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Use of neutron beams for low and medium flux research reactors: R&D programmes in materials science

Report of an Advisory Group meeting held in Vienna, 29 March–1 April 1993

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FOREWORD

Research reactors have been playing an important role in the development of scientific and technological infrastructure and in training of manpower for the introduction of nuclear power in many countries. Currently, there are 284 operational research reactors in the world, including 88 research reactors in 39 developing countries; the number of reactors in developing countries is increasing as more countries embark on programmes in nuclear science and technology. However, full utilization of these facilities for fundamental and applied research has seldom been achieved. In particular, the utilization of beam ports has been quite low.

Neutron beam based research is regarded as one of the most important programme areas that can be implemented even at low and medium flux reactors. The range of activities possible in this field is so wide that it is generally feasible to define R&D programmes to suit different needs and conditions. It is, therefore, important and of direct benefit to identify effective ways to improve utilization of beam tubes and of the research reactors. To this end, the International Atomic Energy Agency organized two meetings during 1993. First, an Advisory Group Meeting (AGM) on Use of Research Reactors for Solid State Studies and second, a Technical Committee Meeting (TCM) on Use of Research Reactor Neutron Beams for Radiography and Materials Characterization. The goal of the AGM was to review and provide recommendations on neutron scattering techniques, while the TCM focused on neutron radiography and other simple methods and applications.

The present report is the result of the Advisory Group Meeting held during 29 March - 1 April 1993 in Vienna with subsequent contributions from the experts. The Physics Section of the Department of Research and Isotopes was responsible for the co-ordination and compilation of the report.

The report is intended to provide guidelines to research reactor owners and operators for promoting and developing neutron beam based research programmes for solid state studies using neutron scattering techniques. It is expected to benefit ongoing facilities and programmes by encouraging use of improved techniques for detection, signal acquisition, signal processing, etc. and new programmes by assisting in the selection of appropriate equipment, instrument design and research plans.

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CONTENTS

1. INTRODUCTION

Worldwide there are 284 operable nuclear research reactors of which 88 are located in 390 developing countries. These research reactors and the laboratories around them represent a significant research potential and a source of highly qualified and trained manpower not only for activities related to nuclear fields but also for several conventional technologies. The acquisition of a nuclear research reactor, in a developing country, is generally justified on the basis that it is a necessary tool for:

- Fundamental and applied research in various disciplines;
- Training of young scientists in nuclear sciences;
- Production of radioisotopes;
- Supporting activities related to nuclear power including operator training;
- Engineering experiments.

These objectives have been achieved with different degrees of success in different countries. Among several factors that contribute toward underutilization of these facilities, the most obvious ones are: inappropriate research programmes, lack of adequate equipment and laboratories, relatively low neutron fluxes, non-availability of sufficiently trained manpower. In particular, the utilization of neutron beam ports remains very low. This is in spite of the fact that neutron beam based research is one of the most important programme areas which can be implemented, even at low and medium flux reactors. The range of activities possible in this area is so wide that it is in general possible to define and carry out R&D programmes to suit specific needs and conditions.

This report covers various aspects of neutron beam research applicable to materials science. It specifies minimal requirements concerning infrastructures, personnel, funding, etc. as these conditions must coexist for a viable research programme over a period of several years. A brief overview of R&D programmes on condensed matter physics with the required experimental methods, instruments and techniques is presented. Depending upon local conditions, the report is intended to serve as a technical document to guide, initiate, review, upgrade or support programmes in neutron beam based R&D. Topics like production of radioisotopes, irradiation, neutron radiography, nuclear physics, etc. are not covered. Related subjects on neutron radiography, irradiation, etc. are covered separately in IAEA-TECDOC-837. The papers presented by the participants are included in the Annex.

The report will benefit the ongoing facilities and programmes by encouraging the use of improved techniques for detection, signal acquisition, signal processing, etc. and the new programmes in the selection of appropriate equipment, instrument design, and planning of research. It will also be useful for upgrading and modernization of the aging experimental facilities.

2. FRAMEWORK FOR SUSTAINABLE R&D

2.1. IMPORTANCE OF NEUTRON SCATTERING TECHNIQUES

Neutron beam based R&D is one of the most versatile activities in scientific and technical applications, particularly for relating the bulk properties to microscopic structure (and dynamics) for the development of new materials and techniques. The neutron scattering techniques have proved very fruitful for investigating structural properties of complex materials and in obtaining information on the relationships between structure, properties,

performance and the effects of various processes. As a tool for the characterization of materials, low-energy neutrons offer several unique advantages over other probes such as protons, electrons or X rays. They are also useful in quality assurance of manufactured products, optimization of process parameters and also in the study of fatigue and stress phenomena. These are particularly important aspects for developing countries who desire to develop or assist manufacturing industries and to increase added value to indigenous raw materials. Since neutrons are chargeless particles, capable of penetrating bulk samples, they do not suffer from large form factor fall-offs at high momentum transfers as is the case with X ray. Neutrons have a magnetic moment and therefore provide information on magnetic state of samples although in this case a form factor comes into play. The energies of neutrons match well with those of excitation energies in condensed matter and are therefore extremely useful to study the nature of excitation spectra across the entire Brillouin zone in solids.

2.2. MINIMUM SIZE OF RESEARCH GROUPS

Fundamental requirement for initiating a viable R&D work in neutron beam research at any research center is the availability of a minimum number of scientists and technicians with appropriate specialization and skills. The smallest 'critical' group should comprise 3 qualified scientists (at least one Ph.D.) and 2 technicians. This group should be encouraged to have reasonable contacts with local and foreign scientists in their fields of work. If at any stage, an experienced scientist must be moved out from this small group, the replacement should be provided as soon as possible and continuity should be ensured. Depending upon the qualification and experience of the newcomer, it will be necessary to allow for a reasonable period of overlap. In any case the group should be maintained by retaining at least one of the experienced scientists. Such a small group can initiate one basic activity, say, neutron diffraction. As the interest of the group enlarges and resources become available, additional groups of similar sizes should be formed following the same pattern.

2.3. INFRASTRUCTURAL FACILITIES

The research group, as mentioned above, can function properly only if its scientific efforts receive adequate additional infrastructural support. Essential requirements include electronics maintenance facilities, mechanical workshop, reliable electricity supply, an up-to-date library with recent journals, good computing support and radiation measuring and safety facilities. These facilities are normally needed to serve other scientific programmes and groups as well. Specific requirements for various usages must be catered for at the planning stage and during subsequent improvements.

2.4. ESSENTIAL INVESTMENT NEEDS

Availability of experimental facilities is necessary for initiating any programme of neutron beam research. At least one operating neutron beam instrument should, therefore, be made available to initiate a research programme, immediately after installation and commissioning of the research reactor. A powder diffractometer would be a good starting point for this purpose. If financial resources do not permit procurement of a complete unit, efforts should be made to assemble one, by fabricating some parts and acquiring components like monochromators, detectors, parts of rotating devices, and control and data acquisition systems. This might be feasible over a period of 1–2 years. Additional assured funds should be allocated to cover running costs — samples, sample holders, consumables, etc. — and for maintenance. If electrical power supply suffers frequent breakdowns, a guaranteed power supply system must also be acquired.

