neutron flux (n/cm^2/s)	$\geq 10^9$ at beam port exit $\geq 10^6$ at sample
Beam size (cm × cm)	10 (height) × 1 (width)
Required space (m × m)	3×3 for 90º and low angle, plus 1×3 for backscatter detector
Minimum time to develop/install technique	2–4 years
Minimum cost of installation (million USD)	0.5–1.5
Human resources needed for operation	1 instrument scientist 1 technician Additional instrument scientists and/or software engineer for support to users

In the last two decades, “total scattering” experiments have been extended to cover some of the other quadrants shown in Fig. 6, e.g., including poorly crystalline and plastic crystals. The techniques of measuring the structure factor $S(Q)$ are similar, although higher resolution designs may be optimal to fit the crystalline part of the signal (Bragg peaks) simultaneously with the more slowly varying amorphous component. Some state of the art instruments dedicated to liquids and amorphous in large installations such as the General Materials Diffractometer GEM in ISIS (UK) [50] or the NOVA High Intensity Total Diffractometer in J-PARC (Japan) [51] have much greater coverage of solid angles with a large number of detectors, and the design approaches that of a powder diffractometer. However, the setup proposed here is according to the limited budget and resources usually available at low and medium flux neutron sources.

The detectors in each bank are placed on a $Qt = constant$ surface, so that all the elastic coherent scattered neutrons reach the detector bank at the same time, regardless of the scattering angle [52]. It is important to note that each of these detector banks (in particular that at 90º scattering) functions as an independent diffractometer, with the Q range given by the TOF spectrum. More detectors around the sample position will give extra information, superimposed in certain Q ranges, with different experimental resolutions. The detector banks can be composed of ^{3}He tubes. Li scintillators are used in accelerator based sources, to detect higher energy neutrons, although the gamma sensitivity is higher than that of ^{3}He tubes. For both CW and TOF liquids diffractometers, good calibration of the detector efficiencies is paramount as is the stability of the detectors over time.

2.4.4. Sample requirements

Normally the sample is at a distance of a few metres from the moderator. The setup geometry requires flat geometry homogeneous samples. The sample area must be chosen to completely cover the beam size. If a higher flux is available a small area beam can be used to scan different parts of an inhomogeneous sample. A sample thickness criterion is that the scattering rate should be about 30% of that of the incoming neutron beam. Though a higher rate will produce stronger signals, it also leads to more multiple scattering processes which need to be subtracted (described below in section 2.4.5 on data). The sample can be self supporting or be inside a container. Isotopic substitution is often used to improve scattering in samples measured with this technique, where it is simpler to implement than in crystalline samples.

Sample environments with control of vacuum, temperature, pressure, humidity, magnetic field, etc., can enrich the information provided by this technique. Normally the sample is at a distance of a few metres from the moderator.

2.4.5. Data

Data processing software is usually developed by each laboratory independently. Software for atomic powder distribution function analysis is sometimes used. An advanced data reduction and an analysis solution is presented by Mantid [53, 54], an open source collaborative software contributed by users and shared with the community. It enables:

- Empty cell subtractions. Sample containers are sometimes used, for instance for liquid, solid or powder samples. In that case, blank measurements, i.e. of empty sample containers, are routinely made in order to subtract their contribution to the signal [55];

- Multiple scattering corrections. The experimental data must be corrected for beam attenuation and multiple scattering effects. This is best achieved by Monte Carlo simulations. As no established software exists, reference [56] and references therein provide a guide to the reader [57, 58].

2.5. SMALL ANGLE NEUTRON SCATTERING

2.5.1. Purpose

The small angle neutron scattering (SANS) technique is an important tool for characterizing materials which provides information about structures in the 10–1000 Å size by investigating scattering in the low Q range ($Q < 0.5$ Å^{-1}). This requires the use of neutron wavelengths covering 3 to 20 Å. SANS instruments are commonly found at medium flux neutron sources. At low flux sources this is more challenging and requires efficient neutron focusing in conjunction with a large area detector. If a cold neutron source is not available, the available wavelength will be limited to around 3 Å or even less, leading to poor resolution and Q coverage. In such case, the technique will be limited to samples that have strong scattering centres such as nanocrystals and nanoparticles with good contrast with respect to the matrix, magnetic liquids, etc. It should be noted that CANS can be optimized for a given application, and therefore small CANS could become useful for SANS.

In SANS, the neutrons are scattered in the [000] direction (i.e. transmitted beam) from scattering length density variations. This can be due to chemical or isotopic composition, density or magnetic structure of the sample. The technique is used to determine the shape and the organization of particles or aggregates dispersed in a continuous medium.

SANS provides information on object sizes, forms, surfaces and possible correlation of scattering objects. It can be applied to the investigation of a wide range of matter, such as small colloidal particles (clay, ferrofluids, nanotubes), surfactant aggregates (micelles, lamellar, hexagonal, cubic, or sponge phases), polymers and all their derivatives, liquid crystals, model membranes, proteins in solution, flux line lattices superconductors, pores and voids, interpenetrating phases, ceramics, catalysers, biological objects, magnetic nanoparticles, spin glasses, skyrmions, chiral solitons, etc. [3, 4, 59–61].

2.5.2. Requirements

SANS usually employs cold neutrons to enable investigation of features with sizes 1–100 nm. However, at low and medium flux reactors, thermal neutrons can also be used for SANS by using an alternative configuration composed of the non-dispersive arrangement of two monolithic channel crystals [62]. Table 7 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate a SANS instrument. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments.

TABLE 7. REQUIREMENTS FOR A TYPICAL SANS INSTRUMENT

Instrument	SANS
Neutron beam spectrum	Cold or sub thermal
Minimum neutron flux (n/cm^2/s)	10^5 at beam port exit 10^3 at sample
Recommended neutron flux (n/cm^2/s)	$\geq 10^8$ at beam port exit $\geq 10^5$ at sample
Beam diameter (cm)	1–2
Required space (m × m)	Width 2 × length 6–50
Minimum time to develop/install technique	1–3 years
Minimum cost of installation (million USD)	1–3
Human resources needed for operation	2–3 instrument scientists 1 technician Additional instrument scientists and/or software engineer for support to users

2.5.3. Instrument description

There are two main configurations of SANS setups, as for other diffraction instruments, i.e. the CW and TOF SANS (see section 2.1). Firstly, we describe the CW SANS setup.

2.5.3.1 CW SANS

A conventional CW SANS instrument (Fig. 8) consists of a coarse monochromator (either a velocity selector with $\Delta\lambda/\lambda$~10–20% or channel cut monochromators composed of slightly misaligned crystals packs), a beam collimation system (which can be a simple series of pinhole elements up to 20 mm diameter to provide a collimated beam with 0.01–0.002 arc minute (~0.14º) divergence), a sample position table and a detector (usually a 2D configuration of BF_3 or ^{3}He gas filled detectors with resolution in the 3–10 mm range; microstrip, B_4C foil detectors, or circular tube arrangement can also be used). To increase the intensity, special beam focussing systems such as neutron mirrors or neutron lenses can be incorporated.

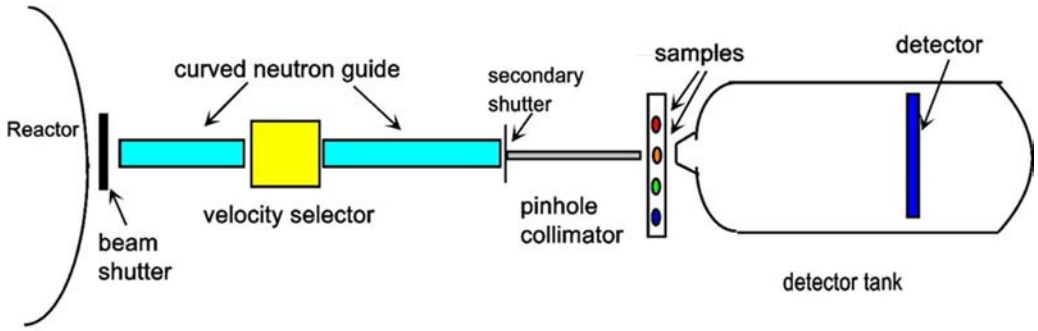


FIG. 8. Schematic setup of a conventional SANS spectrometer that employs a velocity selector as beam monochromator device (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

In the absence of a cold neutron source, the SANS technique can use the cold neutron tail of the thermal beam, in the 3–8 Å range. In this case, an intense incident beam is usually needed. As the neutron beam in medium and low flux neutron sources is less intense, the technique is preferably applied to the research of samples that have high scattering cross sections, such as ferrofluids [4] or amorphous carbon containing materials and ceramics. It is also appropriate for conducting research programmes in those areas, for E&T activities, testing sample environments and preliminary experiments in preparation for submitting proposals to higher flux facilities.

Usually the minimum sample to detector distance is 4 m at a given 2D detector resolution (usually 3 to 10 mm). The conventional collimation system has a similar distance to the sample detector system, around 4 m. If a multi beam setup is used, the collimator system can be shortened. The usual sample size is 10–20 mm × 10–20 mm, adjusted to the beam size and to the detector resolution.

In the presence of a cold source (see section 3.2.1) the wavelength region is extended to 20 Å, consequently the achievable Q range is also smaller. Such instruments are substantially larger, with overall dimensions in excess of 20 m.

2.5.3.2 TOF SANS

In a TOF SANS setup [61], pulses of collimated chopped neutrons are used. The typical flight path in the case of broad pulses (e.g. 300 μs) is 25 m. With shorter pulses (e.g. 60 μs), a 3–5 m flight path is sufficient to obtain good resolution. Cost effective ring shaped detector arrangements are commonly used.

Measurements under external magnetic fields are more complex with TOF SANS than with CW SANS. It should also be noted that in TOF SANS experiments that use shorter wavelength neutrons (less than 6 Å) double Bragg scattering may occur, leading to degradation of the signal quality. Some SANS instruments have options to address this by polarizing the incoming neutron beam, but these are seldom found at low or medium flux neutron sources.

2.5.4. Sample requirements

The sample table very often is equipped with several sample positions (automated sample changer) to ensure effective beam time utilisation since the measurement time is usually short (hours). This enables a full set of measurements in a single run, i.e. measuring the sample, background, sample for normalisation (water/vanadium) and sample holders. The sample size is dependent on the beam size, usually a maximum diameter of 20 mm. The minimum is determined by the beam intensity. Area specific information can be obtained by scanning the sample through a small area beam.

There are no special requirements on the sample phase. Liquid, amorphous, single crystal and even biological living organ (tissue, vegetal, viruses) samples can be measured.

The sample thickness is determined by its neutron transmission to avoid multiple scattering effects. Usually 50% transmission is considered as a limit. In the case of strong absorption, lower transmission values can be accepted as the absorbed neutrons do not contribute to the SANS pattern. Typical thicknesses are a few millimetres.

The sample environment is easily adapted to the various needs of the users. Most SANS instruments are equipped with a remote controlled sample changer with temperature control. Cryostats, cryofurnaces, furnaces, electromagnets are also available. A vacuum chamber can be used for very low scattering samples to reduce the scattering from air or to keep sample environment parameters stable. An automated sample changer is desirable.

2.5.5. Data

Preliminary data treatment can be performed with freeware available packages such as BERSANS [63] and GRASP [64]. For model fits, codes such as FITTER [65], SASFIT (PSI) [66] and SASVIEW (NIST) [67] (the IGOR packages), or GRASP (ILL) can be used [64]. For TOF-SANS data the time of flight data must be transformed to q data [53, 54, 68]. BornAgain is an open source software to simulate and fit neutron and X ray reflectometry and grazing incidence small angle scattering [69].

2.6. NEUTRON REFLECTOMETRY

2.6.1. Purpose

Neutron reflectometry is applied to the investigation of layered systems or lateral structures, with the purpose to determine parameters such as layer thickness and density, magnetic features and surface and interfacial roughness [70–75]. Neutron reflectometry is generally found at medium flux neutron sources. It is less used at low flux neutron sources, but becomes viable with the incorporation of efficient neutron focusing techniques. In such cases, it is usually limited to large area solid samples. Applications studied with neutron reflectometry include:

- Solid films and superlattices: layer magnetizations and hysteresis, lateral patterns, exchange coupling, antiferromagnetic exchange coupling of e.g. Heusler alloys, exchange bias effects, isotopic superlattices, phase transitions, hydrogen and oxygen profiles in metal superlattices, superconductivity and determination of magnetic structures, especially large scale structures, i.e. helical spin density waves or magnetic lattices [74–76];
- Soft films and multilayers at solid–air and solid–liquid interfaces: biomimic membranes, colloids, liquids under shear and their swelling behaviour in presence of various vapours, polymers adsorbed in a confined geometry, self-assembled polymer monolayers [72, 77];
- Materials science: polyelectrolytes, polymer melt interfaces, ion implantation, ionic liquids and ferrofluids. The study of surfaces and buried interfaces of thin solid films and multilayers, surfactant/oil mixtures at the air/water interface [72];
- Technical developments: neutron waveguides as neutron optical devices, coatings, examination of off specular reflectivity from atomic and magnetic in plane structures;
- Kinetic studies of interface evolution [74, 75, 78];
- Dynamics of magnetic excitations (induced by externally applied fields) [76];
- Extreme environments: determination of structures and dynamics under pressure, temperature and other conditions [77, 78];
- Biological systems such as solid or liquid supported membranes (e.g. determination of the morphology and localization of proteins at interfaces) [72].

2.6.2. Requirements

Neutron reflectometry usually employs cold neutron instruments that use a pyrolytic graphite monochromator and filter, the latter needed to remove the higher harmonics from the monochromator[70–73]. Some instrument use neutrons of thermal energies but these are not common [79].

Table 8 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate a neutron reflectometry instrument. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments. Table 9 has further information.

TABLE 8. REQUIREMENTS FOR A TYPICAL NEUTRON REFLECTOMETRY INSTRUMENT

Instrument	Neutron reflectometry
Neutron beam spectrum	Thermal, cold
Minimum neutron flux (n/cm^2/s)	10^6 at beam port exit 10^3 at sample
Recommended neutron flux (n/cm^2/s)	$\geq 10^8$ at beam port exit $\geq 10^5$ at sample
Beam size (cm^2)	> 10
Required space (m × m)	2 (width) × 3–5 (length)
Minimum time to develop/install technique	1–3 years
Minimum cost of installation (million USD)	0.5–1
Human resources needed for operation	1–2 instrument scientists 1 technician Additional instrument scientists and/or software engineer for support to users

A brief summary of neutron reflectivity principles is given in ref. [71]. Consider a neutron beam reflected from a flat surface between the air ($n = 1$) and a medium with refractive index n with an incident angle θ. We can define the wavevector $k = 2\pi/\lambda$ in air and k_n in the medium as $k_n^2 = k^2 - 4\pi Nb$. Then the refractive index is (see Figure 9):

$$n = k_n/k = 1 - \lambda^2 Nb/2\pi \quad (4)$$

where N is the number of atoms per volume unit and b is the neutron coherent scattering length. The product Nb is called the neutron coherent scattering length density.

At the interface between the air and a medium of index n, it holds that $\cos\theta = n \cos\theta_n$. In the case of total reflection $\theta_n = 0$, and the critical angle θ_c is given by $\cos\theta_c = n$, which for n values close to 1 leads to:

$$n^2 = 1 - \lambda^2 Nb/\pi + (\ldots)^2 \quad (5)$$

From $n^2 = \cos^2\theta_c = 1 - \sin^2\theta_c = 1 - \lambda^2 Nb/\pi$ we obtain: $\sin\theta_c = (Nb/\pi)^{1/2}\ \lambda = \alpha\lambda$ where the parameter α is used to characterize the material. Below the critical angle, total reflection occurs. Above the critical angle, the reflectivity is determined by the variation of neutron coherent scattering length density in the direction perpendicular to the surface. This provides information on the nuclear and magnetic depth profile in multilayered samples. Roughness, diffusion and lateral inhomogeneities can also be investigated. This technique is particularly useful in biological and soft matter systems, due to the strong interaction of neutrons with light elements and the low damage they cause.

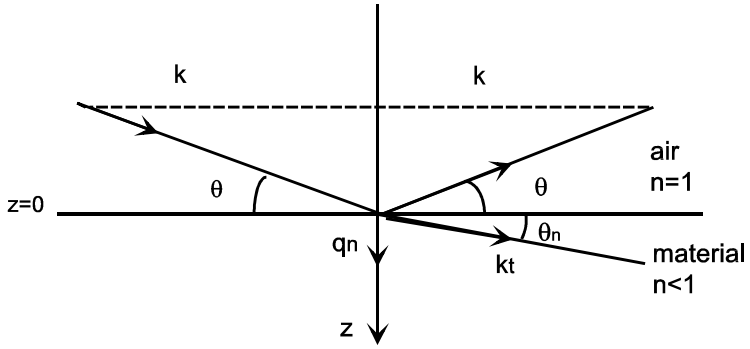


FIG. 9. Plane wave reflection from a flat surface (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences).

TABLE 9. MAIN PARAMETERS OF NEUTRON REFLECTOMETRY INSTRUMENTS

	Horizontal scattering	**Vertical scattering**
Instrument parameters		
Monochromator type	Monochromating supermirror HOPG, Si	
Incident wavelength	0.04–0.045 Å	
Resolution	$\Delta\lambda/\lambda = 0.05$	
Polarizers		
Type	Polarizing supermirrors or ^{3}He	
Flipper	Mezei type or Radiofrequency type	
Beam height/width	10–120 mm in vertical plane 1mm width	10–120 mm in horizontal plane 1mm height
Angular resolution	from 0.01 to 0.06°	
Angular range	0.2° to 15°	
Minimum reflectivity	10^{-5} to 10^{-6}	
Detection		
^{3}He detector	Single or multi	
Background at beam open	1 count/10 min	
XY multidetector	Microstrip with resolution 0.1 mm or Multiwire ^{3}He detector 200 × 200 to 500 × 500 mm (pixel size 1.8–2.5 mm, efficiency 80% at 4 Å)	
Ancillary equipment		
Field	0–1.2 T	
Cryostat	4 K–300 K (2 T)	

Table 10 gives the neutron coherent scattering length density values for a few materials. Typically, a resolution of $\Delta\lambda/\lambda = 0.05$ is required.