The R&D activities may be expanded gradually by adding more instruments and staff over a period of several years. For optimum utilization of resources, the selection of other instruments should match the available neutron flux level of the reactor.

2.5. REVERSE MOBILITY OF SCIENTISTS

Training of scientists from developing research centers and association with research laboratories in industrialized countries play an important role for initiating and promoting R&D programmes. A useful step would be to encourage scientists from active centres in the developed countries to work for suitable periods of time in smaller centres to give them a boost and provide continuity and acceleration in the pace of work. They could help to design, build, and install new instruments, and also imrove utilisation of available resources or take part in cooperative research. They will be able to transfer their expertise more effectively because of their familiarity with local environments.

2.6. INTERDISCIPLINARY COLLABORATION

Neutron beam research is interdisciplinary. It is of great importance to establish a good collaboration between various disciplines such as physics, chemistry, metallurgy and biology at the center. This will help to identify a research programme of mutual interest and enhance the awareness of interesting problems that can be solved by neutron techniques. Such a cooperation will cause a more realistic working programme on one side and help promote the use of neutron techniques.

3. FUTURE RESEARCH PROGRAMMES

One of the important questions that arise in planning a useful and viable research programme in any center, especially at the initial nucleating phase, is related to the choice of materials and scientific problems for neutron beam research.

The choice of a given research area in neutron beam work can be dictated by a number of factors including: (a) local or national interests in specific materials; (b) the balance between applied and basic research interests and motivations; (c) industrial development interests and (d) the interests and expertise of potential collaborators. This last aspect should receive major attention — most highly successful research programmes will require more than neutron investigations alone. The availability of local collaborators with expertise in magnetometry, X ray diffraction, optical studies, study of macroscopic properties like thermal or transport properties, Moessbauer spectroscopy etc. and, above all, specific theoretical knowledge, should have an important bearing in the selection of research problems. From the neutron scattering view point, the type(s) of instruments available for scattering studies (or that can be built at a given reactor) will limit the types of problems that can be tackled, as will the availability of high quality samples. Some specific comments follow on given experimental areas. The summary table below deals with types of condensed matter systems that can be investigated by neutrons using specific techniques. The analysis is focused on only a limited subset of the possible systems that can be investigated with neutrons and is in no way assumed to be comprehensive.

The scientific information obtainable from various materials using various techniques is outlined in the following sections.

TABLE I. MATERIAL SYSTEMS AND APPLICABLE TECHNIQUES

Materials	Techniques				
	Diffrac-tion	SANS	Reflec-tometry	Inelastic Scattering	Depola riza-tion
Magnetic compounds, polycrystalline single crystal	X	X		X	X
Partially disordered magnetic alloys		X		X	
Magnetic films and superlattices	X		X		?
Ceramic high T_c superconductors	X	X			
Liquids and amorphous systems	X	X			
Gels, polymeric materials	X	X		X	
Geological materials	X	X			
Petrogenic materials	X	X			
Polymer films			X		
Zeolites, fast ion conductors	X				
Texture and residual stress	X				
Biological systems	X	X			

3.1. MAGNETIC COMPOUNDS

Much interest has been generated in recent years in the study of magnetic properties of intermetallic compounds, particularly those with rare earth elements, because of their application in areas benefiting from a high magnetic energy product (e.g. $Nd_2Fe_{14}B_1$, R_2Fe_{17}, etc.), as well as the unusual magnetic exchange and anisotropy properties of these compounds. Thus this class of materials can be studied from either an applied (industrial) or basic research motivation. A strong team of collaborators is essential for success, including persons who: (a) make materials in either polycrystalline or single crystal form or both; (b) provide supplementary characterization information such as magnetization and Moessbauer data, and (c) theoretical expertise, particularly if work on exchange and anisotropy properties is to be attempted.

The study of the basic atomic and magnetic structure of these materials in polycrystalline form by neutrons requires only powder diffraction techniques to do successful research, with the proviso that the analysis (say, by Rietveld analysis) must include magnetic scattering.

If single crystal samples can be obtained, neutron scattering work can be expanded to inelastic scattering studies of the dispersive modes using a triple axis spectrometer to determine exchange constants. Crystal field levels can, in principle, be studied on polycrystalline samples for which the triple axis or time of flight method are appropriate.

3.2. MAGNETICALLY DISORDERED SYSTEMS

Study of the effect of disorder on magnetic interactions in materials is of considerable fundamental interest. However, it may not have direct practical applications at the present stage of our knowledge. The disorder may originate from incomplete or random occupancy

of magnetic sites in a crystalline system, or from true topographic disorder such as is found in an amorphous metallic glassy material. The disorder often leads to competing ferromagnetic and antiferromagnetic exchange phenomena (spin frustration, etc.) and may have the effect of destroying long-range magnetic order, resulting in the formation of magnetic micro-domains. Magnetic phenomena in disordered systems are thus frequently in a size regime intermediate between nearest neighbor atomic dimensions and infinite range correlations, and can be studied by small angle neutron scattering (SANS) for amorphous systems, or by wide angle powder diffraction methods in the case of polycrystalline systems. The types of information that one obtains include spin correlation lengths, details of spin fluctuations above and below magnetic transition temperatures, etc.

In the class of disordered magnetic systems are many metallic glassy systems formed by rapid solidification (e.g. Fe_{90} Zr_{10}, Cr_x Fe_{1-x},), ferrofluids (suspensions of fine superparamagnetic particles in a liquid matrix) and magnetic nanostructures (e.g. Fe particles embedded in an immiscible metal host or ceramic).

The question of the existence of long range order in alloys versus a short-range or spin glass order can also be studied by neutron depolarization. While this is principally a qualitative technique, it does provide valuable information on the magnetic state of the system especially when studied in conjunction with other parameters such as the magnetization and Moessbauer spectra. More quantitative results can be obtained from diffuse elastic neutron scattering.

3.3. MAGNETIC THIN FILMS AND SUPERLATTICES

This is one of the areas of magnetism currently of considerable interest. The ability to produce thin films of arbitrary thickness, and to do atomic scale engineering of layered structures by molecular beam epitaxy (MBE) and sputtering, open up almost limitless possibilities to study exchange interactions, interface effects, and surface boundary conditions.

The materials can be studied by a number of neutron techniques including wide angle diffraction, SANS (if disorder is present in a specific system), and by inelastic scattering if sufficient material is available. With extremely high quality materials the technique of neutron reflectometry can provide a wealth of information about variation of the magnetic moment near an interface, details of the interface roughening, and the range of the magnetic interactions.

The greatest limitation to work in this field is the very strict quality requirements the measurements place on film materials. Thin films must be of good crystal quality, free of impurities, and uniform, to be of real use in neutron experiments. These are not trivial requirements and often require very expensive preparation laboratories and labour intensive techniques for both growth and proper characterization of the film materials. This is not an area to be entered into if major resources (both equipment and personnel) are not available for sample preparation.

3.4. CERAMIC (HIGH T_C) SUPERCONDUCTORS

These materials lend themselves well to neutron powder diffraction techniques because of the critical dependence of superconducting properties on the location and occupancy of oxygen sites which can be studied directly by neutron diffraction only. The materials are also

relatively easy to produce and can be studied from many different aspects — oxygen deficiencies, atomic site substitutions, bond lengths, etc.

3.5. TEXTURE AND RESIDUAL STRESS IN FABRICATED MATERIALS

Non-destructive evaluation of engineering materials by neutron diffraction is an important field of investigation of materials reliability. Residual stresses induced in localized regions during fabrication processes can lead to various deleterious effects like stress corrosion or brittle fracture.

Neutrons, being highly penetrating particles, can examine the bulk stress distributions by measuring strains in the materials. Careful wide angle diffraction measurements of the atomic d-spacing throughout small and controlled volumes of material near a weld or other strain-inducing deformation can provide the data to do a full three-dimensional analysis of the local stresses in a material and therefore an analysis of its potential for failure.

Powder diffraction techniques could be used to study texture in a variety of materials, like cast iron, rolled sheets, work-hardened stainless steels, etc. The anisotropy in physical properties is determined by the anisotropy of texture of the material under consideration, that is the relative orientation of crystallites in the material. Conversely material properties can be controlled by developing properly textured materials.

3.6. ZEOLITES

Zeolites are important catalysts in petrochemistry. Knowledge of how the absorbed molecules are attached and interact within the zeolite channels are of interest from a variety of points of view. Usually X rays provide the basic structural information. However, neutrons can "see" the hydrocarbon molecules in greater detail, and thus more useful knowledge can be obtained from neutron powder diffraction.

3.7. PETROGENIC MATERIALS

Studies of petrogenic rocks are important both for the oil industry and for geology. Information about porosity and the surface roughness of the petrogenic rocks are of interest for estimating the migration and formation process as well as to increase the efficiency of oil extraction and production. Much essential information for optimizing these processes can be obtained by measuring the fractal properties of the rocks using SANS techniques.

3.8. MINERALS, POLYMERS, AND AMORPHOUS SYSTEMS

Low and medium flux research reactors can be profitably used for characterization of a variety of magnetic and non-magnetic materials. By characterization it meant the determination of relative composition and identification of constituents, for example, minerals in a composite system by the use of neutron powder diffraction. The provision of this kind of support for mineralogical/geological analysis would be of interest to any reactor center.

For analysis of data one has to develop a data base of diffraction patterns of basic mineral constituents like MgO, CaO, MnO, Fe_2O_3, Fe_3O_4, SiO_2, etc. The mineral configuration can, in principle, be obtained by a multiphase or a multi-component Rietveld analysis.

Expanding the scope of these studies to fibrous materials like cotton, silk, or jute, would be of interest to examine the texture under various processing conditions to be able to select the best processing parameters.

Yet another system of materials are the solid solutions or systems which show amorphous structure. Depending on the concentration/composition of solid solutions, the material can show more "crystal-like" behavior or more "liquid-like" behavior. These structural aspects will govern various properties like conductivity, both thermal and electrical.

4. METHODS AND INSTRUMENTATION

4.1. OVERVIEW

In view of the importance to acquire familiarity with the use of neutrons as a research tool, at many of the existing low flux facilities, a well planned approach is needed. The strategy should be to start from relatively easy-to-implement methods, requiring little or no sophisticated equipment, and to proceed to more demanding ones as the competence of the team grows.

The following list provides an outline of available techniques, their impact on training programmes, and ultimately their application in regular R&D programmes. Of course, it is not mandatory to follow through all these steps to build up the required competence. This is presented only as a 'menu' to choose from, according to the particular situation and resources. For further details for each method, one has to consult detailed literature available in various publications.

4.2. METHODS

4.2.1. Transmission-type measurements

Techniques:
integrated transmission (cross-section) measurements
transmission imaging (radiography)
neutron depolarization studies

Training benefits:
use of neutrons in general
concept of cross section, spin and magnetic moment of neutrons
ways of attenuating neutron beams
detection methods
filter techniques

Uses:
transmission imaging (hydrogen distribution,
macroscopic structural inhomogeneities, porosities)
bulk magnetic properties
quality control

4.2.2. Measurements on Bragg-peaks

Techniques:
double-crystal small-angle scattering
(inverted) Fourier correlation method
static Debye-Waller factors (DWF)
texture determination

Training benefits: better understanding of neutron interaction with matter and scattering theory
higher precision work
more advanced data analysis

Uses: large structural defects (of the order of 1 μm)
microcracks in materials
fatigue and aging effects
polymer science
non-destructive testing
low hydrogen concentrations
strain (integral measure through line width or spatially resolved through line shift)
lattice distortions due to defects (integral measure through DWF)
effect of work process on materials (texture)

4.2.3. Determination of Bragg-intensities (diffraction)

Technique: two axis diffraction, with third axis to eliminate thermal diffuse scattering (TDS) and other spurious effects

Training benefits: science related to background, beam collimation and advanced filtering crystallography

Uses: crystal structure determination
effects of compositional changes

4.2.4. Direct beam small-angle scattering

Technique: using narrow collimated beam based on low resolution wavelength selection and position sensitive neutron detection

Training benefits: theory of scattering from small defects
(isotopic) contrast variation data analysis

Uses: defects in materials precipitations
large periodic structures
phase transformations

4.2.5. Diffuse-elastic scattering

Technique: extremely low-resolution "diffraction" between Bragg-peaks

Training benefits: elastic/inelastic scattering discrimination
order/disorder in materials

Uses: investigation of ordering in condensed matter
structure factors in liquids or quasi-liquids

4.2.6. Inelastic scattering using Be-filter technique

Technique: variation of incident energy with Be-filter in scattered beam to transmit only long-wavelength neutrons

Training benefits: inelastic scattering from materials
advanced background suppression

Uses: incoherent inelastic scattering spectrum from hydrogenous systems
phonon density of states, especially heterogeneous compounds
phonon dispersion of high frequency branches

4.2.7. Inelastic scattering using time-of-flight or crystal monochromators techniques

Technique: Time-of-flight (TOF) technique or triple axis neutron spectrometry

Training benefits: measurement of inelastic spectra
lattice and molecular dynamics
molecular, atomic, magnetic motion studies
crystal field effects

Uses: measurement of inelastic spectra from powders
phonon/magnon dispersion relation data
quasi-elastic scattering studies of reorientational motions
crystal field spectra

4.3. TECHNIQUES AND INSTRUMENTATION DETAILS

The methods listed above require an increasingly high degree of familiarity with both the techniques of neutron scattering and the understanding of the underlying theoretical principles as well as theoretical knowledge in condensed matter science.