TABLE 10: NEUTRON COHERENT SCATTERING LENGTH DENSITY FOR BULK NICKEL, TITANIUM, SILICON, SILICA. N_A IS AVOGADRO'S NUMBER

	Ni	Ti	Si	SiO_2
density ρ (g.cm^{-3})	8.9	4.51	2.33	2.5
Atomic Mass M (g.mol^{-1})	58.71	47.9	28.09	60.09
$N = dN_AM^{-1}$ (cm^{-3})	9.13x10^{22}	5.67x10^{22}	5.00x10^{22}	2.51x10^{22}
b (cm^{-1})	1.03x10^{-12}	-0.3438x10^{-12}	0.41491x10^{-12}	1.58x10^{-12}
Nb (Å^{-2})	9.4x10^{-6}	-1.95x10^{-6}	2.07x10^{-6}	3.96x10^{-6}

2.6.3. Instrument description

The setup of the instrument for reflectometry depends on:

- Sample. For solid samples, a horizontal reflectometer scattering plane (sample vertical) is usually used. For liquid surfaces or liquid–liquid interfaces, a vertical scattering plane is needed (the sample remains horizontal);
- Neutron spectrum. For a monochromatic spectrum, the Q vector is varied via mechanical movement. In the case of a horizontal scattering plane, this is done by a rotation of the sample around the vertical axis (Fig. 10a). In the case of a vertical scattering plane, the incident beam direction is changed by a reflecting mirror and with a synchronized slit system (Fig. 10b).

For a pulsed source or a bunched beam, a TOF reflectometer can be built. The flight time depends on the wavelength of the neutron and consequently on the Q vector. The slit system and mechanics are fixed, and the instrument can be built for both horizontal and vertical scattering plane performance, as shown in Fig. 11a.

Fig. 11b shows an example to increase the intensity on the reflectometer by using a double monochromator system. The first monochromator focuses the full incident neutron beam coming from the neutron beam guide onto the second monochromator. The latter selects a narrow beam with higher $\Delta\lambda/\lambda$ available for the reflectometry measurements. The monochromators are typically made of pyrolytic graphite, which has high reflectivity. The intensity gain can be one order of magnitude, which is very important for medium and low flux neutron sources.

Furthermore, it is possible to distinguish three scattering geometries (see Fig. 12):

- Specular reflection (requiring a position sensitive detector);
- Scattering in the incident plane (off specular scattering);
- Scattering perpendicular to the incidence plane (grazing incidence SANS).

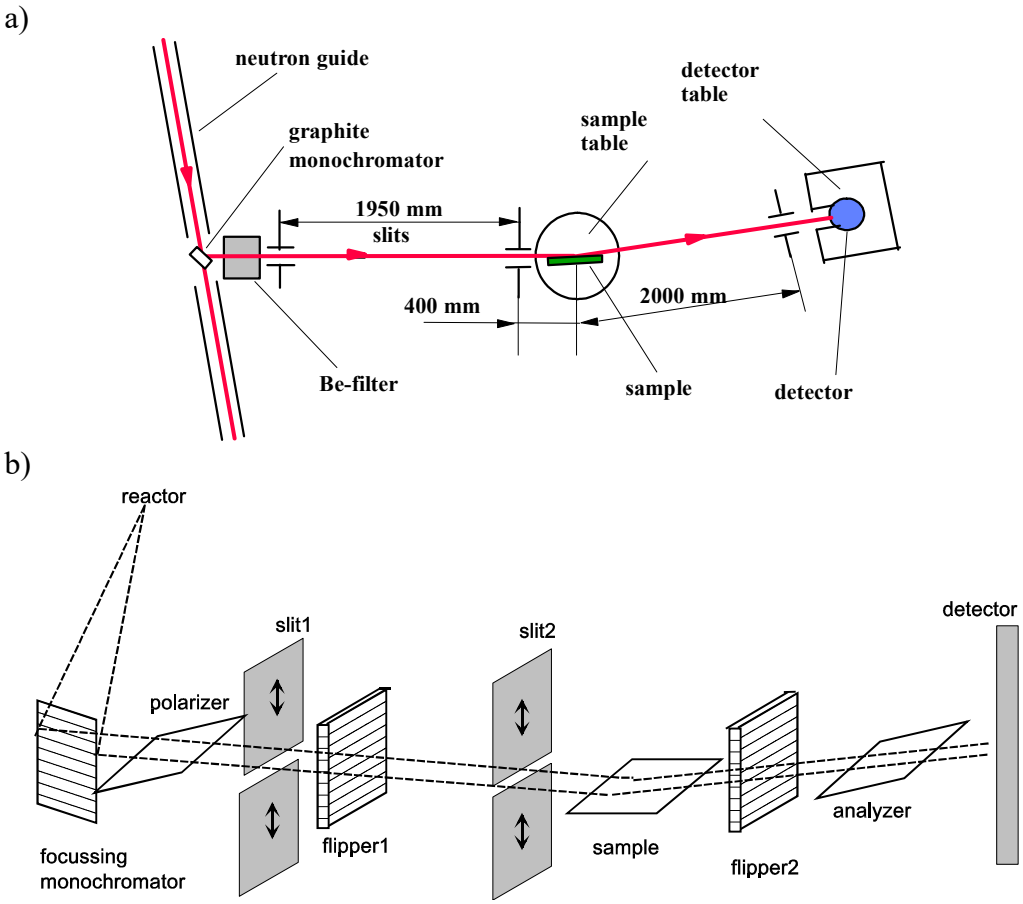


FIG. 10. a) Conventional horizontal scattering plane reflectometer installed at the Budapest Neutron Centre. b) Vertical scattering plane reflectometer with the incorporation of a polarised beam in instrument HZB Berlin V6 (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

These different scattering geometries probe different length scales ξ and directions in the sample surface, enabling the study of a very wide range of length scales ξ, from a few tens of angstrom up to several micrometres:

- Specular reflectivity probes the structure along the depth in the film (30 Å < ξ < 1000 Å; Q range $0.006 < Q_z < 0.3$ Å^{-1});
- Off specular scattering probes surface features at a micrometric scale (6000 Å < ξ < 60 μm; Q range $10^{-5} < Q_x < 10^{-3}$ Å^{-1});
- Grazing Incidence SANS which probes surface features (30 Å < ξ < 1000 Å; Q range $10^{-5} < Q_y < 0.3$ Å^{-1}).

In the case of polarized neutron reflectometry, a neutron polarizer, analyser and flipper are added. Polarized scattering can be a spin flip (S+-, S-+) and non-spin flip (S++, S--) process, so each measurement requires the collection of four spectra. Polarized reflectometry is possible for the different possible setups discussed. However, polarized TOF reflectometry is more difficult because it requires a wide range polarizer and analyser and may not be suited to even medium flux neutron sources. See Table 9 for a summary of characteristics of neutron reflectometry instruments.

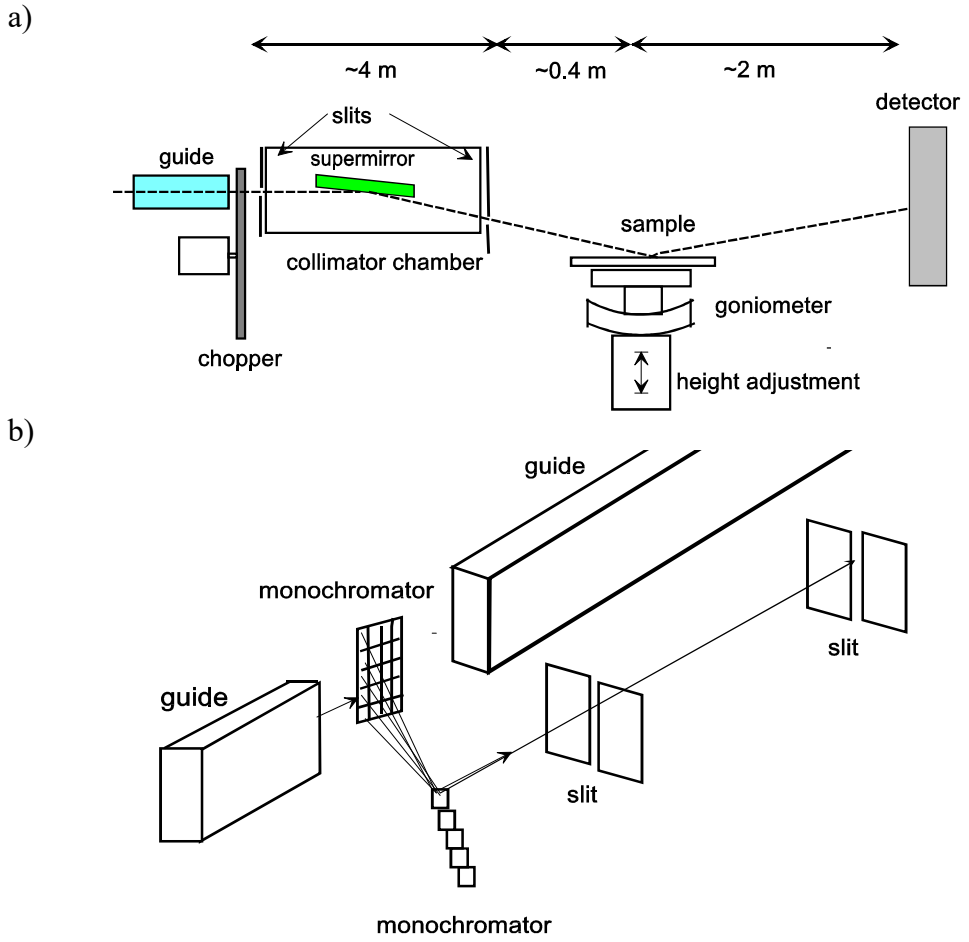


FIG. 11. a) Horizontal TOF Reflectometer HERMES at the Laboratoire Leon Brillouin, Orphee reactor. b) An upgrade of intensity in the Budapest Neutron Centre (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

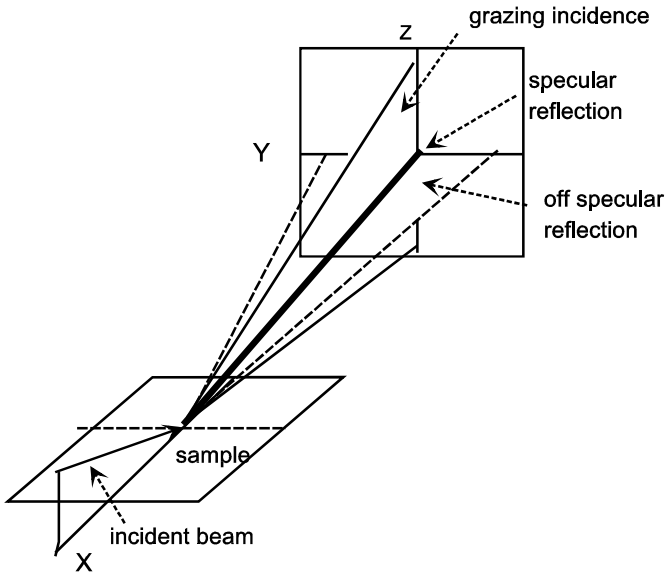


FIG. 12. The different surface scattering geometries. (Black line) specular reflectivity geometry; (dotted plane) off specular scattering plane, corresponding to the incidence plane; (XY plane) Grazing incidence SANS scattering plane, perpendicular to the incidence plane (XZ plane). These different scattering geometries probe a very wide range of length scales and directions in the sample surface (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

2.6.4. Sample requirements

Due to the use of grazing angles, the sample surface needs to be smooth at the atomic scale. The depth probed ranges from a few nanometres to some tens of micrometres. The samples must be larger than the incident beam area. If a higher flux is available, a small area beam can be used to investigate different parts of an inhomogeneous sample.

2.6.5. Data

Specialized software exists for the data treatment of neutron reflectometry experiments. Information about different programs can be found in the references [69, 80–86].

2.7. DEEP INELASTIC NEUTRON SCATTERING

2.7.1. Purpose

Deep inelastic neutron scattering (DINS) is an inelastic spectroscopic technique for the direct determination of the atom momentum distribution and mean kinetic energy (see also section 2.8 on triple axis spectrometry). It makes use of epithermal neutrons typically in the energy range 1 eV–200 keV (wave vector 20 $Å^{-1} < Q < 300$ $Å^{-1}$) and is therefore especially suited for accelerator based neutron sources [87], where an epithermal spectrum exists, with intensity maximised with an adequately designed moderator system.

The DINS method is used to obtain fundamental information on momentum distributions in the dynamics of atoms of light atoms in solids and liquids in many fields of physics, chemistry and materials science, as outlined below:

- Determination of the momentum distributions of the atoms that compose the sample (short time dynamics);
- Non-destructive mass spectroscopy. The technique is especially sensitive for light atoms [88];
- Determination of the effective temperatures of the atoms that compose the sample. The effective temperature differs substantially from the standard thermodynamic temperature because it takes into account the internal molecular degrees of freedom in the atom movement. This technique provides a direct measurement of effective temperatures, which makes this technique a potential source of data with direct applications to nuclear engineering;
- Determination of cross sections. Although not originally devised for this purpose, the technique is sensitive to the cross sections and the composition of the atoms that compose the sample.

The formalism of the DINS technique is derived for the limit of high energy and high momentum transfers, and is based on the Impulse Approximation of inelastic scattering. The main assumptions are that at high momentum transfer spatial atomic correlations, and thus collective phenomena, can be disregarded, and that the high energy transfers lead to very short interaction times where the target particle recoils freely after the collision with the incident particle, assuming that the binding energy of the target nuclei is negligible compared with the energy transferred by the neutron to the target nuclei [89]. The energy transferred to the target particle is significantly greater than its binding energy. Experimentally this requires tracking of both the flight direction of the scattered neutron and its energy.

2.7.2. Requirements

DINS usually employs epithermal neutrons. Table 11 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate a DINS instrument [1, 6, 90]. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments.

TABLE 11. REQUIREMENTS FOR A TYPICAL DINS INSTRUMENT

Instrument	DINS spectrometer
Neutron beam spectrum	Epithermal
Minimum neutron flux (n/cm^2/s)	10^7 at beam port exit 10^4 at sample
Recommended neutron flux (n/cm^2/s)	$\geq 10^9$ at beam port exit $\geq 10^6$ at sample
Beam size (cm^2)	10 (typically 5 cm height, 2 cm width or 5 cm diameter)
Required space (m×m)	3×3
Minimum time to develop/install technique	2–4 years
Minimum cost of installation (million USD)	0.5
Human resources needed for operation	1 instrument scientist 1 technician Additional instrument scientists and/or software engineer for support to users

2.7.3. Instrument description

A standard inverse geometry DINS experimental setup for an electronvolt spectrometer in a small accelerator based neutron source is shown in Fig. 13, and some parameters are given in Table 12 [91]. Incident high energy neutrons travel from the pulsed source/target to the flat geometry sample. Scattered neutrons at an angle θ and a given final energy travel along a path up to the fixed detector positions distributed in a ring like setup to increase the solid angle while keeping the angle fixed. A movable filter with a neutron absorption resonance in the eV energy region is placed in the scattered neutron path, so consecutive ‘filter in’ and ‘filter out’ measurements are performed. The intensity and widths of the signals measured depend on the sample composition, neutron scattering cross section and momentum distribution of the atoms.

DINS is one particular case of electronvolt spectroscopy [92]. With the addition of relatively simple equipment, the same experimental setup could be used for transmission experiments as well.

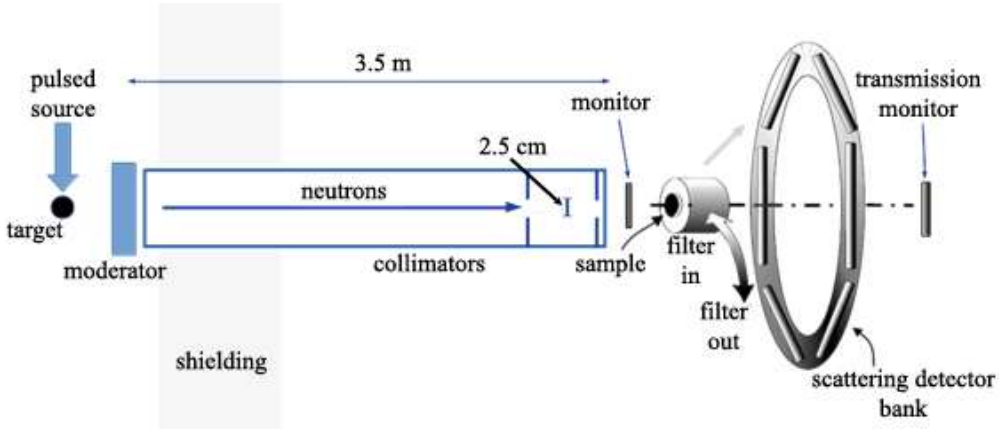


FIG. 13. Experimental setup for DINS experiments (courtesy of Instituto Balseiro, Centro Atómico Bariloche, Argentina).

TABLE 12. GENERAL INSTRUMENTAL PARAMETERS FOR A TYPICAL DINS INSTRUMENT

Space required	3×3 m^2
Monochromator	N/a
Sample size requirement	Larger than beam size
Detector	^{3}He tubes, end window tube Transmission monitor
Resolution	1 μs per channel

2.7.4. Sample requirements

The technique requires flat geometry homogeneous solid or liquid (eventually also gaseous) samples (amorphous, polycrystalline) larger than the incident beam area. If a higher flux is available a small area beam can be used to scan different parts of an inhomogeneous sample. The sample thickness will be chosen to provide a scattering rate of about 30% of the incoming neutrons, typical values being a few mm. The sample can be self-supporting or be inside a container. Sample environments accessories such as cryostats, pressure cells, gas handling devices and magnetic fields, can add extra dimensions and enrich the information that this technique provides.

2.7.5. Data

There is no established data processing software, and usually each laboratory has developed its own [91]. The recently presented data reduction and analysis open source application Mantid [53, 54] is based on collaborative software, so each user can contribute their own routines to be shared with the community.

2.8. TRIPLE AXIS SPECTROSCOPY

2.8.1. Purpose

The Triple Axis Spectrometer (TAS) is a versatile and powerful tool based on inelastic neutron scattering to investigate the dynamics of crystals and liquids and magnetic excitations of materials [93]. Lattice dynamics governs the thermal properties of solids i.e. phase transitions, thermal expansion, specific heat and thermal conductivity. Spin dynamics can

explain magnetic phenomena in superconductors, multiferroics, low dimensional magnetic systems and quantum spin liquid materials. This technique can be found at medium flux neutron sources. It is not appropriate for low flux neutron sources.

In an inelastic neutron scattering event, momentum is transferred from the neutron to the sample or vice versa. This inelastic scattering of neutrons creates or annihilates an excitation inside the sample, so that both the energy of the neutron and the internal state of the sample are modified (the energy gained by the sample is equal to that lost by the neutron, i.e. the total energy is conserved).