They are also suitable for starting with simple equipment for data taking and analysis and proceed to more elaborate systems, building on a standard which should be introduced early on.

Most of the results that can be obtained are of practical importance in the development of a technological culture and an industrial base.

In what follows, the individual methods will be discussed in more detail.

4.3.1. Integral transmission measurements

Measuring the neutron transmission through samples in the direct beam is the simplest form of a neutron experiment. Very little equipment is required: neutron absorbing material (cadmium, B_4C-containing plastics) to limit the beam cross-section and divergence, a neutron monitor in front of the sample and a detector together with the appropriate counting electronics. In order to reduce the gamma-background, a filter should be installed in the beam. Adequate shielding around the equipment to protect personnel should be a common feature of all experimental arrangements.

Apart from basic training in shaping neutron beams and counting neutrons (adjusting the detector threshold, setting up a measuring arrangement, correcting for background) such measurements can be used to determine the content of highly absorbing materials (Hg, Cd, etc.) or strong scatterers (especially hydrogen) with a relatively high degree of precision. A well collimated arrangements can also be used to determine the degree of small-angle

scattering through its effect on the transmitted beam. Neutron measurements can also be carried out in a monochromated beam behind a crystal monochromator ("single-axis spectrometer") with the benefit of much reduced background and more quantitative results since the energy of the neutrons is known.

While such measurements will in general not justify the operation of the reactor in their own right, the technique is very valuable for initial training and can well be developed into a useful tool to determine the composition of specimens whose components are known qualitatively but not quantitatively, if they weaken the neutron flux differently.

4.3.2. Neutron transmission imaging (radiography)

Transmission imaging, frequently also called "radiography", with thermal neutron beams is one of the most straightforward applications of neutrons in materials research and testing. Since neutron flux densities of the order of about 10^5 neutrons/cm^2·s are sufficient to take radiographs in about 10 minutes, it can be done even at the smallest existing reactors. All one needs to perform such an experiment is a conical collimator (typical collimation angles for medium resolution work: 40') which provides for a uniform intensity distribution of neutrons emerging from a "point-like" source over the sample cross- sectional area (ranging in practice from just a few cm^2 to about half a m^2) and a film detector covered by a thin In foil. In the "direct" method the latter is necessary to produce conversion electrons which in turn lead to an exposure of the photographic emulsion of the film. In the "transfer method", which should be chosen in the presence of a large gamma background, one activates the In (or Dy) foil and subsequently transfers the activity image to the film, which then records the local variation of the emitted beta and gamma radiation. In high resolution radiography systems with adequately well collimated beams (around 10') spatial resolutions of the order of 10μm can easily be achieved if, in addition, a microdensitometric scanning procedure is applied to evaluate the radiographs more quantitatively.

There is an almost unlimited range of possible applications of this technique. At the high resolution limit it has been used, for instance, for the investigation of ^{3}H and ^{3}He bubbles in tritium-containing metals and the embrittlement problems associated with their formation [1]. Radiographs of large objects with low spatial resolution, on the other hand, are quite useful on an almost industrial scale. A good example of this kind is the investigation of the humidity transport and humidity distribution in building materials, which is hard to obtain with other methods. Even biological systems can be studied with neutron radiography. For instance, one could study the transport of water in plants by adding just a few percent of heavy water when watering the plant. There, one simply exploits the drastic difference of the total scattering cross-section of hydrogen and deuterium. This (large) isotopic contrast is not the only important advantage of neutron radiography as compared to radiography using X rays. Almost equally important is the ability of neutrons to penetrate thick objects without severe absorption.

In metallurgy, neutron radiography is a valuable non-destructive method. It can be applied as a high resolution neutron radiography technique capable of investigating the microstructure and composition of a metallurgical specimen both in a qualitative and a quantitative manner. This technique is called microneutronography. It can be used either for viewing the internal microstructures in the sample or possibly be applied in a quantitative manner for the determination of the internal composition of a specimen by means of microdensitometric recordings. An image is obtained by placing a thin specimen (100 μm to a few 1000 μm) in close contact with a suitable neutron imaging detector and exposing it to

a collimated beam of neutrons. The microneutronograph is then obtained by enlarging the primary image by means of a light microscope. The complementary method to microneutronography for the determination of boron distribution is neutron-induced autoradiography. In cases where a specimen contains elements like B, Li or U, which by itself emits charged particles upon neutron absorption, the presence of these elements can be revealed without any intermediate converter screen directly by detection of reaction particles with a suitable detector.

4.3.3. Diffuse elastic neutron scattering (DENS)

Diffuse elastic neutron scattering (DENS) spectrometers are in principle very poor resolution diffractometers with a wide range of application. These rank as low priority instruments in the sequence of introduction of the various experimental methods because:

- the cross-sections involved are much lower than for Bragg reflections, so the technique is more flux-limited,
- to compensate for low cross-sections it is necessary to use many detectors at different angles which makes such an instrument more expensive,
- thermal diffuse (phonon-inelastic) scattering and diffuse elastic scattering being of the same order of magnitude in many specimens, a coarse time-of-flight selection or energy analysis by other means is convenient but adds considerably to the complexity of the instrument.

DENS investigates the scattered intensity between the Bragg peaks which results from the presence of different kinds of constituents in a crystal. In a binary system the intensity is determined by the difference in scattering amplitude of the (two) constituents and their relative concentrations:

$$I \sim c_A.(1 - c_A).(b_A - b_B)^2$$

This intensity is modulated as a function of momentum transfer **Q** in characteristic ways if the distribution of the atoms on the lattice sites deviates from random, i.e. different temperature treatments of materials can give information on the effect of this treatment on the microstructural constitution of the material, or it can be used to analyze its state, if the treatment is not known. If the method is used for non-destructive testing, it may allow to characterize specimens either on the basis of experience or on the basis of a theoretical interpretation of the scattering pattern.

DENS spectrometers, when using a sufficiently short wavelength, are also suitable for the investigation of liquid structure factors at low **Q**. They will, however, not cover a wide enough Q-range to avoid truncation effects in a Fourier inversion to get radial distribution function.

The equipment needed is, apart from the usual beam shaping, shielding and monitoring, mainly a coarse monochromator (mechanical velocity selector or double crystal monochromator), a low resolution beam chopper (for time-of-flight) or large mosaic analyzer crystals in front of the detectors.

If crystal analyzers are used the range of incident wavelength will be limited unless substantial mechanical effort is provided to equip the analyzers with variable energy adjustment possibilities. If time-of-flight is used, a low resolution multichannel time-of-flight

analysis should be performed, requiring a multidetector time-of-flight equipment. Alternatively simple gating of the detectors to the time intervals when the elastically scattered neutrons arrive, may be a cheaper way but this limits the possibility of correcting for inelastic contributions to the measured intensities.

While being of somewhat higher complexity, DENS is a very useful tool in materials science and technology.

4.3.4. Neutron depolarization

Although the production of polarized neutrons is inevitably associated with a loss in intensity of at least about one order of magnitude, one can use the polarized neutrons for some special purposes even at low flux reactors. In particular, neutron depolarization, which is conceptually a simple transmission technique, is quite a versatile and sensitive tool to study magnetic structures on mesoscopic scale.

In its simplest way of realization, one places the specimen under investigation between a polarizer and an analyzer (which, for thermal neutrons of wavelength of less than 2.5 A°, are preferably magnetically saturated Heusler crystals; for longer wavelengths polarizing supermirrors are the appropriate choice) and measures the variation of the degree of neutron polarization on transmission through the sample by observing the change of the intensity behind the analyzer. From a very simple algorithm, which dates back to 1941 [2], one can then derive the mean size of the magnetic domains within the sample. In many respects this method resembles the investigation of transparent objects by placing them between polarizing optical filters.

With a somewhat more sophisticated equipment, which essentially involves a 3-dimensional analysis of the neutron polarization [3], one can extract more information about the domain structure, as e.g. the mean square direction cosines of the domain magnetizations, the mean macroscopic magnetization of the sample and the presence of various types of correlations between adjacent domains. The main drawback of this technique, which has probably prevented it from being widely used, is the lack of simple, generally applicable evaluation algorithms to extract the domain structure information from the measured data of such a 3-dimensional neutron depolarization experiment. Until now one needs supplementary information from other techniques (magnetization measurements, neutron small angle scattering, electron microscopy etc.) to correctly interpret the experimental results in a quantitative way.

Its big advantage is its very high sensitivity to the presence of magnetic long range order in the sample, which scales up exponentially with the square of the wavelength, and — since it is a transmission technique — its applicability at even very low neutron fluxes. Even at a reactor of typically 100–200 kW it allows one to study the domain structure of magnetic materials on a "real time" scale, that means it is possible to detect magnetic relaxation effects of the domain structure after periodic excitation by an external magnetic field with a time scale of several μs up to times which are only limited by the available operation periods of the reactor itself. For relaxation time-constants larger than about 1 minute, it is, of course, even not necessary to vary the sample environment repeatedly. The high sensitivity to magnetic inhomogeneities makes the neutron depolarization method also very well suited for the investigation of magnetic phase transitions.

4.3.5. Small-angle neutron scattering

Small-angle neutron scattering (SANS) is an important tool to reveal the microstructure and macroscopic properties of materials. It occurs whenever the inhomogeneities of 10–1000 A° are present in any condensed substance- for example in the presence of pores or precipitates in a matrix, macromolecules in solution, grain boundaries, submicron-cracks etc. These are important in homogeneities to characterize the microstructure of most materials: metallic, polymeric, composite, biological and geologic. The SANS technique provides information on the average size and number of inhomogeneities and also on their shape. Systematic SANS studies can also be carried out to optimize the processing of new materials.

It is fortunate that this technique, with such a wide range of applications, can be implemented at a modest flux reactor. This is possible because:

- only a roughly monochromatic beam of neutrons is required, which means that a relatively wide slice of the neutron spectrum is actually used in the measurement;
- area position sensitive detectors are commercially available which can collect the scattering (at typically 4000 individual detector channels) simultaneously which is equivalent to increase the reactor flux by a few orders of magnitude; and
- simple multichannel collimators can be utilized in the incident flight path to make effective use of the full source area maintaining the small required divergence of the incident beam. For instance, a channel collimator with 4 × 4 channels is equivalent to having 16 SANS machines in parallel carrying out the same measurement simultaneously, which means a gain in neutron flux of more than one order of magnitude. A further gain of 4 to 5 could be achieved through the use of a cold moderator such as methane cooled down to liquid nitrogen temperature. This should be considered as a possible future development of a SANS facility.

As far as investment prices are concerned the most expensive components are the area position sensitive detector and the velocity selector for rough monochromatization. The latter costs about US $10 000 whereas the detector costs about US $120 000 or US $220 000 for effective areas of 25 × 25 cm^2 or 64 × 64 cm^2, respectively, both at a pixel width of 1 cm. The larger detector has a count rate which is 5 times higher than that of the smaller one for the same measurement and the same neutron source. The remaining components can be constructed locally if a sufficiently well equipped mechanical workshop is available.

(Note: The costs and sources in some cases from where some of the components can be commercially procured *only* as examples. However these are not to be construed as recommendations, the only suppliers or fixed authentic prices.)

A well performing SANS facility for research at a modest flux reactor would thus require the largest available area position-sensitive detector, the velocity selector and an optimized design with a multichannel collimator which can be built locally. It should ideally be installed at a tangential beam tube to avoid high levels of gamma-radiation and fast neutrons. In this case a scatterer has to be placed inside the beam tube which in a first stage could be a water scatterer at ambient temperature. At a later date, this could be replaced by a cooled moderator such as methane. In any case the scatterer can also be constructed locally.

A less expensive SANS using a single linear position sensitive detector for modest research could be built using a BeO filter acting as a coarse monochromator. The same setup

is upgradeable by replacing the linear position sensitive detector with a large 2-dimensional area detector as outlined above. Such a simple SANS has been in operation at DHRUVA reactor in India for research in a variety of surfactants, ferrofluids, etc.

4.3.6. Double-crystal SANS

To study inhomogeneities, at the length scale of 1 μm (10^4 A°) such as precipitates and cracks, a simpler and less expensive SANS instrument can be built. It is based on the non-dispersive arrangement of two perfect crystals. The principle of the measurement is to analyze the width of a perfect crystal reflection with the help of a second perfect crystal. Changes of the width introduced by a specimen in the beam path which causes small-angle scattering can thus be determined with high precision.

The main components of this instrument consist of two "channel-cut" perfect silicon crystals, one of them fixed, acting as monochromator, while the other is placed on top of a precise goniometer and rocked through the entire reflection, and a standard neutron detector at a fixed position. The channel cut silicon crystals can be obtained with the assistance of the Atominstitut, Vienna at quite moderate costs, the goniometer can be such as the one used for standard triangulation purposes and a neutron detector at a cost of about US $1000.

This is an interesting instrument to install at a modest flux reactor because neutron flux can be gained effectively by using a rather divergent beam incident upon the monochromator set. Due to the fixed scattering geometry, SANS measurements can easily be carried out at different temperatures, different surrounding atmospheres, with external magnetic and/or electric fields, different pressures or tensile stresses, etc.

Thus the SANS technique applied either to the length scale of 1 μm or to 0.1–0.001 μm, offers an almost unlimited field of applications for materials testing and for both applied and basic research.

4.3.7. Two axis (powder) diffractometry

Two axis diffractometers (one axis at the monochromator for variable incident wavelength and one axis at the sample for variable scattering angle) are the most widely used instruments for crystal structure determination and for investigation of a variety of phenomena that affect the crystal structure. As a domestic tool, training device and for research, they are a valuable asset in every reactor neutron scattering outfit.