The instrument is called a triple axis because this type of spectrometer has three vertical axes of rotation, located in the monochromator, sample and analyser goniometers, as shown schematically in Fig. 14. The required resolution is typically $\Delta E/E < 10^{-1}$, as for a usual energy of 14.7 meV the energy resolution is often around 1 meV.

2.8.2. Requirements

Table 13 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate a triple axis spectrometer. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments.

TABLE 13. REQUIREMENTS FOR A TYPICAL TRIPLE AXIS SPECTROMETER

Instrument	Triple axis spectrometer
Neutron beam spectrum	Thermal
Minimum neutron flux (n/cm^2/s)	10^7 at beam port exit 10^4 at sample
Recommended neutron flux (n/cm^2/s)	$\geq 10^9$ at beam port exit $\geq 10^6$ at sample
Beam size (cm^2)	10 (typically 5 cm height, 2 cm width)
Required space (m×m)	5×5 including monochromator drum
Minimum time to develop/install technique	2–4 years
Minimum cost of installation (million USD)	1–2
Human resources needed for operation	1–2 instrument scientists 1 technician Additional instrument scientists and/or software engineer for support to users

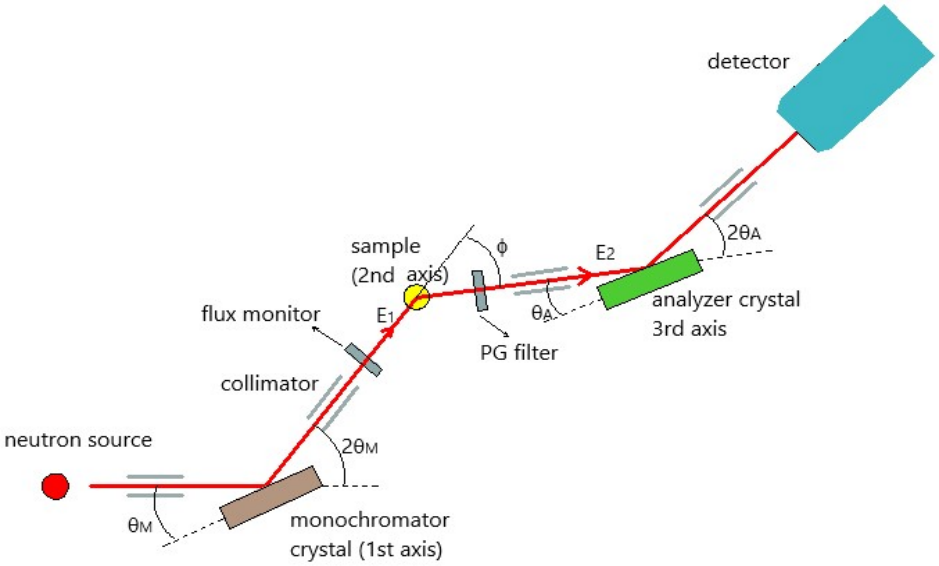


FIG. 14. Inelastic neutron scattering process in TAS (courtesy of BATAN, Indonesia).

2.8.3. Instrument description

Fig. 14 illustrates the working principle of a TAS. The monochromator produces a neutron beam, with initial wavenumber k_1 and energy E_1, which is then scattered inelastically by the sample. The scattered beam has new values of wavenumber k_2 and energy E_2, which are quantitatively determine using a crystal analyser. The momentum transfer $Q = k_2 - k_1$ and energy transfer $E = E_2 - E_1$ represent the characteristics of the crystal lattice and magnetic spin dynamics. Higher order reflections from the monochromator/analyser can be suppressed by a neutron filter. In the case that the phonon spectrum is also measured, the higher order reflections lead to signals in different positions in angular space, so they can be separated in the data treatment. Nevertheless, a filtered beam is usually preferred.

A typical TAS has two detectors which include a beam monitor (before the sample) and a main detector (single, multi or area detector). An analyser crystal is used to determine or analyse the final energy and wave vector of neutron scattered by the sample. The details of the common components/parameters of a conventional TAS are summarized in Table 14.

TAS instruments can be upgraded using a multidetector system. Multiple analysers with separate detectors in one detector bank enable the recording of several transmitted energies. Another possibility is to use a large flat analyser and a one dimensional position sensitive detector, as was done in the RITA instrument [94]. Facilities with considerable resources have transitioned from a multi detector strategy to combined multi analyser multi detector options, but these are difficult to implement in most low or medium flux neutron sources.

For magnetic applications the shielding and other instrument components should be composed of non-magnetic materials whenever possible, in order not to affect the measurement process [95], which increases the cost of the setup. Similar considerations may apply to other techniques as well.

Because the sample is a single crystal, during the experiment Bragg reflection can lead to a sharp and intense beam, which needs to be taken into account in the shielding arrangement.

TABLE 14. CONVENTIONAL TAS PARAMETERS AND COMPONENTS CHARACTERISTICS [96, 97]

Instruments/Parameters	Value	Conditions
Beam narrow (D)	170 mm	Neutron thermal flux of 10^6-10^7 n.cm^{-2}s^{-1}
Monochromator	Cu [002]	d_{hkl} = 3.437 Å
	HOPG [002]	
	Heusler [111] $CuMn_2Al$ (230x150mm^2)	
	Si [111] (190x200mm^2)	d_{hkl} = 3.135 Å with horizontal and vertical focusing
Analyzer	Heusler [111]: Polarised beam	d_{hkl} = 3.437 Å
	Si [111]: No second order contamination	d_{hkl} = 3.135 Å with horizontal and vertical focusing
	HOPG [002]: Highest reflectivity	d_{hkl} = 3.355 Å with horizontal and vertical focusing
^{3}He Detector	4 x 10 cm^2 (vertical)	
Beam cross section	20 x 50 mm^2	
Collimations (FWHM)	Premonochromator: 20', 30', 40' Monochromator–sample: 20', 40', 60' Sample–analyser: 20', 40', 60', Analyzer–detector: 30', 40', 60'	

2.8.4. Sample requirements

Samples for lattice and spin dynamics experiments must be solid phase single crystals with known lattice parameters and orientation. In the case of bulk liquids, no orientation is required. The required sample size depends on the intensity at the sample position. For medium flux neutron source, a minimum dimension around 1 cm^3 can be considered. Larger crystals will give stronger inelastic signals. If larger crystals cannot be grown, another possibility is to coalign multiple crystals to make a composite of greater scattering power [98]. TAS can also be equipped with sample environments for high pressure, low temperature (cryostat), high temperature (furnace) and external magnetic fields (cryomagnet).

2.8.5. Data

Depending on the mode of measurement, the steps to plot phonon or magnon dispersion relation differ slightly from one mode to another. As an example, for measurement with constant Q and energy transfer mode scans, typical raw data of a TAS consist of neutron scattering intensity as a function of energy transfer E at specified scattering vector Q. There are at least two steps to plot the dispersion relations from raw data. Firstly, the raw data are fitted to get the value of E at specified Q. Any software capable of performing Gaussian fitting will work. Second, the fitted energy transfer E vs Q is tabulated. Plotting E as a function of Q can be performed using any free or commercial plotting software.

Spurious signals called “spurions” can be generated by many processes that include higher order reflections from the monochromator and the analyser, and multiple scattering either

from within the sample itself, or often involving interactions with sample container walls [99] and need careful evaluation.

Besides plotting the dispersion relation, a number of well developed software packages exist for calculating resolution and performing Triple Axis Spectrometer simulation, i.e.:

- The ResLib code [100] analyses neutron inelastic scattering data for a triple axis spectrometer. It is based on the Gaussian approximation for the resolution function, supports different methods and includes fitting capability;
- The McStas code [101] is a neutron ray trace simulation package for neutron scattering instruments and experiments. This package is available for simulation a triple axis spectrometer functions and measurements;
- The VTAS [102, 103] software is a virtual three axis spectrometer with optional multiplexing, showing the instrumental setup and the resolution and intended energy impulse transfer;
- The RESTRAX package [104] is a Monte Carlo neutron ray tracing simulation mainly for TAS instruments;
- The Takin package [105] is an open source software for experiment planning, visualisation and data analysis, including calculation of resolution;
- SpinW [106, 107] is an open source MATLAB library that can plot and numerically simulate magnetic structures and excitations of given spin Hamiltonian using classical Monte Carlo simulation and linear spin wave theory;
- Phonopy [108] is an open source package for phonon calculations at harmonic and quasi-harmonic levels.

2.9. NEUTRON TRANSMISSION

2.9.1. Purpose

The neutron transmission technique is especially suitable with low and medium flux reactor (bunched beam) or accelerator based neutron sources, since it uses the direct beam. Its implementation is relatively simple, and transmission measurements can be done even at low power research reactors [6] or small electron accelerators [109]. Neutron imaging also uses neutron transmission but is outside the scope of this publication [8].

A well collimated neutron beam to limit the beam divergence, a sample changer, incident and transmitted beam monitor detectors and appropriate shielding around the equipment are common requirements. The most relevant information that can be obtained from this technique is through the energy analysis of the detected neutrons, for which the TOF technique is used in pulsed beams. In the case of accelerator based sources, pulsed beams are naturally obtained, while in continuous sources such as reactors, chopper systems must be implemented. A summary list of capabilities of the technique is:

- Total cross sections of molecular systems; employed to feed nuclear data libraries required by nuclear engineering (mainly thermal neutrons);
- The total cross section of crystalline materials. Through the study of Bragg edges, the crystalline structure as well as the texture of material can be studied (thermal neutrons). The crystal size and their orientation distribution and the texture of materials can be characterized;

- Neutron nuclear absorption resonances for nuclear physics studies (epithermal and fast neutrons);
- Main dynamical features such as resonant frequency of condensed systems (vibration of atoms);
- Small angle neutron scattering effects are also present in the transmission spectra, which enables the characterization of particle and pore sizes (sub thermal and thermal neutrons).

2.9.2. Requirements

Table 15 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate a neutron transmission instrument. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments. Typical timing resolution required is 1 μs.

TABLE 15. REQUIREMENTS FOR A TYPICAL NEUTRON TRANSMISSION INSTRUMENT

Instrument	Neutron transmission
Neutron beam spectrum	Fast to thermal
Minimum neutron flux (n/cm^2/s)	For research reactors: 10^6 at beam port exit 10^3 at sample For accelerators: Count rate: 10^2 counts per pulse
Recommended neutron flux (n/cm^2/s)	For research reactors: $\geq 10^8$ at beam port exit $\geq 10^5$ at sample For accelerators: Count rate: $\geq 10^4$ counts per pulse
Beam size (cm^2)	25 (typically 5 cm height, 5 cm width)
Required space (m×m)	10 m length × 2 m width
Minimum time to develop/install technique	1–3 years
Minimum cost of installation (million USD)	0.2 plus sample environment cost
Human resources needed for operation	1 instrument scientist 1 technician Additional instrument scientists and/or software engineer for support to users

2.9.3. Instrument description

Fig. 15 shows a typical layout for transmission experiments, based on an accelerator pulsed source. We distinguish the following parts of the system:

- Fast neutrons produced in a target. For electron accelerators, the target is a heavy element such as lead, tungsten, etc. For proton accelerators lithium and beryllium targets can be employed. The accelerator is typically pulsed at repetition rates of 12.5, 25, 50 and 100 pulses per second. The beam size is typically 5 cm in diameter;
- Moderator: In order to change the neutron spectrum, the moderator temperature can be heated or cooled;
- The main shielding between the target environment and the sample changer room is typically heavy concrete;
- Collimator systems inside a vacuum tube for beam shaping consisting of materials that absorb epithermal neutrons;
- Beam monitor placed before the sample, used to record the beam stability and to normalize the measurements;
- Sample changer, that must be automated to perform alternative measurements in different samples as well as open beam measurements;
- Detector system: Minimum single detectors with fast timing electronics for TOF.

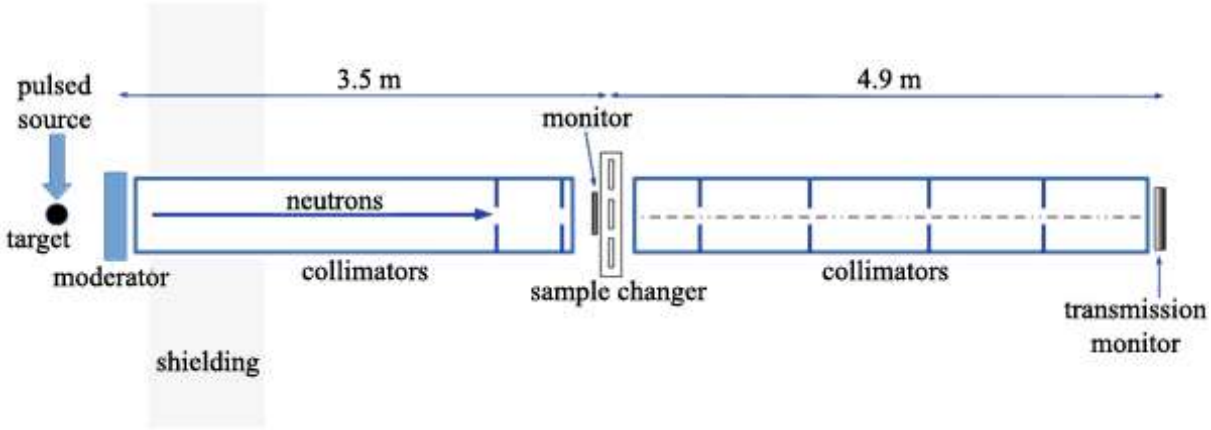


FIG. 15. Schematic of the experimental layout for transmission experiments. The layout is essentially similar for pulsed or reactor setups where the latter requires the inclusion of a chopper at the beam entry (courtesy of CNEA, CONICET, Centro Atómico Bariloche, Argentina).

These parameters depend on the flight path of the neutrons. In the minimum requirement for normal operation, two simultaneous spectra must be acquired: the first to monitor the transmission and the second to monitor the beam.

2.9.4. Sample requirements

To determine total cross sections, flat geometry samples with constant thickness are needed. The sample area must be chosen to be larger than the beam size (adjusted to the size of the sample). The sample thickness is usually chosen to provide a transmission of about 50% of the incoming neutrons to ensure proper contrast. Since this will depend on the neutron energy, the choice will have to be made as a function of the required energy range to be investigated. Typical sample thicknesses range from 1 mm in the case of hydrogenous samples to several centimetres depending on the cross sections of the sample.

The measuring program should include instrument control and movement of the sample changer and start/stop experiments. This is essential if different physical parameters are controlled. Sample environments accessories such as cryostats, pressure cells, gas handling devices and magnetic fields, can enrich the knowledge that this technique provides. The duration of an experiment varies from a few minutes to several days.

2.9.5. Data

The data must be collected in the form of histograms as the number of counts as a function of elapsed time from the pulse trigger. The data collection can be performed by a multichannel analyser, for which there is a wide variety of options available. Nowadays the usual data collection is a so called list mode, when all events are recorded together with other necessary parameters such as time stamp, coordinates of detection, etc. To determine cross sections alternative “sample in” and “sample out” measurements must be made. The spectra must be normalized by the number of counts collected in the beam monitor shown in Fig. 15.

There is no established data processing software; usually each laboratory has developed its own. The recently presented data reduction and analysis open source application Mantid [53, 54] is based on collaborative software, so each user can contribute with their own routines and share it with the community.

2.10. TIME OF FLIGHT INDIRECT GEOMETRY SPECTROSCOPY

2.10.1. Purpose

TOF inelastic neutron spectrometers are categorized in two classes: direct geometry and indirect geometry spectrometers. The former (direct geometry) utilize a monochromatized incident neutron beam (typically by either Fermi or disc choppers, see section 3.2.7), and the energy analysis is done with the TOF method, for a fixed initial energy. The latter (indirect geometry) utilizes a pulsed beam of incident neutrons with a wide energy distribution, and the energy discrimination is usually done with the crystal analyser mirrors, so scattered neutrons with a fixed energy are detected. The requirement for a pulsed beam makes the technique appropriate for accelerator based neutron sources, and not for research reactors.

The indirect geometry technique is mainly used for the high energy resolution backscattering technique, which is not appropriate for low and medium flux neutron sources. Nonetheless, a number of indirect geometry spectrometers with less demanding energy resolution (e.g. larger than 0.1 meV at the elastic position) have been built to date. The advantage of the indirect geometry technique is that a smaller number of detectors is needed. This reduces electronic and other noises, while allowing for a wide coverage of the scattering angle by using large focusing analysers. This is advantageous for medium flux sources, where the number of neutrons reaching the detectors is quite limited for inelastic scattering.

Applications of this technique include the study of molecular vibrations and condensed matter physics, such as low energy excitations in heavy fermion compounds [110], quasielastic scattering from glass forming polymers [111] and dynamical spin correlations from single crystalline quasicrystals [112]. Strong incoherent scattering from the hydrogen makes measurements of quasielastic scattering from polymers quite feasible with the indirect geometry spectrometers, and hence is one of the best applications of indirect geometry spectrometers using a cold source. On the other hand, indirect geometry spectrometers with a thermal source can reach a few hundreds of meV, and hence are suitable for the study of the

molecular vibration of organic materials, lattice vibration in the thermoelectric and/or superconducting materials and crystal electric field splitting of rare earth materials, to note a few.

2.10.2. Requirements

Table 16 shows the minimum and recommended neutron flux, beam size, required surface space for the instrument, the time and cost typically needed to develop and install the technique and the required staff to operate an inverted geometry spectrometer. Note that the values given are order of magnitude indications for typical set-ups and may vary between instruments. The development time and the cost of installation are indicative only and can be much larger for some set-ups. The human resource requirements have to be expanded when a user programme is supported, to adequately support inexperienced users with data treatment, processing and visualization. The costs and staff mentioned do not include advanced sample environments.

TABLE 16. REQUIREMENTS FOR A TYPICAL INDIRECT GEOMETRY SPECTROMETER

Instrument	Indirect geometry spectrometer
Neutron beam spectrum	Thermal, cold
Minimum count rate (n/cm^2/s)	10 counts per pulse
Recommended count rate (n/cm^2/s)	$\geq 10^2$ counts per pulse
Beam size (cm^2)	15 (typically 10 cm height, 1.5 cm width)
Required space (m×m)	3 × 3
Minimum time to develop/install technique	2–4 years
Minimum cost of installation (million USD)	1
Human resources needed for operation	1 instrument scientist 1 technician Additional instrument scientists and/or software engineer for support to users

[1] Depending on number of analysers. The approximate cost for one analyser–detector set may be around 0.1 million USD (with Be filter at ambient conditions), and 4 to 8 sets may be a good start, with future upgrades to cover a larger solid angle.