Neutrons from a (filtered) beam are monochromated with a crystal monochromator. In designing the diffractometer, take-off angles of 90° or more should be chosen to keep the option for improved resolution open, as experience and demand increase. Also, the possibility of inserting collimators in front of the monochromator and between monochromator and sample must be foreseen, even if initially only a relatively coarse collimation is used for intensity reasons.

In its simplest form, a well shielded detector is moved around the polycrystalline sample to scan the positions of Bragg reflections from the specimen. This should be automated and requires mechanical gears of fairly high precision (at least 1/10 of a degree). It is of course an essential advantage and helps to overcome flux limitations in the reactor, if measurement is possible at many scattering angles simultaneously. This can be done by using an array of many detectors or by going to position sensitive detectors. If many

detectors are used, corrections for sensitivity may be necessary but can be performed with the help of standard incoherent scatterers. Position sensitive detectors are available as linear ones, which are relatively cheap but count-rate-limited and require some corrections for scattering angle and resolution, or as curved ones which are rather expensive.

Although two axis diffractometers are comparatively simple to build and to operate, they have a very wide range of applications. It is possible to start with a rather simple setup of moderate resolution for testing and training purposes and improve the performance of the instrument by exchanging parts of the spectrometers.

Air cushion technology is not necessary to move the detector arm but is very advantageous to increase the instrument's flexibility (e.g. by varying the sample-detector distance, improving the precision of positioning, etc.). It also allows to add a third axis to the instrument. On a low flux reactor the main reason for doing so would be to reduce substantially parasitic contributions to the scattering by adjusting the analyzer to the elastic scattering from the sample. In favorable cases such an instrument may be used to measure also inelastic scattering.

4.3.8. Fourier time-of-flight (TOF) neutron diffraction

Time-of-flight neutron spectroscopy of white chopped neutron beams is a standard method for high resolution powder diffractometry. However, with conventional single-slit neutron choppers it suffers from a bad neutron utilization at steady neutron sources because of the low chopper duty cycle.

With the so-called "Fourier-TOF" technique, introduced by Colwell et al. in 1969 [4], the situation is quite different. Here one does not measure the flight time of individual neutrons but modulates instead the incident neutron beam periodically in time with a series of discrete frequencies by means of a multislit disk chopper in combination with a similarly equally designed stator and records by means of a phase sensitive detection system the relative decrease of the modulation depth and the phase shift of the observed intensity oscillations with respect to those produced by the chopper. By doing so one effectively measures for each of these frequencies the Fourier amplitudes and phases of the time-of-flight spectrum, which can then easily be calculated by proper Fourier inversion.

The essential advantage of this technique is the high duty cycle of the Fourier chopper, which is 0.25 due to the 50% opening time and the 50% area losses of the rotor-stator combination. Usually this technique was intended for inelastic scattering work, but it finally turned out that with such a correlation procedure the absolute statistical error of all calculated points of the TOF spectrum is the same and hence only those parts of the time-of-flight spectrum which lie above the mean intensity can be determined more accurately within a given measuring time than with conventional TOF. Clearly this means that the elastic scattering dominates the much smaller inelastic peaks which consequently suffer from large relative statistical errors. For neutron diffraction, however, where one is interested in the elastic peaks only the Fourier method may be of extreme advantage and is surely worthy to be applied more often than it is up to now. The experimental requirements of this technique are very moderate and can be implemented practically at every laboratory irrespective of the level of sophistication of other techniques already in use.

In a modified version of this technique, the so-called "Inverted Fourier-TOF Method" invented by Hiismaki et al. [5] in Finland one can even achieve an on-line measurement of

the time-of-flight spectrum without the need of explicit Fourier-inversion of the data. The additional hardware requirements of this special technique are negligible. Since one might want to preserve also the possibility to measure the Fourier components of the spectrum, one could easily design the spectrometer as to operate either in the "normal" or in the "inverted" mode, virtually without any significant extra costs to provide for this flexibility.

4.3.9. Inelastic scattering of neutrons

(a) T-O-F Method using a rotating crystal monochromator

Through neutron inelastic scattering experiments one has gained detailed information on phonon spectra and dispersion curves, phonon lifetimes, thermal diffusion of atoms etc., especially in the case when a sample contains atoms with a high incoherent or coherent scattering cross-section for neutrons.

When hydrogen atoms are present in the molecule, neutrons are scattered mainly by these atoms, providing relatively easy interpretation. The magnitude of the energy transfer involved in these phenomena is about 0.1 to 100 meV, which corresponds to interaction times of 10^{-11} to 10^{-13} s. (Ultrasonic and dielectric relaxation methods give information on a time scale of 10^{-7} s and in nuclear magnetic resonance (NMR) measurements it is possible to reach a time scale of about 10^{-11} s. A time scale of about 10^{-12} s can be reached with infrared and Raman scattering but as the wavelength of the electromagnetic radiation is large, the momentum transfer is always almost zero).

The incoherent inelastic scattering spectra can be measured using cold neutron (cold neutrons have an energy lower than 5 meV) energy gain processes by a time-of-flight (TOF) method. As an example, at the 250 kW research reactor TRIGA in Ljubljana (Slovenia) with a maximum thermal neutron flux of 10^{13}n cm^{-2} s^{-1} a pulsed beam of almost monoenergetic neutrons obtained by means of a rotating lead monocrystal (λ_0 = 4.06 A°) is scattered by a sample into a bank of ^{3}He neutron detectors arranged at 4 different scattering angles to the incident beam direction at distance of 2-3 m from the sample. In the beam port a beryllium filter at liquid nitrogen temperature is placed (10 cm x 8 cm x 30 cm). The beryllium filter transmitting only the part of the neutron spectrum below the Bragg cut-off (5.2 meV) helps to reduce the background and provide a coarsely-monochromated neutron beam. The energy spectrum of the scattered neutrons is analyzed by measuring the time-of-flight of the scattered neutrons from the rotating crystal to the detector by means of a multichannel analyzer. The momentum transfer in the collision depends on the scattering angle. Because we are faced with the problem that the flux of neutrons in the low energy tail of the thermal spectrum is quite small in a low to medium flux reactor, a shift of the Maxwellian distribution to longer wavelength by means of a low cost liquid nitrogen cooled moderator is of great advantage. For neutrons of 4 A° wavelength an increase of the flux by a factor of 4 is achieved in comparison with an equivalent water moderator if liquid methane 110 K is used as moderator substance. Such a liquid methane moderator can be a useful source of cold neutrons on research reactors with a power of up to 1 MW [6].

Together with the nitrogen cooled methane moderator the rotating crystal time-of-flight spectrometer represents a very valuable experimental tool for research in solid state physics, chemistry or biology. To build such an instrument is not an extensive task because only a monocrystal, a bank of ^{3}He detectors and a multichannel analyzer are required besides some vacuum pumps and proper shielding materials.