2.10.3. Instrument description

A typical indirect geometry spectrometer is schematically shown in Fig. 16. Such instruments can have a 1% to 2% dE/E resolution, which is suitable for chemical spectroscopy, as well as for condensed matter spectroscopy. By placing a number of analyser–detector sets to cover a broad range of scattering angles, indirect geometry spectrometers can be implemented as quite effective inelastic spectrometers at pulsed neutron sources. While the energy resolution is mostly fixed by the analyser angle, the energy coverage can be tuned by selecting an appropriate source (either thermal or cold). Both options have been successfully utilized.

Early realizations of indirect geometry spectrometers include the LAM-40/LAM-D/LAM-80ET setups at the medium intensity accelerator based neutron source KENS, KEK in Japan [113], and the QENS setup at the Intense Pulsed Neutron Source of the Argonne National Laboratory, USA [114]. LAM-40 at KENS had 8 analyser–detector sets, fixed on one

movable table, and covered a wide Q and energy space by rotating the table. The TOSCA spectrometer at the high performance spallation source ISIS uses a graphite analyser for the outgoing path, with a cooled Be filter, with an energy range 0–500 meV [115].

Typical incident flight path is 5 to 20 m, which can be chosen with regard to the requirement of the energy resolution. Insertion of supermirrors for the incident flight path is not a requirement, but it significantly increases the neutron flux at the sample position, in particular for the cold neutron energy range. The scattering chamber may be under vacuum to reduce the background. To obtain a higher counting efficiency, pyrolytic graphite (PG) is usually selected as analyser crystal, nonetheless mica crystals are a possible choice for high energy resolution when used with a cold source. For PG analysers, higher harmonic neutrons may be eliminated by inserting a Be filter of approximately 10 cm length, which may be cooled to liquid nitrogen temperature to obtain higher transmission. The number of analyser–detector sets may be chosen according to the available budget. Although not necessary for chemical spectroscopy applications, for condensed matter applications the analyser-detector sets may be placed on the rotation table so that continuous data in the Q space may be obtained.

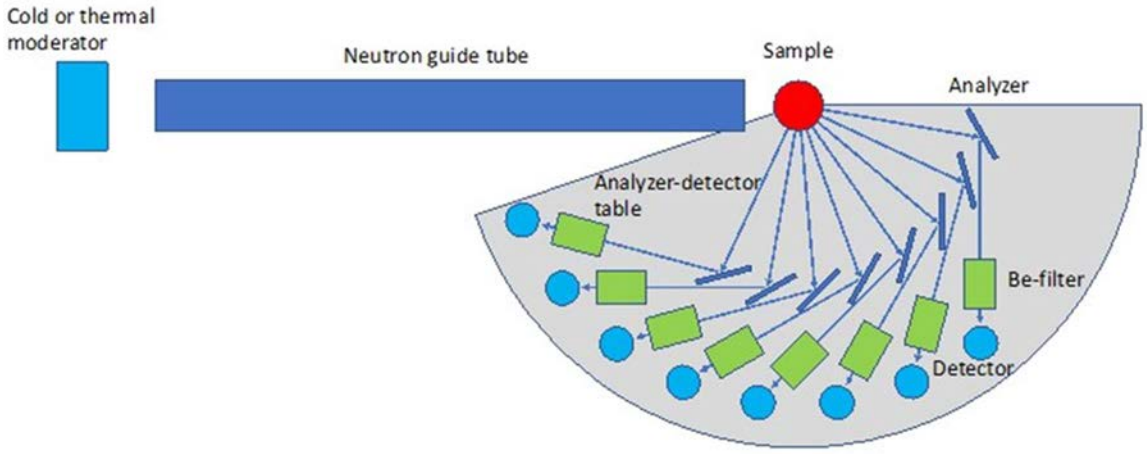


FIG. 16. Schematic of an indirect geometry spectrometer (courtesy of Tohoku University, Japan).

2.10.4. Sample requirements

Measurements can be carried out on a variety of samples, including polymers, glasses, liquids, superconductors and magnetic materials.

Typical sample size is 1.5 cm × 10 cm, depending on the moderator and beam tube size, as well as the requirement for the energy resolution.

Indirect geometry spectrometers may be used for inelastic scattering experiments using single crystals. For this purpose, a sample rotation table has to be installed.

Various sample environments may be used in combination with indirect geometry spectrometers, such as low temperature cryostats, superconducting magnets and high-temperature furnaces.

2.10.5. Data

Typically, the data are obtained in a time histogram format for each detector and are converted into the scattering function $S(Q, \hbar\omega)$ using in house software. There is a trend for

data acquisition in event list mode, and open source collaborative software such as Mantid [53, 54] may be a good future choice. Different options exist to analyse the resulting $S(Q, \hbar\omega)$ function. For example, lattice vibration (phonon density of states) results may be directly compared with the theoretical results obtain in the density functional theory calculations (using e.g. the software Quantum ESPRESSO [116]), whereas spin excitation results may be compared to the linear spin wave calculation (e.g. spinW [106]) or exact diagonalization type calculations(e.g. Hϕ [117] [ref]),, depending on the systems measured.

3. INSTRUMENTATION

This chapter presents technical information on the main neutron scattering instrumentation components. A summary of modern neutron detection technology has been presented previously [118] and is out of scope of this publication.

3.1. TECHNICAL FEASIBILITY STUDY

When initiating the development of a given spectrometer or diffractometer, a careful technical feasibility study to determine the optimized instrument performance is essential. This includes defining approximate values for the most important parameters, such as resolution and intensity, as well as establishing that the proposed configuration can be physically achieved. The analysis is intended to demonstrate that set parameters are sufficient for the intended purpose of the instrument and that the needs of the potential facility users are met.

It is advisable to contact experienced scientists from established facilities in the early stages of conceptual design to assist in setting realistic performance goals such as resolution and intensity once it has been decided what type of instrument is to be built.

It is also important to define both the national and site specific regulations and standards that may be in force at any given neutron scattering centre. These usually represent hard constraints on the engineering of the instrument. Practical requirements and standards to be considered cover aspects such a floor loading, electrical safety, pressure vessels, working with lead, seismic requirements and fire regulations. Not defining them early in the design process can require expensive redesigns or may result in refusal of authorities to have the instruments installed.

In order to turn the concept into a real instrument, engineering design studies are required. The engineering design of neutron instruments is a specialised field. The International Society of Neutron Instrument Engineers (ISNIE) was formed in 2017 to provide a forum for exchange of good practice concerning neutron scattering instrument design [119]. It acts as the organizing society for a series of regular meetings known as DENIM (Design and Engineering of Neutron Instruments Meeting) where modern development in these topics are discussed. ISNIE and DENIM represent forums where invaluable practical advice can be gained.

During the design and installation process, the choice of weakly activating materials is beneficial for disposal of components after use, and can decrease the operational costs significantly, besides improving the working conditions of technicians and scientists.

3.1.1. Design and simulation

The preliminary engineering design of an instrument is initiated through simulations of the instrument, using different methods. These can be:

Analytical. Using known and verified values of the necessary parameters, such as the energy spectrum of the neutron (and gamma) beam and the geometry for the optimal beam extraction required for the given spectrometer. The requirement of shielding, including its dimensions and weight, must be considered since the available space and floor carrying capacities may impose constraints.

Monte Carlo type. Using computer code packages (such as the Simres and Restrax package [120, 121], Mcstas [122] and Vitess [123, 124]) the calculated analytical setups for neutron optics and intensity at different parts of the spectrometer need to be verified. This also helps in the planning and choice of the necessary shielding parameters.

Other Monte Carlo codes such as MCNP [125] and FLUKA [126] are capable of calculating the shielding requirements and the scattering in the material. Some limitations in the definition of the geometry may exist, and there are important processor time requirements for the calculations.

It should be noted that in the design optimization, the entire system from the neutron source to the detector system, and not only the instrument, should be modelled.

3.1.2. Environmental conditions

The overall environmental conditions in the experimental site usually need to be controlled and kept within predefined limits, which match the usual ambient temperature and humidity in the laboratories. Temperatures in the range +15 to +30 ºC and humidity within 30 to 60% promote the prevention of condensation and corrosion and assist in keeping electric and electronic contacts in good working condition. Good electrical grounding is also needed. If these conditions cannot be maintained during operation the construction requirements and cost can increase significantly.

3.2. BEAM HANDLING

The neutrons generated in the reactor core must be transported to the instrument, often with modification of the energy spectrum and filtering of the gamma component of the beam. This is done with different components and instrument parts. These are:

3.2.1. Cold source

Cold sources are used to shift the reactor thermal neutron energy spectrum from a Maxwellian distribution with a peak at 1.3 Å (for a moderator temperature of 300 K) to a peak at 4–5 Å (corresponding to a cold source temperature of 30–60 K depending on the moderation level [127]. Such neutron wavelengths are essential to conduct studies of phenomena in the order of 1000 Å which are observed at low Q scattering.

Commonly used cold sources usually contain cold liquid or supercritical hydrogen or organic compounds with high hydrogen content (such as methane or mesitylene). Some accelerator based neutron sources (and some research reactors) have solid methane cold moderators. Due to the optimization for maximal output of cold neutrons, the shape of cold neutron sources, which have ellipsoidal form in many setups, has now been modified to cavernous or tubular forms (see Fig. 17). Other materials and designs are being actively investigated.

Large liquid-hydrogen cold sources serving multiple beam tubes and instruments that are placed in core at a research reactor can be quite expensive and require extensive engineering expertise and staff to operate. An option for smaller sources includes the use of closed cycle refrigerators serving moderator materials such as mesitylene, noting on the one hand that radiation damage remains an issue, and on the other hand that they have fewer security and safety issues than cold neutron sources shared by several instruments at research reactors. These have been implemented in the past at some university research reactors [128, 129].

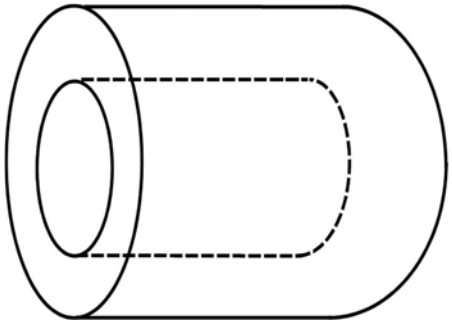


FIG. 17. The upgraded form of cold sources resulting in higher intensity (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

3.2.2. Hot source

Hot sources are used to increase the neutron energy, usually for high Q diffraction. Hot sources are usually made from thermo isolated graphite, which is heated by the gamma radiation generated in the fission process. The temperature of the graphite can reach well over 2000 K, resulting in a shift of the maximum of the Maxwell energy distribution from 1.3 Å to 0.3–0.5 Å, accompanied by a broadening of the neutron flux distribution, which leads to a reduction of the flux at any given energy. Hot sources exist in a limited number of high performance research reactors and are not usually found at low or medium flux neutron sources.

3.2.3. Filters

Filters of fast neutrons are liquid nitrogen cooled Si or quartz (SiO_2) single crystals, or single crystals of sapphire (Al_2O_3) or MgO at ambient conditions. These filters, with typical length around 100–200 mm, are installed in the primary beam between the reactor core and the monochromator. They predominantly reduce the fast neutron component in the beam, by one to two orders of magnitude, while the thermal component is decreased by only 10–20 %.

The gamma contamination of the beam can largely be reduced with filters such as liquid nitrogen cooled Bi single crystals. The typical length is 100–150 mm, reducing the gamma intensity by around two orders of magnitude. Similarly, the insertion of a Be filter limits the transmission of neutrons to $\lambda > 4$ Å. Since filters also reduce the beneficial neutron flux, their length is a compromise between filtering and transmission requirements.

It is important to note that these filters scatter neutrons or gamma rays out of the primary beam, rather than absorbing the unwanted radiation. Therefore, the filters need to be surrounded by substantial shielding. For this purpose, a fast neutron filter is usually placed in the beam collimator or plug inside the biological shielding of the neutron source, and the bismuth is near the beam port where cooling with liquid nitrogen can be more easily achieved.

The presence of higher order wavelengths in monochromatic neutron beams is suppressed by the insertion of a filter in the beam between the monochromator and the sample. An often used filter is Pb. In longer wavelength neutron powder instruments that typically utilise 2.41 Å neutrons, HOPG is a very efficient and convenient filter solution requiring minimal additional shielding.

3.2.4. Neutron guide

Neutron guides, similarly to optical cables for light, can guide neutrons using the feature of total reflection from their internal surface. The neutron guides are under vacuum to reduce the scattering of neutrons by the air, considering that typically 10% of 4 Å neutrons are scattered per 1 m of air at ambient conditions. The critical angle for total internal reflection of 1 Å neutrons for natural Ni coating is 6º and increases linearly with wavelength. Using a specially manufactured NiTi multilayer system, the critical angle can be typically extended by a factor 3 to 4 [130, 131].

An important feature is that the beam divergence increases with the wavelength. Using a curved neutron guide, the fast neutron and gamma component of the beam is reduced since it cannot follow the guide direction, leading to a beam with substantially lower fast neutron and gamma contaminations. Using specially designed forms (such as elliptic, parabolic, hyperbolic), the beam can be focussed and filtered (see section 3.7.3.3).

When possible and available resources permit, it is advisable to have a dedicated neutron guide for each instrument, in order to have a clean beam at the instrument. Otherwise, the neutron spectrum at the instrument may not be constant due to the extraction of monochromatic beams for other instruments located upstream in the same beam line, i.e. closer to the source.

3.2.5. Monochromators

A monochromator is a device that facilitates selecting and directing a neutron beam of a specific wavelength from the incident spectrum. It selectively extracts a narrow wavelength band from the neutron spectrum. The acquired neutron wavelength λ is related to the crystal planes spacing d, and reflected beam direction, 2θ, relative to incident beam by the Bragg Equation (1), from which it can be deduced that the angular spread $\Delta\theta$ of the incident and diffracted beam results in a divergence of wavelength $\Delta\lambda$:

$$\Delta\lambda = 2d \cos \Delta\theta = (\lambda \cot \theta)\Delta\theta \quad (6)$$

With a monochromator perfect crystal, the acceptance of angular spread is small and the corresponding diffracted neutron beam intensity is also low. In practice, monochromator crystals are normally made with deliberate mosaic structure. These consist of small perfect crystal blocks slightly misoriented from each other. This geometry provides a considerable gain in diffracted intensity, but at the cost of angular and wavelength resolution. The mosaicity can be increased by plastic deformation, either permanently or by mechanically controlled elastic bending, that enhances the diffracted beam intensity by delivering diffracted neutrons with a broader band of wavelengths. The wavelength band width is a trade off between the intensity and resolution of the diffractometer.

Monochromators are arranged in two dimensional structures, which focus the beam horizontally and vertically. While the vertical beam divergence has little influence on resolution, the horizontal beam divergence directly controls the resolution [132, 133]. Therefore, the intensity of an incident neutron beam can be enhanced with vertical focusing, that is easily achieved by mechanically orientating strips of monochromator crystals mounted side by side vertically using a shaped backing plate, or cams. Horizontal focusing is achieved by in situ elastic bending of the monochromator. This is easily achieved with Si and Ge crystals, preferably thin wafers, but is not possible for more rigid materials such as HOPG or

Heussler crystals. Additional combined gain in resolution as well as intensity can be achieved simultaneously with vertical and horizontal focusing by assembling the elastically bent individual crystals in the horizontal plane and mounting them vertically on a flexible backing plate.

The most commonly used monochromator materials are HOPG, Si, Ge, Cu, with Zn also used in older designs. The typical wavelength distribution $\Delta\lambda/\lambda$ of crystal monochromators is in the 1–5% range [134–136].

Since these monochromators substantially change the beam direction, they have consequences on the instrument geometry and background. If the change is not desirable, a double monochromator system can be used.

Monochromators reflect only around 1% of the total flux coming from the neutron beam tube. Therefore, the monochromator cavity must be shielded from fast neutrons and γ rays.

Easy to find inexpensive natural monochromators that can be used for demonstration and E&T purposes are, for instance, silica crystals, fluorine rich mica, beryl $(10\bar{1}0)$, calcite $(10\bar{1}1)$, hematite (0002), magnetite (111) and quartz $(10\bar{1}0)$.

The analysers used in TAS (see section 2.8) are, essentially, monochromators and the considerations given in this section apply. They need less shielding and do not need to be transparent to neutrons, so they can be made also of materials such as Ge.

3.2.6. Velocity selector

Some instruments do not require strong beam monochromatisation. This is the case with techniques such as SANS and Spin Echo, for which the typical requirement is 10–15%, which can be easily achieved by using a velocity selector. The velocity selector has helical channels rotating at a given speed (see Fig. 18), enabling transmission of specific neutron wavelengths. This helical system can be built from specially arranged disks having holes or slits for neutron transmission with the regions between the holes or slits covered with neutron absorbers. Only neutrons with velocity matching the helical and rotation speed (typically 2000–12000 rpm) can pass through. The transmitted neutron wavelength package therefore depends on the rotation speed, which is adjustable [137, 138].

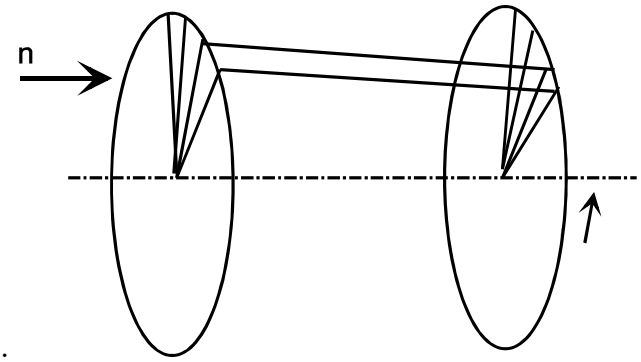


FIG. 18. Schematic diagram of a mechanical velocity selector (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

3.2.7. Chopper

Neutron velocity depends linearly on wavelength. Thermal neutrons have an average speed of 2200 m/s. Choppers are used to interrupt the beam periodically, creating neutron bunches. A typical bunch has a duration around 10–100 μs, achievable with chopper rotation speed up to 12000 rpm at 80 cm diameter [139].

The chopped beam will contain neutrons with different wavelengths, which therefore travel at different speeds, so they will spread out along the flight path. The TOF technique serves to determine such velocities.

Another possibility to make a neutron pulse is the so called Fermi chopper, which is a cylinder that rotates around the vertical axis and has a slit where neutrons of a certain compatible speed can go through. The slit is divided in smaller straight or curved channels to decrease the beam divergence. The curvature radius and the rotating speed determine the transmitted neutron speed/wavelength. The typical rotation speed is 100000 rpm.

A group of choppers can be used to decrease the background, control the resolution and avoid frame overlap of pulses generated by the first chopper.