(b) Inelastic scattering using a triple axis neutron spectrometer

Analysis of energy of monochromatic neutrons scattered by a sample can be achieved using an analysing crystal and a detector. The instrument used is a Triple Axis Neutron Spectrometer, which is the workhorse at several medium and high flux reactors. In a Triple Axis Neutron Spectrometer, a monochromator is mounted on axis I, sample at axis II, and an analyser at axis III. This is followed by a detector and a monitor in the monochromatic beam. The data acquisition system can be a PC-based motor control system and a simple counting system. This allows to control various angular devices, independently of the instrument. Since no T-O-F technique is used, a multi-channel system or a device like a rotating chopper are not required. The TAS can be built as a natural upgrade of a two-axis diffractometer and can be used for a variety of purposes:

- as an 'elastic' diffractometer when the analyser wavelength is the same as 'incident wavelength',
- to study phonon or magnon density of states from powders,
- to study crystal field excitations from powders,
- to study dispersion relation of phonons or magnons using single crystal samples and
- to study small angle scattering at any incident wavelength by positioning the sample in between two 'channel-cut' single crystals one situated at the II axis and another at the III axis.

The instrument has the unique advantage of being able to measure $S(Q,\omega)$ the scattered neutron intensity at any momentum transfer and any energy transfer as required. Hence, for dispersion relation measurements or for obtaining $S(Q,\omega)$, say, for a liquid, one can have 'constant-Q' scan or 'constant-ω' scan or any general scan along any direction in (Q,ω) space.

By replacing the single crystal analyser by a long analysing crystal and the conventional $^{10}BF_3$ or ^3He detectors by a linear position sensitive detector, one can improve the throughput manyfold. So also, one can design specific variations of the instrument for multi-analysis purposes or for converting the instrument for a 'filter-detector' spectrometer.

(c) Inelastic scattering using Be-filter technique

A two-axis spectrometer (or a TAS suitably modified) can be used for inelastic scattering studies if one imposes a filter between the sample and the detector. The filter could be a polycrystalline Be or BeO block (having cut offs at 5 meV or 3.5 meV respectively) and neutrons can be detected in the conventional mode after getting transmitted through the filter or a Be-BeO combination energy window (of 0.5 meV around 3.7 meV) if the detector is replaced by an annular detector around the filter combination to accept the scattered neutrons from the filter combination.

Since the filters provide a coarse fixed energy analysis window, inelastic spectra from samples are measured by varying the incident energy of neutrons from the monochromator. So unlike a diffractometer, where the incident energy is held fixed and only an angular distribution of scattered neutrons is measured by rotating the detector about the sample, here the geometry of scattering angle is generally held fixed at 90° and the entire spectrometer is moved around the monochromator to change incident energy.

The throughput of the instrument is better than that of a TAS and excitation energies from various compounds can be easily measured. However one cannot select a specific point in (Q,ω) space.

4.4. DATA ACQUISITION AND CONTROL

Since the fields of electronics in general and small computers in particular are under extremely rapid development, on the one hand, and the need for data acquisition and control capabilities, on the other hand, may increase as the spectrometers develop at a given reactor, it is important to adopt a strategy which allows a modular architecture based on established standards which can be expected to be around and supported also in the future. This, at the same time, ensures compatibility between hardware and software on a broad scale. Important advantages of such a strategy are:

- efficient use of the available technical staff,
- interchangeability of modules between different instruments,
- expandability on the basis of locally proven and established components.

4.4.1. Requirements for a modest structure

Use of a PC to control the experiment should be aimed at in all cases. For a system of limited capabilities it may be sufficient to work with add-on boards on the motherboard. Many different variations of such add-on boards are available serving a large range of needs, such as motor control, temperature regulation, etc. Many of them come with good standardized software and can be programmed in BASIC. Interfacing of external electronic equipment can also be done via add-on boards which provide multiple TTL and/or analog input/output (I/O) ports.

4.4.2. Requirements for multiple or more complex structures

As the need for control functions increases with increasing functionality of the instrument, space for add-on boards in the computer will not be sufficient, nor will there be enough specialized boards available. Nevertheless, PC control of the spectrometers is still possible but external devices should be connected. They can control the fundamental functions of the experiment in a dedicated way such as pulse counting, positioning of components, control of the sample environment such as temperature, pressure etc. Also data acquisition from position sensitive detectors can be accomplished by such external modules.

The modular nature of such a system allows to decouple the various functions completely and provides for a large degree of flexibility in testing, maintenance and interchange of compounds between different instruments.

4.4.3. Standard of communication

The modular nature of a control system relies crucially on the use of a well established and widely supported standard for the computer and the external devices. Standards are commercially available, and widely used systems should be preferred. Such standards are RS232 or IEEE488, an industrial standard also called GPIB (General Purpose Interface Bus). We recommend to settle for these. GPIB has a high data transfer rate and allows multiple connections on the same cable. The cost of commercial products with GPIB standard is sometimes rather high, but, as long as the standard is respected, home-made solutions also can be incorporated. Such solutions may be available at other laboratories and information interchange should be encouraged, e.g. through brief reports in "Neutron News", etc.

With the electronic functions of the 'home-made' device realized on a printed circuit board, it must be connected to a central processor unit (CPU) board by TTL input-output

ports. The CPU board can be external to the computer but it must be equipped with IEEE488 and/or RS232 interfacing and its PROMs (programmable read-only-memories) must contain the resident command interpreter specially written for the respective application. In this way the device can be tested and developed using any computer and is also portable to other instruments or laboratories.

5. CONCLUSIONS

When considering the utilization of a research reactor (independently of the original motivation for acquiring such a facility) one should realize that, in a developing country, it is often the largest single facility for scientific R&D. Its utilization is usually limited by severe constraints related to financial, material and human resources. Therefore, most operators are satisfied if a reactor is operated with moderate success. Even in this situation, it can make a significant contribution to what may be called the 'scientific and technical culture' at the national level.

Nuclear techniques have a wide range of multidisciplinary applications and can have significant economical impact in several areas. The production of radioisotopes for medical and industrial purposes, environmental studies, assessment of impact of industrial activities, acquisition of know-how related to nuclear power, etc., are some of the common examples. It is therefore understandable why the number of research reactors is steadily increasing in the developing countries. On the other hand efforts are needed to enhance research reactor utilization beyond the simply 'satisfactory' level in order to make better use of the corresponding investment, human and material.

The report provides guidelines for initiating new programmes in neutron beam research and for upgrading ongoing activities in this field. It will be useful for defining appropriate research programmes, and to make more cost effective use of limited financial and human resources, the available equipment and the research reactor itself.