3.2.8. Shutter

The beam shutter is an essential safety component of any instrument, with the function to interrupt (close) the neutron beam to reduce its intensity outside the biological shielding of the reactor to acceptable dose levels that permit safe access to the instrumental parts, or the sample and its environment. Shutters typically have a length along the beam ranging from 0.5 to 1.5 m.

3.3. INSTRUMENT PARTS

3.3.1. Movement components

Most instruments have different moving mechanical parts. That includes air cushion systems for moving the whole instrument, actuators and rotators moving with typical linear and rotational accuracies of 0.01 mm and 0.01º, respectively. This requires the incorporation of encoders or other optical devices to accurately measure movements. Take note that in high radiation fields (typically around the neutron beam, including the goniometer and sample movement) optical sensors are usually not employed due to the risk of damage. Therefore, mechanical encoders or resolvers are typically used.

When high magnetic fields are necessary, or in measurements using polarized neutrons (such as in reflectometry, spin echo, polarized SANS, and in some cases also in techniques such as powder diffraction or triple axis spectroscopy), it is useful to use piezoelectric motors and attenuators. The additional force from stray magnetic fields can cause motors to stall. In addition, the structural and shielding elements of the instruments should not be magnetic. Structural features in the facility, e.g. rebar in the floor, may also cause problems. In the presence of an external field, a magnetic shield (e.g. of mu metal) also needs to be incorporated. Shielded magnets that eliminate stray fields are a cheaper alternative that may be considered with advantage, particularly in the design of new instruments [140].

3.3.2. Collimators and slits

Modern scattering instrumentation typically uses collimated beams. It is possible to create collimators to be two direction radial collimators using modern mechanical machining. Collimators placed in the direct beam before the sample needs to be manufactured from radiation resistant materials. In such cases, plastics such as polyester (e.g. biaxially oriented polyethylene terephthalate), polyimide or chlorine and fluorine containing plastics should be avoided, as their structure can be damaged or activated at relatively low neutron fluences (e.g. 10^{11} n/cm^2). Radiation resistant collimators are often made of Boral, have blades coated with Cd, or employ metal sheets covered with B_4C.

Slits are important element for mapping studies in inhomogeneous samples. They are used to localise investigations to specific regions of interest in samples (for example gauge volume in stress measurement) and screen scattering from unwanted regions in samples and sample environment containers. Slit materials are high neutron absorbing materials such as Cd, Gd, ^{10}B and ^{6}Li for thermal neutron beams.

3.3.3. Detectors

For thermal neutron detection, single detectors, linear 1D position sensitive detectors and 2D position sensitive detectors can be used. Gas filled ^{3}He or BF_3 detectors with charge division or delay line read out electronics are commonly used. These detectors have high detection efficiency, are stable, have low electronic noise, have excellent gamma discrimination and good timing. Unfortunately, the ^{3}He isotope has become expensive. As an alternative, a ^{10}B coated plate system has been recently developed [141], which is sensitive for thermal neutrons and can also be expensive.

Fibre detectors exist using a ^{6}LiF/ZnS:Ag scintillator screen have higher gamma sensitivity than gas filled tube detectors.

For fast neutron detection, scintillation detectors sensitive to neutrons (Li glass) or proton recoil detectors are used.

For gamma measurements, Ge–Li and Bismuth germanate (BGO) detectors are used. In rare cases, NaI(Tl) detectors are still used. Detectors based on Lu_2SiO_5:Ce have been developed as promising scintillators due to their desirable properties such as high density, fast decay time and high light output.

The recently developed Gd_2SiO_5:Ce scintillator systems have advantages, such as having higher *Z*, faster decay time than NaI(Tl), higher light output than BGO, higher radiation hardness and less radio activation than most of the known scintillators. Furthermore, they are non-hygroscopic and can be cut and polished easily.

For beam distribution, films, or more recently charge coupled device camera and converters are used. Spatial resolution around some tens of μm can usually be achieved.

3.3.4. Beam stop

The beam stop is used to eliminate the direct beam after the sample position. In addition to beam absorption, it needs to minimise backscattering of the incoming beam. Typically used materials are boron containing plastics and iron and lead. The components can be adjusted to

the expected neutron and gamma spectra of the beam. One further request for the materials is low activation or short time activation (days).

3.4. SHIELDING

3.4.1. Neutron rigid shielding

Rigid neutron shielding is usually made of concrete containing boron (B_4C), ^6Li, H_3BO_3, B_2O_3 in plastic (polyethylene), Boral, Cd, Gd, H containing material (paraffin, polyethylene, polypropylene, wood), Fe and ZrH_2 (for high temperature use). A requirement is that materials should not become activated, except for short half-life or prompt gamma. Use of ^6Li may generate neutrons, which also must be considered.

Recently, a reusable shielding material was developed for neutron and gamma radiation that is composed of steel granules, liquid paraffin and ferroboron, with higher attenuation than commonly used heavy concrete [142]. The material does not solidify and is poured in a container. It can thus be reused if the shielding has to be modified, or for shielding of a new facility, also reducing the need for disposal of old shielding, which is often costly.

In some facilities, due to fire regulations, it may not be permitted to use large quantities of hydrocarbons as neutron shielding. Some alternatives have been developed that are non-flammable, including water extended polyester [143, 144].

3.4.2. Neutron flexible shielding

Flexible, elastic materials such as B_4C and plastic polimethylsiloxane (silicon rubber/glue) are radiation resistant. This is used for covering the internal part of the vacuum chamber of SANS instruments and other experimental installations such as PGA sample chambers. Inexpensive alternatives such as boraffin, consisting of a mixture of boron carbide and paraffin can be effective [145].

In choppers or slit systems the ^{10}B isotope is used. A ^6Li containing rubber is also used for special slit and shielding system for PGA, or for some other (n,γ) measurements (e.g. neutron holography).

3.4.3. Gamma shielding

Gamma radiation is shielded with heavy elements such as Pb, Bi or concrete. The decrease of the high gamma radiation background is an essential requirement in many neutron scattering techniques. In some cases, the neutron detectors also have substantial sensitivity to gamma rays, and thus require substantial gamma shielding. It is important to note that neutron and gamma radiation occur together. Shielding materials where (n, gamma) reactions occur need to be avoided (for example, if Pb or W are used, Sb cannot be present).

Use of rare earth elements such as Gd, Cd, Eu, Sm, etc. in instruments (such as velocity selectors) may lead to generating a hard gamma background. Such instrumental components need to be adequately shielded. In order to maintain a polarized neutron beam, a guide field is usually required, often involving high field permanent magnets (see section 3.7.5). Judicious choice of magnetic may be required in high neutron fluxes as either the rare earths or other components like Co can become strongly activated.

3.5. SAMPLE ENVIRONMENT

The sample environment is usually controlled to establish different temperatures, pressure, magnetic fields and applied mechanical stress or pressure in samples. This section indicates what are the most general sample environments used for in-situ studies. High performance instruments may have further requirements.

Temperature sample environments can be attained with: Cryostats with range 77–4 K, closed cycle refrigerator and furnaces with range 1–2000 K, with different constructions; thermostats for slightly decreased or elevated temperature from 220 to 570 K. Other sample environment parameters can be light, moisture, magnetic field (up to 2 T, usually in 3 directions, although higher fields are available in some facilities), pressure cells of different constructions, stress rigs and even chemical reactors. For the sample, an adequate sample holder is needed. It should be transparent to neutrons and it should not affect the state of the sample, isolating it from other components of the environment.

In the last decades, technological advances have improved the performance of sample environments for in-situ neutron scattering investigations. In cryogenics the use of liquid nitrogen and helium has been substantially reduced, being replaced by modern closed cycle refrigerators that enable reaching sample temperatures substantially lower that 4 K upwards to even 800 K in cryofurnaces. Combining this with the convenience of sample top loading via a "stick", the refrigerator remains at base temperature during sample changes [146–148]. Similarly, ^{3}He sorption fridges exist as closed cycle systems that can bring samples to a few hundred mK with long hold times. Such closed cycle systems substantially reduce the operational costs in the long term compared to liquid helium. In addition, cooling systems based on adiabatic demagnetization can reach temperatures down to 100 mK, avoiding the use of cryogens.

Split-coil magnets based on cryogen-free technology are also available that facilitate fields up to 12 T and can be configured to have fields in the horizontal or vertical planes.

Another significant development has been the large volume Paris-Edinburgh cell for neutron scattering [149, 150]. These cells substantially increase the attainable pressures to several GPa for in-situ studies of materials.

Finally, one should note that instrumentation for establishment of sample environments are specialised and subsequently often expanded and developed in house by dedicated experienced groups. This often reduces costs while allowing the development of specialized systems that cannot be obtained commercially. An international consortium, the International Society for Sample Environments, provides information and support on this subject through regular Workshops and online Forums [151].

3.6. DATA ACQUISITION, TREATMENT AND ANALYSIS

Large neutron centres, with established user communities, use a variety of systems. Small reactors have fewer requirements for complex data acquisition software and hardware, but there is often some similarity at the level of individual instruments. An important feature is the capability of visualisation of the data.

Different facilities and data analysis software packages use a variety of data formats. This is a barrier to mobility and to collaboration between users from the low and medium flux neutron

sources and from the large high performance facilities. The shift towards increased efficacy in research data management as evidenced by the formalisms of "Findable, accessible, interoperable and reusable" (FAIR) data proposed by Wilkinson et al. [152, 153] and the developing concepts around open data as seen by the development of the European Open Science Cloud [153] have catalysed many collaborative efforts to develop and deploy common data formats and common scientific software systems specifically for neutron scattering experiments. Projects such as PaNOSC [154] and EXPANDS [155] build upon the existing excellence in research data management that has been developed at neutron facilities over many years, much of which was pioneered at national neutron centres in Europe and North America. The current overall objective is to build FAIR concepts into the facility data pipeline and process.

The scientifically diverse user community that surrounds neutron facilities have for many years worked towards methods to increase reproducibility and reliability of data. Many scientific communities synergistically use neutron and photon scattering, and the diversity of the scientific user community has produced many niche analysis applications. The resulting increase in experimental complexity and flux has necessitated a move from simple human readable data formats. The development of these complimentary research programmes have prompted a move to a standard data format. Currently many neutron facilities are moving to deploy a standardised data container that utilises the NeXUS format [156] to codify and store experiment raw data and experiment metadata. Nine large scale neutron facilities worldwide utilise NeXUS as the facility data format.

In addition to facility standardization, a number of neutron techniques have specific domain standards for research data. The small angle scattering community utilise canSAS [157] which provides an interoperable data format for small angle scattering data. The crystallography community, many of whom are neutron scatterers, use the standard developed by the Crystallographic Information File (CIF) format developed by the International Union of Crystallography [158, 159]. The benefits of standardisation in research data formats provides the foundation for the development of common and interoperable open source data services and applications. The Mantid project [53, 54] and the SASView project [67] are good examples of scientific software that is based on standard data formats and provides interoperable data treatment and analysis.

While these initiatives originate in large scale user facilities, they will also benefit the users from the low and medium flux neutron sources.

3.7. OPTIONAL ELEMENTS

Many of the components and systems described in this section are not usually found in low and medium flux neutron sources, as they are more common in high performance neutron sources.

3.7.1. Vacuum systems

The neutron beam tubes are usually under vacuum because of the relatively high scattering of the beam by air. Primary vacuum (10^{-3} bar) is usually necessary and sufficient to fulfil this request. Cooling systems and sample environments may require higher vacuum, around 10^{-4} to 10^{-5} bar, and rarely even better. The detectors are often also under vacuum. TAS usually operates in ambient conditions and diffractometers often use He filling instead of vacuum.

3.7.2. Neutron lenses

The refractive index n of neutrons is less than 1 for most materials, with $n=1-(\lambda^2 Nb)/2\pi)$, where N is the number of atoms in 1 cm^3 and b is the scattering length of the atom (see section 2.6.2). In this case, a concave lens is convergent while a convex lens is divergent. When other effects such as absorption, activation and incoherent scattering are low, good refractive optical elements can be manufactured. Commonly used materials are MgF_2, Si and graphite. Using biconcave MgF_2 lenses with curvature R the focusing distance is $f_o = (R/\rho b_c)\,(\pi/\lambda^2)$, where ρ is the atomic density, b_c the bound coherent scattering length of an atom and λ the wavelength of incident neutrons. Consequently, these lenses have strong dispersion dependence with wavelength [160–162].

These systems, commonly used in SANS, are still under development in other areas, where they are expected to lead to improvements in the beam intensity.

3.7.3. Mirror systems

Optimization of mirror systems needs to take into account that neutron sources are not point sources, but instead have a finite size.

3.7.3.1. Supermirror

In materials where the refractive index of neutrons is less than 1, the neutrons undergo total reflection below a certain critical angle. A multilayer device consisting of bilayers of two materials that have strong neutron scattering length contrast density creates high reflectivity Bragg peaks even beyond the critical angle. Neutron beam guides based on such multilayers are used as optical components to transport and direct the neutron beam. The commonly used multilayer materials are Ni and Ti, because their scattering length densities have high magnitude and opposite sign and they are easy to deposit [163].

In supermirrors, non-periodic multilayer devices of hundreds of bilayers are used. In these multilayer systems, the thickness of a bilayer gradually increases from the substrate to the top of the device. A supermirror can be imagined as a stack of several multilayers having their individual Bragg peaks with positions that vary continuously, leading to closely spaced superimposed Bragg peaks. These peaks extend the critical angle for total reflection as compared to a single layer film. The performance of the supermirror is generally described by its '*m* value' which is defined as the ratio between the critical angle of total external reflection of the supermirror and the critical angle of natural Ni. Different alternatives with different gains are available and can be obtained. The most advanced can have a very high cost: from 2*m* (i.e. two times the critical angle of natural Ni layer, which are still relatively inexpensive), to 3*m* and even 4–7*m* (requiring around 1000 multilayers) supermirrors.

3.7.3.2. Mirror benders

When curved neutron guides are used (see section 3.4.2), there is an important feature, namely the existence or absence of direct view through the guide. When a direct view at the end of the guide exists, the beam is still contaminated with fast neutrons and gamma radiation. The lateral size of the guide is specified by the requirements of the instrument. So, when possible, the length of the neutron guide is chosen so as to exclude direct visibility. In such cases, usually the lateral deflection is twice the guide width. When such length is not possible due to geometric constraints, a neutron bender can be used, which in practical terms

is a laterally divided neutron guide. In the benders, the lamellas (i.e. vertical mirrors splitting into parts the original width of the guide) are covered by a multilayer mirror, and underneath they are covered with an absorbing (GdO) layer. Consequently, the lamella thickness is as small as possible. The manufacture of such benders is technically demanding and they are relatively expensive. In these benders the possibility of individual adjusting of elements, that exists in neutron guides, is not available [130, 131].

3.7.3.3. Elliptic guides

Supermirror coatings with index m up to $m = 7$ have been constructed, which extends their capability to transport epithermal neutrons with wavelength reaching 1 Å. However, in straight neutron guides this results in the existence of many neutron reflections in the multilayers. As each reflection leads to a reduction in the intensity, the transmission in such straight supermirrors is strongly decreased [164].

Using bent elliptic neutron guides, the number of reflections is essentially reduced to two. These guides focus the neutrons from the moderator exit to the sample from the point to point imaging provided by an ellipse. This can raise the flux at the sample by one order of magnitude.

In such elliptic guides there is a direct line of sight between the sample and the moderator, which can lead to a large background at the sample. This disadvantaged can be mitigated by introducing beam blockers in the central part of the guide. Also, if a succession of several elliptic guides is used, beam blockers are not needed [164].

In the case of elliptic guides, the different mirror elements have different m multiplicity (the highest is located in the middle of the guide), which can reduce the cost substantially when compared to supermirror systems.

3.7.3.4. Montel mirrors

Montel guides were developed for focusing X rays [165] and are commonly found in many beamlines at synchrotron light sources. A Montel mirror consists of two elliptical mirrors adjacent to each other (see Fig. 19), arranged in an L-shape perpendicular configuration. Montel guides have many advantages and are also used in neutron optics [137, 164].

3.7.3.5. Kirkpatrick-Baez mirrors

These mirrors were developed largely at synchrotron sources. A pair of bent mirrors can yield one doubly focussed (vertical, horizontal) beam of neutrons with a spot size of several tens of microns. These have been applied as a focusing method for using pressure cells with quasi-Laue diffraction [166].

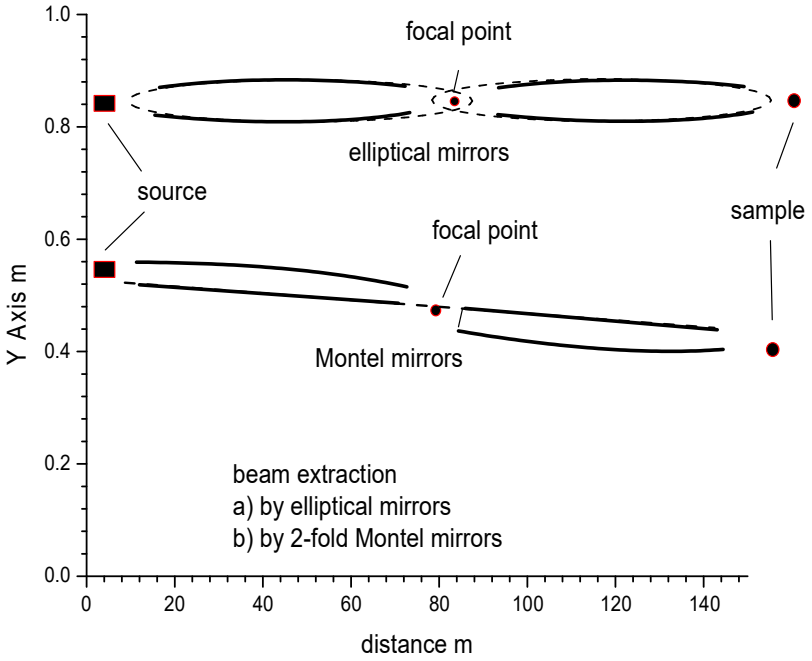


FIG. 19: The different extraction methods and guide designs. a) double elliptic guide, b) the two fold Montel guides (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

3.7.4. Field gradient optics, quadrupole and hexapole magnets

Neutrons can be accelerated by a magnetic field gradient due to their magnetic moment. Quadrupole magnetic fields thus provide a suitable optical potential gradient to separate the spin states in a beam. This is the well known Stern–Gerlach effect, and it provides a useful method of measuring the absolute polarization of a linearly polarized beam, by physically separating the two polarization states into two detectors.

Applying a hexapole magnet, the field gradient has a cylindrical symmetry and linearly decreasing toward the centre. That magnetic field is able to focus the neutrons. A hexapole field can focus one spin state of the beam onto a focal plane with full polarization, while the other spin state will be scattered out. Hexapoles have not recently been applied as polarizers, because the magnets required for typical beams (around 30 mm diameter) need a field strength of 80000 T/m^2 field, which is close to the technology limits presently available. The hexapole focusing is able to focus the beam to some square mm.

3.7.5. Polarized neutrons

There are four main (passive) methods of beam polarization, each with specific advantages in particular experimental situations [167]:

1. Polarizing crystals (e.g. $Co_{92}Fe_8$, Heusler (Cu_2MnAl) crystals using preferential Bragg reflection);
2. Polarizing mirrors and supermirrors (using preferential reflection);
3. Polarizing filters (e.g. preferential absorption by polarized ^3He nuclei);
4. Polarizing with polarized proton target.

3.7.5.1. Single crystal polarizers

In materials with magnetic ordering, certain Bragg reflections produce polarized neutrons due to nuclear magnetic interference (i.e., diffraction due to the ordered magnetic moments). The most commonly used polarizer single crystals are given in Table 17. These crystals can be used to produce polarized and monochromatic neutron beams and to analyse the energy and polarization of neutron beams, and are used in diffractometers and TAS [168]. Recently, the quality and the reflectivity of Heussler monochromators have been improved, allowing to use these crystals at relatively short neutron wavelengths.

Polarizing single crystals and thin films have decreasing efficiency as the neutron energy increases and they can only accept a low angular divergence of the beam. On the other hand, their maintenance is simple.

TABLE 17. THE MOST COMMON POLARIZER CRYSTALS

	$Co_{92}Fe_8$	Cu_2MnAl (Heussler)	Fe_3Si
Matched reflection	(200)	(111)	(111)
d spacing (Å)	1.76	3.43	3.27
2θ at λ=1Å	33.1	16.7	17.6
Maximum λ (Å)	3.5	6.9	6.5

3.7.5.2. Polarizing mirrors and supermirrors

The neutron reflectivity depends not only on the nuclear scattering length but contains a spin dependent component also. Thus, the spin dependent refractive index of magnetised mirrors for neutrons of wavelength λ is: $n\pm = 1 - \lambda^2(Nb/2\pi \pm B\mu \cdot m_n/h^2)$, where b is the mean coherent nuclear scattering length, N is the number density of scattering nuclei, B is the magnetic flux density applied in the plane of the surface, m_n is the neutron mass, μ is the neutron magnetic moment and h is Plank's constant.

The critical angle of reflection (defined when $\theta_2 = 0$) is $n_{1,2} = \cos\theta_c \approx 1 - \theta_c^2$. Between these two critical angles (θ_2 and θ_c) the reflected beam is effectively fully polarized, and in some circumstances the critical angle for one spin state can be made zero. The critical angle is usually small (typically 10 arc minutes) [130, 131].

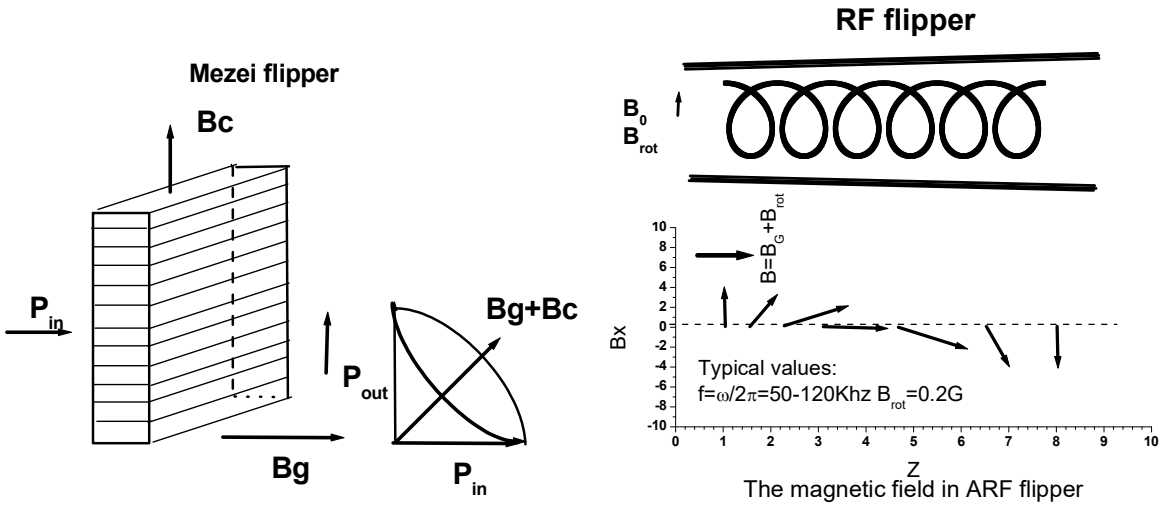
3.7.5.3. Polarizing filters

Polarizing filters, for instance ^{3}He spin filters, having a broadband characteristic and high divergence are of particular interest in spectrometers that have large area detectors [169].

The absorption cross sections of nuclei depend on their polarization and the neutron spin direction where σ_a is the mean absorption cross section and P_N is the nuclear polarization. For fully polarized ^{3}He ($P_{He} = 1$), one spin state goes through the filter with zero absorption. The other spin state is almost fully absorbed since $\sigma_a = 5000$ barn. In that way, fully polarized ^{3}He can filter out a given spin state.

3.7.5.4. Changing of polarization

In some instruments, it is necessary to change the spin polarization. Different types of neutron spin rotators and flippers exist. The most widely used spin flippers are the Mezei flipper and the radiofrequency adiabatic resonance flipper (see Fig. 20) [170, 171].



a) b)

FIG. 20. a) Mezei flipper. b) Radiofrequency adiabatic resonance flipper. (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

In a Mezei flipper, the neutrons having a longitudinal polarization and going through to the magnetic field of the coil change their polarization due to the magnetic field of the coil, by Larmor precession (see Fig. 20a). Neutrons having vertical polarisation and going through on a RF field that has the same frequency as the Larmor precession frequency in the vertical magnetic field are flipped due to resonance (see Fig. 20b). The Mezei flipper has a rather simple performance but some materials in the beam cause extra scattering. The radiofrequency flipper does not have any material in the beam and the wavelength region is broader, but it is more costly.

3.8. MAINTENANCE

The instrument components need periodic maintenance. When procuring instrument parts, it is important to obtain and record expected lifetime, required maintenance and its periodicity, as well as other factors influencing the quality and service readiness.

Modern industrial mechanical attenuators and air cushion systems usually require minimum maintenance, if the environmental conditions in the experimental hall are within the advised temperature and relative humidity ranges. Rotation components usually require ball bearing maintenance every 2000 working hours. The electronic components also need adequate ambient temperature and humidity. Special attention is needed for the high voltage parts of electronics, to avoid micro arcs due to humidity, powder containment of air and dirt in the insulator surfaces.

The instruments should have a quality management system that records and analysis non-conformities, leading to early diagnose of potential issues and necessary preventive maintenance. Such procedures help to reduce the service cost.

Special attention needs to be given to radioactive contamination and activation of parts of the instrument, particularly those positioned in and close to the beam.

3.9. DEVELOPMENT OF COMPONENTS

Low and medium flux neutron sources play an important role in performing test experiments for large facilities, to train and educate technical and scientific staff as well as students and in the development of instrument components. Testing new ideas in the development of instrumentation is a cost effective method before transferring new methods to large user facilities. In this section, some examples of such pioneering work are discussed.

3.9.1. Shaping of source

In new accelerator based neutron sources and small research reactor sources, defining the geometrical shape of the source plays an important role in technological performance and optimisation. In research reactor based sources the shaping of cold and hot sources is possible, but changing their form and size can affect the reactor characteristics and requires careful analysis. In newly designed installations, a double (thermal and cold) source may be used, providing a broader spectrum of neutrons with energy ranging from the thermal to the cold region.

3.9.2. 2D Soller and divergent (2D radial) collimators

Technical mechanical development beyond the traditional Soller collimators consisting of thin plastic or metal foil covered by neutron absorbers (GdO, B_4C) enables the fabrication of 2D shaping collimators in rectangular or hexagonal form. This can find applications in multi beam experiments in SANS or in other instruments that use divergent beams.

3.9.3. Neutron optics

The neutron optical mirror system can only be built if the angle from the plane of reflection is very low (typically from 6 arc minutes to 1 degree). These are so called grazing incidence mirrors. In the Wolter mirrors system, it is possible to create a telescope with a wide field of view. Such a system is already used in beam shaping for PGA and is proposed for use in neutron radiography and SANS as well [161, 162]. The system of such type of mirror arrangement is shown in Fig. 21.

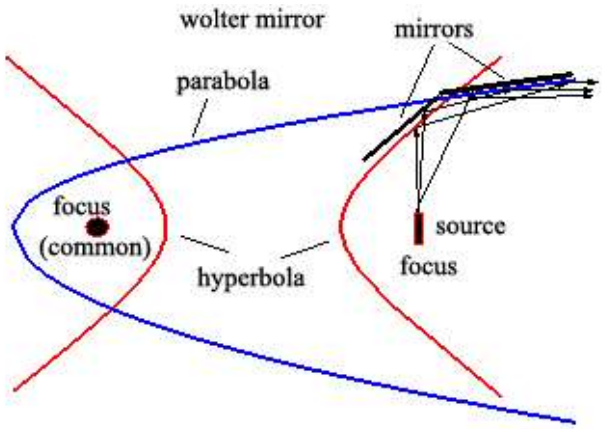


FIG. 21. The Wolter focusing system with common focal point of parabolic and hyperbolic mirror (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

3.9.4. Samples for standardization

For many types of instruments, the instrument performance can be characterized using standard samples. The standardization with certified samples enables simpler communication between different communities and assists in promoting neutron scattering results to other communities. Low and medium flux neutron sources can be used to develop such samples.

Standard samples commonly used are, for instance: Al_2O_3, Ni powder and $Na_2Ca_3Al_2F_{14}$ for diffraction measurements; certified ring and plug samples for residual stress measurement certification; vanadium sample for determination of triple axis spectrometers resolution; silver behenate and vanadium for SANS instruments; polystyrene spheres for verifying the resolution and Bragg mirrors (standard equal Ni–Ti multilayers) for reflectometry.

3.10. INSTRUMENT OPTIMIZATION

In this section, a very brief overview is made of techniques intended to optimize the performance of some neutron scattering instruments, including a guideline on how to upgrade or judge the instrument quality.

Small research reactors usually have beams of relatively large section. This comparative advantage can be leveraged by using well designed beam optics to build competitive instruments. As was noted in section 3.1.1, the entire system from the neutron source to the detector should ideally be considered at the design stage.

3.10.1. Resolution and brightness

The instrument resolution and brightness of neutron scattering instruments depend on the geometrical parameters shown in Fig. 22. They can be optimised by design or changing the geometry in accordance to the purpose of the instruments. For this, it must be taken into account that phase space conservation leads to $A_m\, d\Omega_m \sim A_s\, d\Omega_s$, (see Fig. 22 for definition of the symbols), and $d\Omega_s$ must be limited to ~1° for most scattering techniques.

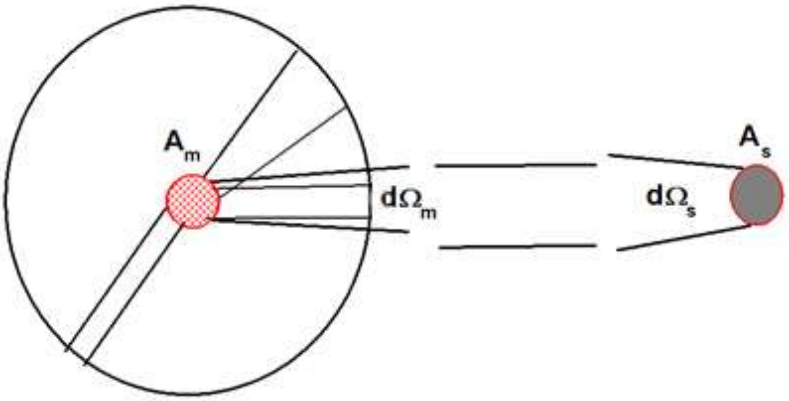


FIG. 22. Typical geometry of a scattering instrument. A_m: moderator area; A_s: sample area; $d\Omega_m$: solid angle accepted by the optics; $d\Omega_s$: solid angle usable by the instrument; $\Delta\lambda/\lambda$: wavelength relative uncertainty (courtesy of G. Török, Centre for Energy Research, Hungarian Academy of Sciences, Hungary).

3.10.2. Intensity gain

Possible methods to increase the intensity of the beam at the sample are:

1. Increase $d\Omega_m$ (5–10°) (corresponding to A_m ~ 15–30 cm); this is possible in many cases. The gain in intensity will depend on the optics of the system;
2.a. Implement a large illumination area (e.g. 20–30 cm) and lower divergence (0.5 –1°);
2.b. Implement a small illumination area (2–3 cm) and very high divergence (5–10°);
3. Adjust $\Delta\lambda/\lambda$ to the requirement of the given measurement. i.e. 1–5% for diffraction, 10–20% for SANS.

3.10.3. Some examples of application

3.10.3.1. SANS

Using focusing techniques, the $d\Omega_m$ solid angle accepted by the optics with collimation and the $d\Omega_s$ solid angle usable by the instrument can be optimized. In this case, the A_s sample size can be larger. For example, instead of using a parallel beam determined by pinholes, a convergent beam focussed to the detector surface can be used, thus increasing $d\Omega_m$. In such a setup, the total flux on the sample and consequently the scattered total intensity is higher [172–174]. Larger area detectors lead to an increase of $d\Omega_s$.

3.10.3.2. Transmission diffraction

The $d\Omega_s$ solid angle usable by the instrument can be increased. The sample size does not impact the resolution and usually A_s is small. The efficiency of the technique has to be evaluated with respect to standard powder scattering, for which the $d\Omega_s$ is 0.3–1° in the horizontal direction and some degrees in the vertical direction.

3.11. OUTLOOK ON COMPACT SOURCES

Strong technological development in CANS has occurred in recent times and is still on going, with envisaged strong performance characteristics and beneficial applicability [175]. This is expected to open new opportunities in several application fields, including neutron scattering. In this section, a brief summary of the status of new neutron sources and their influence on instrumentation is given. The design of compact neutron sources still needs to overcome some technological challenges but, at the same time, there are a number of opportunities that can be explored.

The term CANS covers a wide range of neutron sources. It excludes the large spallation sources at the high energy end and usually excludes the classical “tube” DD and DT neutron generators at the low energy end. Accelerator designs under development include a variety of electrostatic accelerators, electron linear accelerators (LINAC) that produce photo neutrons from Bremsstrahlung, proton LINACs and cyclotrons. These designs cover a range of prices and performance characteristics. At the forefront are developments at the Forschungszentrum Jülich in Germany for the replacement for a research reactor suitable for a small institute such as a university. Reference conceptual designs are based on a commercial electrostatic accelerator suitable for a small institute such as a university (NOVA ERA [176]) with capital costs of ca. M€4 for the accelerator and targets and M€5 for a full suite of instruments. The CANS is based on a 100 mA, 70 MeV drift tube proton LINAC, with capital estimate of ca. M€121 for the accelerator and targets, M€94 for a full suite of instruments and a total of

M€370 for the entire project including a dedicated building [177]. Current plans foresee the development of CANS sources with target power of 50 to 100 kW, which, while falling in the medium flux category, can support large scale user facilities [178].

Compact target/moderator assemblies result in a larger fraction of the neutrons produced to be moderated to cold or thermal energies as compared to research reactor neutron sources. The smaller target/moderator assembly may enable optimized neutron optics, which can be located in close proximity to the neutron emission surface. Beam optical elements (for example a Wolter mirror) can be installed even inside the moderator to create a given desired divergence.

CANS can be either continuous or pulsed. For most neutron scattering applications, a pulsed source may be preferred. The repetition rate of CANS instruments can be tuned (e.g. 10, 40, 100 and 400Hz), and accordingly the pulse length also can be tuned (from around 100 μs to around 2 ms) and optimized to the experiment being conducted. It is possible that a single accelerator can have multiplexed pulses with different pulse frequencies and lengths delivered to different target [177].

Advantages with CANS are that instruments can be very close to the source, at distances smaller than 2 m, requiring less shielding due to the lower background and without the need for neutron guides. This is closer than at a research reactor. This is also the case at spallation sources where the faster neutrons and gamma rays require extensive shielding. This can lead to potential competitive advantages of CANS notwithstanding their weaker neutron strength.

If fast neutrons need to be removed, guide/bender systems can be used. Nevertheless, as these sources function in TOF mode and can work with short neutron pulses, shorter neutron guides are needed than in research reactors.

Some technical problems in the construction of CANS are still being investigated. A fundamental one is the choice of the target material, including handling the ageing process due to the high irradiation fluences. Currently, candidates chosen for research include Be, Li, C, V, Ta and eventually also liquid Pb. Heat extraction through a ≈100 cm^2 surface is also challenging.

In research reactors, usually one cold source is shared by several instruments. For CANS it is in principle possible to develop one cold source per instrument, and colder neutrons should be available due to the absence of the strong gamma heating that exists in research reactors. Finally, it is noted that accelerator based neutron sources are usually subject to less stringent regulations and constraints than research reactor facilities.

4. APPLICATIONS OF NEUTRON SCATTERING

This chapter presents the main applications of neutron scattering that could be the object of R&D or E&T at low and medium flux neutron sources [1, 8]. Some of the techniques discussed require a high performance neutron source and state of the art instruments if the objective is to perform leading edge R&D in all possible areas, but nevertheless can be implemented at lower flux neutron sources for R&D in specific classes of materials within the given application. In such cases, the scope for low and medium neutron sources is stated.

Neutron scattering is extensively applied to address major societal issues facing humanity, such as energy, environment, health, as well as information and communication technologies [179–182]. Leading edge research in these applications is predominantly done at high flux neutron sources due to their extensive supportive infrastructure in techniques and resources, resolution, neutron flux and accessible energy regimes and therefore they are not the object of specific dedicated sections in this publication. Notwithstanding low and medium flux neutron sources also contributing to these areas, this becomes more relevant in specific niches. To illustrate the general value addition of low and medium flux neutron sources, this chapter is subsequently structured thematically.

Each theme is the object of a dedicated section, following a common structure. The purpose of the application is first stated, mentioning the most common and important objectives and information that can be gained with the application. This is followed by a brief description of the techniques that are most commonly employed in the area. Each section concludes with suggestions to users interested in the given applications.

4.1. EDUCATION AND TRAINING

4.1.1. Purpose

The most common utilization of research reactors worldwide, including low and medium flux ones, is E&T [1]. The term education is commonly used for university level activities, often conducive to an academic degree, and also covers scientific research activities at MSc, PhD or postdoctoral level. Training is directed at professionals, often from the nuclear industry or with a radiation protection background, conducive to professional qualification and sometimes certification. Neutron scattering is often part of education activities and sometimes also in training of professionals.

4.1.2. Techniques

At research reactors with neutron beam facilities, demonstrations and experimental classes are usually limited to students of disciplines such as Nuclear Physics or Engineering, following introductory lectures on scientific applications, experimental methods, performance of neutron beam experiments and continued after the class with data analysis of results with computer based analytical tools. Examples of simple experiments that demonstrate the basic principles and potential of neutron scattering include techniques such as neutron powder diffraction of simple samples, transmission, radiography, PGA, SANS and reflectometry.

For advanced degrees, such as MSc or PhD, for science and engineering students, the neutron beam instruments can be used for research projects. Any neutron scattering technique can contribute to the education of tertiary level students based on gaining practical experience, from mundane tasks to very sophisticated research investigations. All students need to be

supervised by authorized local staff. A written instruction on the given instrument must be available, including procedures covering both normal and non-regular operation. (for example: activation of sample, sample falling from the sample holder, etc).

Neutron scattering techniques are extensively used in the fundamental research of systems in biology, chemistry, geology, medicine, physics, etc., in applied research and in industrial problems. Sophisticated use in research is reliant on prior topical knowledge, on understanding the interaction of neutrons with matter and the added value that neutron techniques can bring to a given scientific discipline.

Training of newcomer researchers and technicians can be in various subsystems ranging from radiation safety to specific components that require further development such as beam ancillaries (sample environments), instrument control electronics and software, detection systems, data treatment and reduction, etc.

Training on the use of neutron scattering techniques contributing to the research of materials covers most scientific and engineering disciplines and is strongly dependent on the existence of fully functional instruments. The efficiency of experiments done on state of the art instruments at high flux neutron sources can be strongly increased if the users are already trained by doing experiments at standard instruments in lower flux sources, thus shortening the learning curve considerably. Also, the existence of a wide base of neutron scattering practitioners at low and medium flux neutron sources, not only improves the utilization of those sources, but also feeds human resources, ideas and technical developments into the high performance sources.

4.1.3. Users

Educational institutions, i.e. mainly universities, with courses on subjects such as physics, nuclear physics, chemistry, biology, archaeology and engineering, can effectively use a low or medium flux neutron source such as accelerators or research reactor facilities for the execution of experimental work as part of the courses. If the neutron source is already integrated in a university, this will be easier; otherwise, the facility managers may approach the faculty and suggest common work, for instance with joint supervision of students within a course project or even for a sponsored research project.

Smaller facilities often have easier and more flexible access than large user facilities. In this case, the smaller facilities can be used to provide training on specific instruments to experimentalists (technicians or researchers) before accessing the high flux user facility.

Some low and medium flux reactors are engaged in training in radiation protection, reactor physics, activation, etc., and expansion to neutron diffraction is possible.

4.2. CULTURAL HERITAGE, PALAEONTOLOGY AND ARCHAEOMETRY

4.2.1. Purpose

A combination of information obtained from different analytical techniques is required for determining historical and cultural time frames, as well as material and method of production of ancient artefacts and artworks. The information is also necessary for the selection of less invasive restoration and preservation methods. Neutron analytical techniques such as NAA, PGA and neutron imaging, including neutron scattering, are extremely useful for the

characterization of cultural heritage. As neutrons are non destructive and can penetrate deep into objects, they can reveal information in a depth resolved way, such as texture, phase composition, residual stress and microscopic structures, providing information on the technology of production and on the object's history [183–185]. Studies using a low flux neutron source can be made for sufficiently large sample sizes. In this case, sample homogeneity needs to be considered, as the information will be averaged over the probed volume. Due to the low energies of neutrons used, in the range of milli-electron Volt, nearly no chemical modifications or radiation induced damage occurs. Samples may become activated, but in most cases will decay within short times, allowing for release back to the museums or custodians. Care must be taken with samples that contain elements that may produce prolonged activation.

4.2.2. Techniques

Neutron techniques are widely used in the investigation of cultural heritage and archaeometry [186, 187] such as natural stone, ceramics and metal artefacts. This is often combined with X rays in synergistic ways.

The information that neutron diffraction can extract includes [188–190]: the original source of raw materials; excavation site dating; cultural exchange; trading patterns and trading networks; study the dynamics of mineral hydration; molecular binding; ion exchange, etc. This information can be used to generate dynamic models of degradation or transformation of materials governed by water, carbon dioxide and sulphate ions. The knowledge is also of value in restoration. Some examples are:

- In ceramics, during production, solid state reactions of the raw materials were unavoidable. The information related to phases or ratios of phase proportions is very useful for classification and investigation of artefacts. For example, artefacts composed of a series of mineral phases result in particularly complex diffraction patterns of clay minerals and layered silicates that require high resolution for analysis. Neutron diffraction is a suitable technique for this group of samples as it does not lead to modification of the materials during investigation;
- For metal samples, it is common to find that the microscopic structures were altered during mechanical or thermal treatments in the production processes. The structural change information given by neutron diffraction can be used to study manufacturing processes in ancient times. In addition, after further development, it is possible to use this technique to reveal volume textures and grain distributions in metal artefacts;
- SANS can reveal the burning temperature of clays (including for instance microstructure of voids, fractal exponent, orientation of sample structure and others), origins of marbles, jewellery, etc [191–193];
- The authenticity of artworks, paintings and gemstones can be verified by their phase analysis using neutron scattering. For instance, in paintings, ultramarine blue pigment consisting of light elements can go undetected with XRD [194]. The timeline of ancient objects can be traced back and established by extracting the information of their phases and comparing them with existing available knowledge about the stone age, bronze age and iron age.

4.2.3. Users

Museums, universities, cultural heritage national institutions and other stakeholders in cultural heritage.

4.3. CHEMICAL AND MOLECULAR STRUCTURE

4.3.1. Purpose

This application is generally done at medium flux neutron sources applied to studies such as phase transitions and structure refinements. It becomes challenging at low flux neutron sources for the investigation of materials with low coherent scattering and especially if higher resolution is required to resolve overlapping peaks. Structure solution on the other hand generally requires very high resolution and the higher fluxes at intense sources are required.

The chemical structure of a material is regarded as the three dimensional (3D) network of atoms, ions or molecules in a periodic arrangement. The periodicity can exist in the form of long range order or short range order in crystalline and amorphous materials, respectively. The development of new materials with improved properties relies extensively on knowledge of the chemical structure, i.e. how the atoms are organized in the material, as their properties are directly linked to the atomic structure.

XRD is the most frequently applied technique to determine the chemical structure of materials, due to ease of access to the technique and the high X ray fluxes available for instance at synchrotrons. Neutron scattering techniques can nevertheless provide complementary or unique information, taking advantage of their different scattering behaviour and of the lower radiation damage induced by neutrons e.g. in biological matter [195]. Thus, they find areas of application in both organic and inorganic chemistry, where the scientific tasks and questions are usually different. The main research areas investigated by neutron scattering in inorganic chemistry are:

- The position of atoms in a crystalline lattice can be determined by neutron diffraction (both powder diffraction and single crystal diffraction), very often used together with X ray (or electron) diffraction to determine the crystal structure. Due to the differences in weighting of scattering of X rays and neutrons across the periodic table, neutrons have an almost similar sensitivity to H(or D) as compared to heavy atoms, enabling refinement of H atoms from powder diffraction involving heavy atoms such as Pb, Br, I. In neutron scattering isotope labelling extends the diffraction possibilities, providing more detailed information on specific parts of chemical compounds [196, 197];
- In amorphous or liquid materials, neutron diffraction can give information of pair correlation of atoms or small molecules, such as e.g. water. The isotope substitution of one or two components of the material assists in establishing spatial correlation between different atoms, atom groups or small molecules;
- The motion of small molecules is measured by quasielastic neutron scattering techniques, including indirect geometry spectrometry and cold neutron TAS. The typical time scale is 10^{-11}–10^{-9} s, corresponding to e.g. the rotation time of a specific molecule or a part of molecule. This gives information about the nature of diffusion processes, which is a key part of chemical processes [198]. This technique is not appropriate for low flux neutron sources and is challenging in medium flux sources, where optimized setups are needed.

In organic chemistry, the basic information is the shape and conformation of molecules in different mediums and circumstances, usually in solvents and in different temperature, field and pressure conditions. The relative arrangement of parts of macromolecules can also be determined. The relative arrangements and shape of parts of a molecule can be isotopically labelled, providing contrast to the rest of the molecule. In SANS experiments [199] a

relatively simple technique is used, by changing the solvent to one that has no contrast towards the rest of the molecule, and only the labelled parts of the macromolecule are seen. Some of the main research areas investigated with neutron scattering are:

- In the study of biological samples. One of the advantages of neutrons is that the neutron beam produces much less radiation damage than X rays, and the same sample can be used multiple times in further measurements, whereas the intense flux and energy of e.g. synchrotron radiation may alter the sample condition. More importantly, biological samples contain light elements such as hydrogen, which is almost invisible to X rays but to which neutrons are highly sensitive. Finally, biological matter such as proteins can be studied under ambient conditions, including in situ and in vivo studies [200].
- The different scattering lengths of hydrogen and deuterium is used to study the kinetics and evolution of the structure of deuterated proteins and drug, and it is possible to develop extremely complex temperature and pressure phase diagrams such as that of water (H_2O-D_2O) [201]. Also, neutron diffraction experiments have revealed key information to understand the enzymatic function [202].
- In chemical processes of macromolecules, the dynamics of the processes are studied, i.e. movement as a whole and of specific parts or segments of the macromolecule. The time scale of such motions is 10^{-9}–10^{-7} s [77]. When only one single molecule is labelled, its motion can be followed.

4.3.2. Techniques

This area of applications concerns many disciplines. In crystallography, chemistry, condensed matter physics, geology, mineralogy, materials science and biology neutron scattering is particularly applied to structural analysis:

- Neutron powder diffraction is used to determine the nucleus position in the material. This is the most frequently applied technique to determine the chemical structure of materials, due to the simplicity of sample preparation and short measurement times when compared to other neutron diffraction techniques. Notwithstanding powder diffraction providing a one dimensional picture of reciprocal space compared to a single crystal measurement in the 3D scattering space, a powder diffraction measurement may provide sufficient information to many research questions [203];
- Single crystal elastic scattering can be used to obtain structural details of atomic arrangements and their vibrations, including exact atomic positions, thermal parameters, bond lengths, bond angles and site order;
- Single crystal inelastic neutron scattering is used to investigate the dynamics of the crystalline structure (phonon, polarons, etc.) and also the dynamic magnetic properties of matter (spin waves including magnons and skyrmions);
- The isotope labelling (or isotope substitution) method is widely used in chemistry and biology.

In organic and macromolecular chemistry, neutron scattering techniques are applied to the study of molecular dynamics and macromolecular chemistry:

- Quasielastic neutron scattering techniques, including indirect geometry spectrometry and cold neutron TAS, provide direct information about the diffusion processes of molecules, and on the motion of groups of atoms in macromolecules and biological

objects [198]. This technique is not appropriate for low flux neutron sources and is challenging in medium flux sources, where optimized setups are needed;
- SANS is an important tool in macromolecular chemistry, showing the conformation of molecules or parts of molecules labelled by isotope exchanging. SANS also plays an important role in the investigation of catalysers, in the solvation/aggregation processes, colloid chemistry, membrane structures and membrane diffusion [199];
- The spin echo technique is adequate for the time scale of macromolecular motion [204]. It is, however, not appropriate for low and medium flux neutron sources;
- Neutron reflectometry is used in surface chemistry, for instance, to study the arrangement of surfactants and the details of surface structure [75];
- Inelastic scattering, together with complementary Raman scattering measurements, is used to investigate chemical bonds and their strength.

4.3.3. Users

A large scientific community working in different fields such as natural sciences, materials science, petrology, earth sciences, pharmaceuticals, archaeology and others is involved in the structural and dynamical analysis of materials. Potential users include scientific institutes, industrial partners working in the pharmaceutical industry, polymer producers, all areas where catalytic processes are used, oil industry where the diffusion and percolation processes play an important role, geochemistry, mining and bio industry.

4.4. MAGNETIC STRUCTURE

4.4.1. Purpose

Magnetic neutron scattering is, arguably, the most powerful technique used to determine and understand the microscopic properties of magnetic systems, including the fundamental nature, symmetry, moment distributions in magnetic materials at the atomic scale and dynamics of magnetically ordered materials [1, 205, 206]. Neutrons, by virtue of their magnetic moments, can determine the spatial arrangement and directions of atomic magnetic moments of individual atoms in the material, and the value of the ordered moments as a function of thermodynamic parameters such as temperature, pressure and applied magnetic field. Neutron diffraction is thus one of the most powerful techniques for understanding the cooperative magnetic phenomena occurring in magnetic sublattices of ferro, ferri and antiferromagnetic materials. Magnetic symmetry is complex, but in recent years group theory approaches based on magnetic co-representations have simplified the analysis of magnetic diffraction [207, 208].

The fabrication of new artificial layered materials gave rise to new physical phenomena. Magnetic layers separated by non-magnetic spacer layers can be magnetically coupled, giving rise to spintronics properties such as the giant magneto resistance effect [209].

Magnetic neutron scattering is also applied to the study of other nanomagnetic systems, for example, magnetic phase separations in crystals, self-organized magnetic domain structures, critical fluctuations near the Curie temperature and steels containing magnetic inclusions [210]. Magnetic nanowires or nanotubes can also be studied.

Neutron inelastic scattering (see section 4.6.1) can reveal the magnetic fluctuation spectrum of materials over wide ranges of energy (from 10^{-8} to 1 eV) and over the entire Brillouin zone [211]. Neutron scattering plays a truly unique role in that it is the only technique that can

directly determine the complete magnetic excitation spectrum, whether it is in the form of the dispersion relations for spin wave excitations, wave vector and energy dependence of critical fluctuations, crystal field excitations, magnetic excitons, or moment fluctuations.

High flux neutron sources are most commonly used in these studies, but medium flux neutron sources can also play a role in this area, because, even though the incident flux is smaller, the signal to noise ratio is similar. Under special conditions, low flux neutron sources may provide some information if large magnetic moments are present.

4.4.2. Techniques

The following techniques can be used:

- Reflectometry: Studies of surfaces and interfaces within a depth of around 1000 Å such as magnetic thin films and magnetic liquids (ferrofluids). With polarized neutrons magnetic properties and depth profiles near surfaces can be studied [1]. In low and medium flux neutrons sources, use of cold neutrons is advisable to improve the useful flux. Magnetic off specular scattering has been mostly used to probe the magnetic domains sizes in multilayers. Detailed quantitative analysis of the magnetic off specular scattering can be performed. This technique is seldom applied in low or medium flux neutron sources, but some implementations have been achieved;
- SANS: The neutron beam technique most used for the study of large scale magnetic structure determination, in materials where structural correlations exist over a length scale from around 10 Å to around 0.1 μm. Magnetic nanoscale objects such as magnetic particles and clusters are studied using polarized neutron beams. It is also possible to perform SANS studies to characterize magnetic critical scattering, the penetration of magnetic flux in superconductors (vortices), skyrmions, or any other type of magnetic correlations that take place at the nanometric scale [212];
- Diffraction: Experiments can be performed to probe the magnetic order at the atomic scale [213]. It can determine the location of the magnetic atoms (magnetic form factors, spin density maps in magnetic crystals), complex magnetic orders (antiferromagnetic, helical magnetic sublattices) and provide information on magnetic phase transitions such as spin reorientation phenomena;
- Triple axis spectroscopy and indirect geometry spectrometry: Quasi elastic neutron scattering (QENS) measurements are usually carried out in a dedicated instrument, such as an inverted geometry spectrometer. The energy resolution should be carefully chosen with regards to the time scale of interest for the systems to be measured. For the test or preliminary experiments, a TAS instrument can also be used. QENS leads to a broadening around elastic scattering peaks [214], which can be used to study diffusive processes such as the movement of ions or spin waves [1]. This knowledge is particularly useful for understanding the atomic origin of thermoelectricity or the magnetic coupling responsible for the superconductivity in ceramic cuprates. Inelastic Neutron Scattering is used to study spin dynamics, from local to collective excitations, helping to obtain a microscopic description of the systems [215]. Using polarized neutrons the magnetic nature of the excitations can also be studied [216]. Cold neutron sources are useful for such measurements.

4.4.3. Users

The potential users of magnetic structure can be universities, research institutions and industries producing electronic devices, magnetic sensors, medical application and defence.

4.5. NANOMATERIALS

4.5.1. Purpose

Nanomaterials are defined as materials with any external dimension in the nanoscale or having an internal structure or surface structure in the nanoscale, which is taken to correspond to a size ranging approximately up to 1000 Å. The European Commission's recommendation on the definition of nanomaterial is "a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50% or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 to 100 nm" [217, 218].

Nanomaterials have a substantially larger surface to mass ratio than bulk materials. The atoms and molecules on the surface can be chemically more active and their internal structure also differs from the bulk one. The vibration spectrum is shifted or may be absent (optical mode). The mechanical, thermophysical and magnetic properties also usually greatly differ from the bulk properties.

Nanomaterials have extensive fields of application in many modern technologies. They are present in consumer electronics, innovative medical applications (such as contrast and drug carriers), food industry (stabilizers, product forming), ceramics (insulator, trap for specific molecules, filters, electromagnetic shielding) and many other areas present in the daily lives of people.

There are several neutron scattering techniques that greatly contribute to the study of nanomaterials, and some have been developed at medium and low neutron flux sources. The wavelength of cold (in some cases, thermal) neutrons is appropriate to reveal the atomic arrangement of matter. By carefully selecting the technique and experimental conditions, structural features that occur on length scales between approximately 10 and 1000 Å can be probed. The size range spans a vast range of science, from proteins and viruses (biology and medical sciences) to emulsion and microemulsions (polymer and material science) to phase separation and fractal growth (physics, geology and metallurgy). Liquid, amorphous and nanocrystalline samples can be studied [60].

4.5.2. Techniques

SANS is the neutron diffraction technique of choice to study nanomaterials. SANS is highly complementary to small angle X ray scattering (SAXS), and the two techniques are often used in conjunction [219]. Both X rays and neutrons cover the length scales from the atomic structure of individual building blocks to the configuration of assembled, functional structures, making them essential tools for the elucidation of nanostructures. The complementarity arises from the different properties, such as spin, mass and sensitivity to different elements, including hydrogen (and deuterium), omnipresent in biological systems. Further details are given in section 2.5. SANS provides information on the average nanostructure of the material, such as surface area, volume and shape of the inhomogeneities, their size distribution function and the inter particle distances. If the diffractometer can measure q values sufficiently high in order to separate stress and particle broadening effects, NPD can also be used to study nanoparticles.

Inelastic scattering is also used to study the dynamics of nanoparticles. Nanoparticles behave in a sharply different way than bulk materials, and inelastic neutron scattering, often together

with complementary Raman spectroscopy and optical methods, provides a great deal of information [220].

4.5.3. Users

University and research institutions, R&D departments of pharmaceutical, electronics, food, cosmetics, defence, geology and other industries.

4.6. LATTICE DYNAMICS AND MAGNETIC EXCITATIONS

4.6.1. Purpose

Lattice dynamics is the study of the atom vibrations in crystals or molecules. This contributes to the understanding of basic physical properties such as specific heat, thermal conduction and expansion, melting temperatures, electron transport, superconductivity, sound transmission and other phenomena [221]. At present there is a renewed interest in lattice vibrations as the basis to study systems and devices such as high temperature superconductors [222] and quantum microphones (laser interferometry) [223]. These new promising and challenging fields of knowledge require a good description and control of lattice dynamics.

For these studies, the forces among atoms in the lattice must be known. They can be determined from density functional theory techniques and from molecular dynamics [224]. The harmonic travelling waves propagate throughout the crystal with specific energies. The quantized energies of this lattice vibration are known as phonons that are characterized by three parameters, i.e. wave vector, energy and polarization vector. Information about relative atomic vibrations along the three orthogonal directions can be achieved from polarization vectors. Phonon dispersion, i.e. the energy wave vector relation of the thermal motions of the atoms, provides information on the physical properties of any solids through the interatomic interactions. Interpretation of the measured spectra can be greatly aided by modern symmetry analysis programs [225, 226], which can assist in assigning symmetry and calculating mode intensities thereby unambiguously assigning modes [227].

Spin waves are elementary excitations in ordered magnetic systems with localized magnetic moments interacting with each other (cooperative phenomena). For magnetic materials, from magnon dispersion relation, information regarding spin orbit coupling and other spin dependent properties can be studied.

In general, the understanding of lattice dynamics and magnetic excitations is the key to explain many important phenomena such as [228]:

- Phonon dispersion in crystalline materials, from which the interatomic forces can be determined;
- Spin wave dispersion, which allows one to determine exchange and anisotropy parameters;
- Dynamic of biological model membranes;
- Lattice and magnetic excitations in quantum magnet and superconductors;
- Phase transitions to study critical behaviour.

This area of application is most often found at high flux neutron sources, and under special conditions at medium flux sources in the investigations of materials that have large coherent

nuclear scattering or large magnetic moments. It is not appropriate for low flux neutron sources.

4.6.2. Techniques

Optical techniques such as Infrared and Raman spectroscopy are common, but limited in the information they can provide, in lattice dynamics studies. Due to the high hydrogen cross section, neutron techniques become highly relevant when hydrogen dynamics is studied, also considering the almost non-existent signal in optical techniques. Recently experimental inelastic neutron scattering, Raman and ab initio lattice dynamics proved to be an auspicious combination of techniques. Moreover, neutron measurements can provide directional information.

A brief summary of the main neutron techniques of interest for lattice dynamics and magnetic excitation studies is given in the following.

- Time of flight spectrometry. This technique can resolve lattice frequencies and their occurrence. One great advantage of the method is that surveys of the excitation spectra can be quickly obtained from polycrystalline materials [1];
- Triple axis spectrometry. This technique allows the measurement of the phonon and magnon branches of lattices. It also provides insight on the role of the atomic polarization in dielectric properties. This type of spectrometer has played a major role in understanding collective atomic and magnetic dynamics in condensed matter physics and solid state chemistry [1]. Neutron TAS, covered in Section 2.8, is the most versatile and useful technique to study lattice dynamics and magnetic excitations because it allows one to probe nearly any coordinate in energy and momentum space in a precisely controlled manner [93].

4.6.3. Users

Users can be scientists working in the areas of mineral sciences, molecular crystals, chemical vibration spectroscopy, magnetism and nuclear data libraries relevant to nuclear reactor simulation. Universities and research institutes support most of the activities related with this application. Chemical industry is the most important industrial applicant for these services. Among the topics of interest, we find polymers, catalysts, hydrogen and fuel storage systems, fuel cells, magnetism and batteries. Also, industries related to solar energy, environmental engineering (in particular for water and soil remediation) and cement industry can find applications in this area.

4.7. NUCLEAR ENGINEERING

4.7.1. Purpose

Nuclear Engineering encompasses all the activities of the nuclear industry, such as the design, operation and maintenance of nuclear facilities. It also includes nuclear safety, nuclear materials, nuclear fuels, radiation induced damage and related technology (radioactive waste disposal), issues related to nuclear proliferation and medical applications of radiation, particularly ionizing radiation. Despite being an eminently applied activity, nuclear engineering is a fertile field for basic research, since many of its activities require research in materials and processes that frequently lead to the creation of new design concepts. Due to the nature of their activities, the final users of nuclear engineering are often governmental

agencies, which are usually responsible for the design, management and control of the nuclear activity. However, the private industry participates in the different phases of reactor design and construction and is usually the main applicant for these services.

It should be noted that most of the applications and techniques mentioned in this section are most often performed at high flux research reactors and accelerator based neutron sources. Nevertheless, opportunities remain for low and medium flux sources to find niches of application which are relevant.

4.7.2. Techniques

A brief summary of the main neutron techniques of interest for Nuclear Engineering is given in the following [229].

- Powder diffraction. Nuclear Engineering has an interest in the development of new materials. With this technique the goal is usually to understand the connection or correlation between material properties and materials structure due to radiation exposure. As examples, we can cite the study of phases present in a system depending on the manufacturing process, with an impact on its resistance and durability [230], or the study of nuclear grade materials such as steels and aluminium alloys, graphite, etc. in which the ability to quantify the disorder in the atomic structure provides an essential starting point for the investigation of the structure upon exposure to neutron irradiation [231]. Shielded cells can also be integrated into the diffractometer in order to measure spent fuel [232]. Investigation of waste management related materials such as zeolites, ceramics, glasses, clays, cements, etc. can also be investigated to follow the structure and phase changes as a function of time and environment;
- Residual stress and texture. With this neutron diffraction technique, it is possible to probe material stress distributions in real engineering materials. The study of the stresses caused by welding is an important application with direct impact in Nuclear Industry. Being a non-destructive technique, it is possible to study complete parts that are used as components in nuclear reactors. In this field, radiation damage of the materials used in the reactors is a topical issue [233];
- Time of flight spectrometry. The studies of vibrational excitations of moderator systems provide the knowledge of the moderation process that is essential information to build nuclear data libraries, such as the widely used ENDF/B-VII [234]. The thermal neutron scattering cross sections are needed to accurately model the scattering of neutrons in the moderator. Also, quasielastic scattering studies of diffusional behaviour (including using indirect geometry spectrometry) are of interest to describe the low energy behaviour of the frequency spectra. This technique is challenging at medium flux sources, where optimized setups are needed. It is inadequate for low flux neutron sources;
- Deep inelastic neutron scattering. It is usual practice to determine the effective temperatures of moderator materials indirectly from models of frequency spectra. This technique gives the unique possibility to make a direct measurement of the effective temperatures of each atomic species composing a system, since it is a mass resolved technique [55];
- Transmission. The measurement of total cross sections in a wide range of energies is a stringent test for the models of moderators used to produce nuclear data libraries. Knowledge of the materials total cross sections is essential for the different stages of nuclear reactor calculations. The various Monte Carlo methods available today require its detailed knowledge as a function of energy. This is also an important tool in the

practical study of shielding to be applied in the construction of nuclear reactors, repositories and ancillary facilities;

- Small Angle Neutron Scattering. For the study of the nanostructure and control the integrity of materials on a nanometric scale in metals, plastics, polymers, etc, and to assist materials development. SANS is very useful for controlling material manufacturing processes, the influence of radiation on nuclear materials and initial stages of phase transition. Irradiation processes, such as materials structures, fatigue can be followed;
- Test beams for neutron engineering. Low power Accelerator Driven Neutron Sources with a relatively low neutron flux can provide very good opportunities for the design/optimization of target/moderator/reflectors, moderator materials, novel devices and instruments, detectors, collimators, neutron guides and neutron optics, prepare experiments on high power sources, sample characterization, new sample environments and others [235].

4.7.3. Users

Governmental institutions usually have primary control over the nuclear activity of their respective countries, and often lead the research activities in this area. Therefore, they should be considered as the main applicants of the described techniques.

Research institutions such as universities and research institutes also support activities in nuclear engineering, not so much from the point of view of control and operation, but from the various aspects of applied research, therefore they should be considered as applicants for these services. Among the many research activities related to nuclear engineering we find fusion technology, materials under intense particle beams irradiation, modelling of materials evolution in extreme environment, fuel cycle and waste management, nuclear materials, nuclear systems simulation, reactor physics, thermal hydraulics and reactor safety.

As contractors of nuclear infrastructure works, industrial partners also participate in construction of power plants, logistics of raw material and waste and manufacturing of parts, among others. As far as these activities demand research and quality control, the industry is a possible candidate to request these services.

4.8. FORENSICS

4.8.1. Purpose

Forensic is a Latin word describing public debate or examination as in ancient times trials were held in public. Nowadays, forensic science is the application of science to the identification, individualization and evaluation of physical evidence related to any area subject to criminal or civil regulation. Its ultimate goal is to find linkages between people, things, places and events. Scientifically, it is a multidisciplinary approach which encompasses a wide range of methodologies from analytical chemistry techniques to biological procedures.

Possible areas of forensics applications include crime investigation, food safety and health related issues, cultural heritage artefacts, environmental and biological samples, engineered materials. Other areas where the study of provenance, history and usage, counterfeiting, mislabelling and adulteration of samples and materials are of paramount relevance to forensics.

4.8.2. Techniques

The application of neutron diffraction techniques to forensic sciences shares many of the characteristics of other applications such as to cultural heritage, chemical and magnetic structure and engineering, including the techniques employed (see sections 4.2, 4.3, 4.4 and 4.7). Some further examples are:

- In forensic anthropology, retrieving information and the biological profile from the remains of burnt or damaged bone remains is a challenging task. Bone is composed of organic and inorganic components which gradually decay after death, undergoing chemical changes. Heat exposure alters the hydrogen bonding pattern within the framework of bone, which can be studied with neutron diffraction owing to its sensitivity to hydrogen. Moreover, the large penetration depth of neutrons ensures that information is obtained from the bulk structure [236];
- In forensic engineering, the cause of failure of materials or components is investigated, often with the goal of determining corrective and preventive measures. Forensic failure analysis can improve the performance and life of engineering components and consequently prevent catastrophic failure. Neutron diffraction can shed light on the failure of materials by forensic analysis of residual stress pattern of fractured surfaces or from a similar unfractured material [237];
- To investigate the study of structural failures of materials in nuclear power plants. The orthorhombic crystal structure of uranium leads to anisotropic negative thermal temperature, which creates integrity problems at high temperatures. Therefore, uranium is often used as a nuclear fuel in the form of uranium oxide and uranium silicide. These and other nuclear phases present in the nuclear fuel, for investigating the study of structural failures of materials in nuclear power plants, can be identified using neutron diffraction with better accuracy than with XRD [238].

NAA has also been widely used in forensic investigations [239, 240, 241].

4.8.4. Users

Forensic scientists in governmental and private institutions, agencies and industry, customs, museums, art galleries and other stakeholders.

5. CONCLUSIONS

Neutron scattering is used worldwide to address societal challenges that humanity faces. It is an engine for the generation of new knowledge and fundamental research, and it helps to shape the new generations of researchers, scientists and engineers that constitute the future workforce in nuclear sciences and technologies. Neutron scattering is applied in health and life sciences, for instance to solve problems and provide information on biological function, including viruses, proteins and degenerative diseases and to assist in the development of new drugs and therapeutic approaches. Neutron scattering contributes to understanding processes relevant to production, pollution, purification and conservation of food and water. It plays an important role in studying new energy sources to protect the environment and combat climate change, including hydrogen storage, fuel cells, solar cells and new types of batteries. It is applied to unveil crucial information in cultural heritage, archaeometry and forensics, with applications to conservation and restauration, dating, understanding weathering phenomena, past technologies and fabrication methods and authentication of artefacts and objects. Neutron scattering allows the study of electronic and magnetic nano systems, spintronics systems, novel superconductors, molecular electronics and other systems, with applications to the development of new information and communication technologies. It is used in many industrial and engineering applications, such as metallurgy, cement and concretes, aerospace and automotive components, nuclear materials and many others.

Neutron scattering is, as yet, a relatively unexplored application at low and medium flux research reactor and accelerator based neutron sources. The perception — and often reality — of high cost, relative complexity of the instruments and high level of technical expertise required to install and operate neutron scattering instruments are some of the factors that contribute to that situation. In fact, leading edge R&D made with state of the art neutron scattering instruments is usually done at high performance high flux neutron sources that often function as user facilities with several scattering (and imaging) instruments available to the user community.

Low flux research reactor and accelerator based neutron sources are effectively limited in the techniques and applications of neutron scattering that they can implement. Their capability in the area is often confined to the simplest neutron scattering techniques, used for demonstration purposes. Such capability, however, can be effectively used in practical experimental classes for the education of university students of nuclear science and technology subjects. Besides immediately increasing the utilization of the facility, this can also attract new stakeholders to use the facility.

At the same time, there are ample opportunities for neutron scattering techniques to be implemented at *medium flux* neutron sources. This can be not only for demonstration purposes, as in low flux sources, but also to develop techniques and instrument components, to perform test experiments prior to experiments at high performance facilities, to train and educate technical and scientific staff as well as students and to perform R&D in many areas where medium flux neutron sources can provide competitive conditions, especially with the incorporation of advanced in situ sample environments that enable studies of systems under relevant parametric conditions.

In particular, ongoing technological development in compact accelerator based neutron sources is expected to open new opportunities in neutron scattering. The repetition rate and neutron pulse length of such sources can be tuned and optimized to the experiment being conducted. Compact target/moderator/shielding assemblies may enable optimized neutron

optics, leading to instruments that may be competitive with those installed at high performance neutron sources and may even be capable of supporting large scale user facilities.

One further aspect that may increase the utilization of low and medium flux neutron sources for neutron scattering is data digitization. Agreement within the neutron scattering community, with adoption by the low and medium flux sources of the data formats used at high flux sources, would facilitate the use of the software packages developed at or initiated by the use of high flux sources, which usually are the most advanced. This would promote the establishment of connections and networks between users of the different facilities, opening up possibilities to perform experiments that do not require high fluxes in lower flux facilities. Finally, it would also contribute to the training and professional development of neutron scattering scientists at low and medium flux neutron sources, which would also positively impact high flux facilities.

In recent decades, X ray techniques based on either synchrotron or free electron laser sources, have developed significantly, supported by extensive resources and by technical developments in a number of high performance facilities, which were applied to the exploration of new instrumentation concepts. With neutron scattering the situation is different. In fact, most of the most striking conceptual and proof of concept developments in neutron scattering have been carried out in the relatively distant past at low and medium flux sources and were later exported to large facilities. This has led to slower technical improvement and development than in X ray techniques. The contribution of low and medium flux neutron sources in this essential incubator role can be intensified, particularly if strong linkages to the high neutron flux facilities are established.

Without exploiting the value added provided by low and medium flux neutron sources, the high performance sources and high performance instruments will not have enough capability to maintain a thriving neutron international community. The low and medium flux neutron sources are needed to support the community, providing a broad scientific and technical basis as well as crucial human resources. A great deal of experiments can be performed at such sources, liberating the high performance sources for those cases where higher experimental complexity or higher flux is needed. In this regard, this publication is intended to inspire low and medium flux research reactor and accelerator based neutron sources to develop and expand their neutron scattering capabilities.

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ANNEX: INDIVIDUAL PAPER CONTRIBUTIONS

The online supplementary files for this publication can be found on the publication's individual web page at www.iaea.org/publications.

Member State	Main Author	Paper title
Algeria	Mr Tarik Segueni	Neutron scattering instruments at the 1MW NUR research reactor
Argentina	Mr Javier Dawidowski	Relevance of neutron techniques based on a small flux accelerator pulsed source
Bangladesh	Mr Shahzad Hossain	Neutron diffractometer and its application for research and development in Bangladesh
Egypt	Mr Tarek Abd El Slam	Small angle neutron scattering and its applications in enhancing neutron imaging
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Indonesia	Mr Iwan Sumirat	An overview of Indonesia neutron scattering facility and its utilization
Jordan	Mr Khaled Toukan	The Jordan Research and Training Reactor (JRTR) Utilization Programme
Malaysia	Ms Julie Murshidi	Development on neutron scattering instruments at reactor TRIGA PUSPATI
Pakistan	Mr Khurram Shahzad	An overview of neutron scattering techniques at PARR-1
Romania	Mr Marin Dinca	High resolution neutron diffractometer facility from INR Pitesti
South Africa	Mr Andrew Venter	Diffraction facilities at the SAFARI-1 research reactor
Thailand	Mr Jatechan Channuie	An overview of neutron diffraction at Thai research reactor (TRR-1/M1)

LIST OF ABBREVIATIONS

BGO	Bismuth germanate
CANS	Compact accelerator neutron sources
CIF	Crystallographic information file data format
CW	Constant wavelength
DINS	Deep inelastic neutron scattering
E&T	Education and training
FAIR	Findable, accessible, interoperable and reusable
HOPG	Highly oriented pyrolytic graphite
LINAC	Linear accelerator
MCNP	Monte Carlo n-particle transport code
NAA	Neutron activation analysis
NPD	Neutron powder diffraction
PG	Pyrolytic graphite
PGA	Prompt gamma analysis
QENS	Quasi elastic neutron scattering
R&D	Research and development
SANS	Small angle neutron scattering
SAXS	Small angle X ray scattering
SCND	Single crystal neutron diffraction
TAS	Triple axis spectrometer
TOF	Time of flight
XRD	X ray diffraction

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Meetings

Technical Meeting on Neutron Scattering and Spectroscopy with Low and Medium Flux Neutron Sources, Vienna, Austria: 13–16 May 2019

Consultancy Meeting on Neutron Scattering and Spectroscopy with Low and Medium Flux Neutron Sources, Vienna, Austria: 11–15 November 2019

(Virtual) Consultancy Meeting on the Development of a Publication on Neutron Scattering with Low and Medium Flux Neutron Sources: 12–15 October 2020

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