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Annex

PAPERS PRESENTED AT THE ADVISORY GROUP MEETING

FUNDAMENTAL AND APPLIED NEUTRON RESEARCH AT THE 250 kW TRIGA REACTOR VIENNA

G. BADUREK
Institut für Kernphysik,
Technische Universität,
Vienna, Austria

Abstract

A review is given about the research activities at the 250 kW Vienna university low flux reactor, which are adequate for implementation at other neutron souces of comparable size. The topics selected for presentation range from neutron radiography, materials irradiation, neutron small-angle scattering, neutron activation analysis, neutron depolarization in magnetic materials to neutron diffraction experiments for structure investigations of condensed matter.

1. Introduction

As it is the aim of the present meeting to stimulate programs for more efficient use of low power nuclear research reactors with neutron flux densities of the order of 10^{13} cm^2 at the center of the reactor core, we briefly describe the experimental facilities installed at the 250 kW TRIGA reactor of the Austrian Universities in Vienna (Fig. 1) and present a summary of a great part of the current research activities performed with them. We believe that most of the techniques and experiments presented here are adequate for implementation at other reactors of similar or even higher power. Those technologies which require extremely spezialized know-how not generally available at every research reactor institution will not be treated here or are just mentioned without any further details.

It is common knowledge that due to the relatively low neutron fluxes of such reactors one of the most important applications of neutron scattering on condensed matter, namely the study of atomic and molecular dynamics of solids and liquids, *a priori* must remain out of consideration. However, this does not mean that it is not possible to develop new or to improve existing techniques for such experiments at low power research reactors. In fact such developing work has always been a crucial point of our research efforts in the variety of fields of applied and fundamental neutron physics we were engaged in. But for the sake of conciseness here we will concentrate us just to a "grosso modo" description of the instruments and methods currently available at our institution. The order in which the individual topics will be treated is roughly correlated with both their growing experimental difficulty and their increasingly less general applicability.

2. Neutron Radiography

Neutron radiography is one of the most typical applications of small reactors because of several reasons. Firstly, it allows to perform useful experiments even with moderate

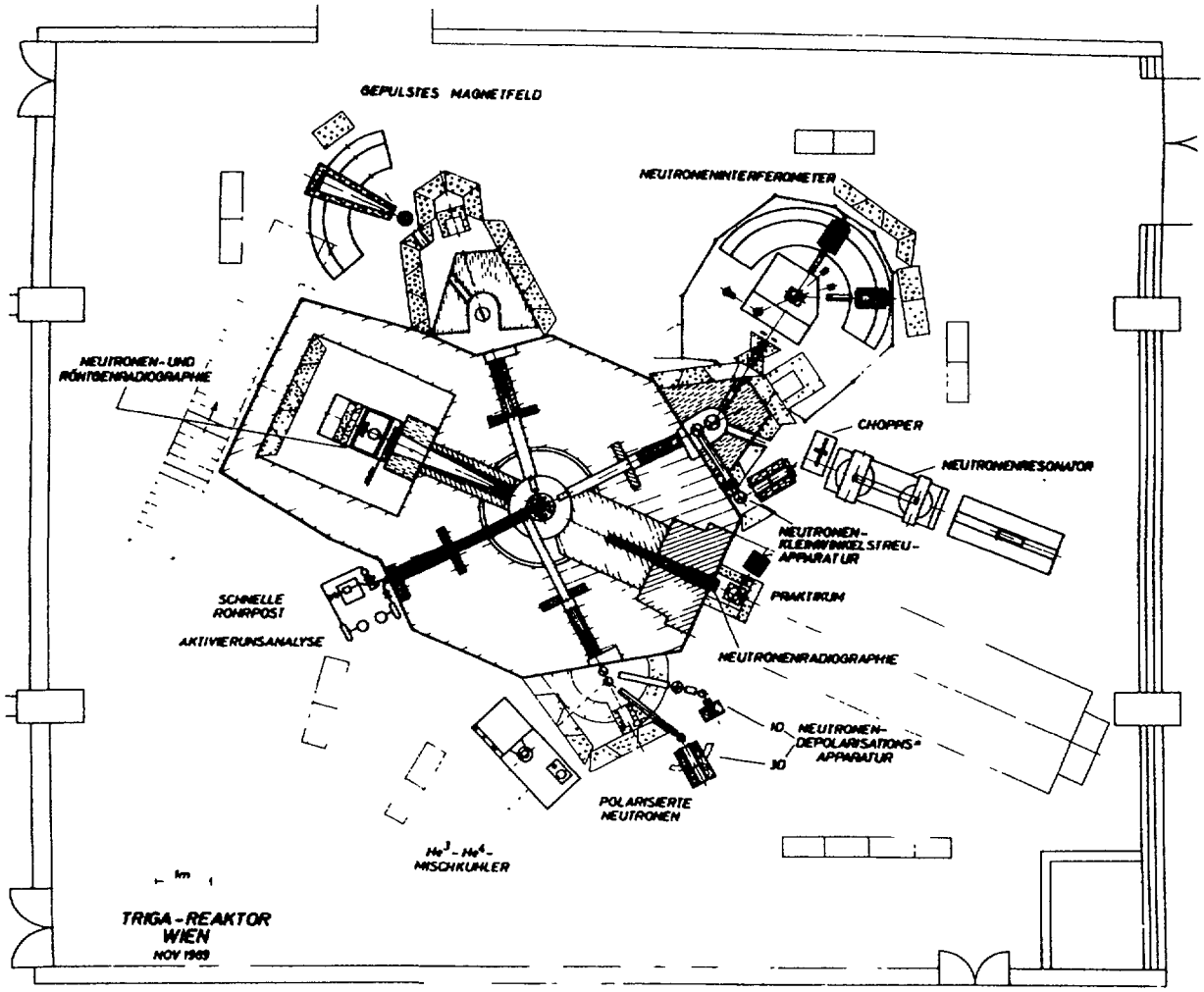

Fig. 1: Sketch of the experimental facilities installed at the Vienna University reactor.

neutron fluxes. Secondly, due to its conceptual simplicity it can be realized quickly without the need of extremely specialized and trained personnel. And last but not least, it can be considered as an immediate and almost always profitable step towards material oriented research.

Two radiographic facilities exist on the TRIGA Vienna, one is installed at the thermal column and mainly used for high resolution investigations of small samples not larger than about 9 cm in diameter. The other serves for inspection of larger objects (d_{max} = 35 cm) on an industrial scale (Fig.2). Conical collimators are installed at both facilities to produce uniform collimations and intensity profiles. The gamma background of the reactor is reduced by means of bismuth filters (polycrystalline, 4 cm at system 1; single-crystalline, 14 cm at system 2). The walls of the collimators are coated with materials containing boron and lithium to suppress (n,γ)-radiation. The geometrical beam collimation, the neutron flux density at the sample position and the gamma dose rate are as follows:

- system 1: 26′, 1.5×10^5 cm^{-2} s^{-1} , 0.02 Gy h^{-1}
- system 2: 75′, 2.6×10^5 cm^{-2} s^{-1} , 0.1 Gy h^{-1}.

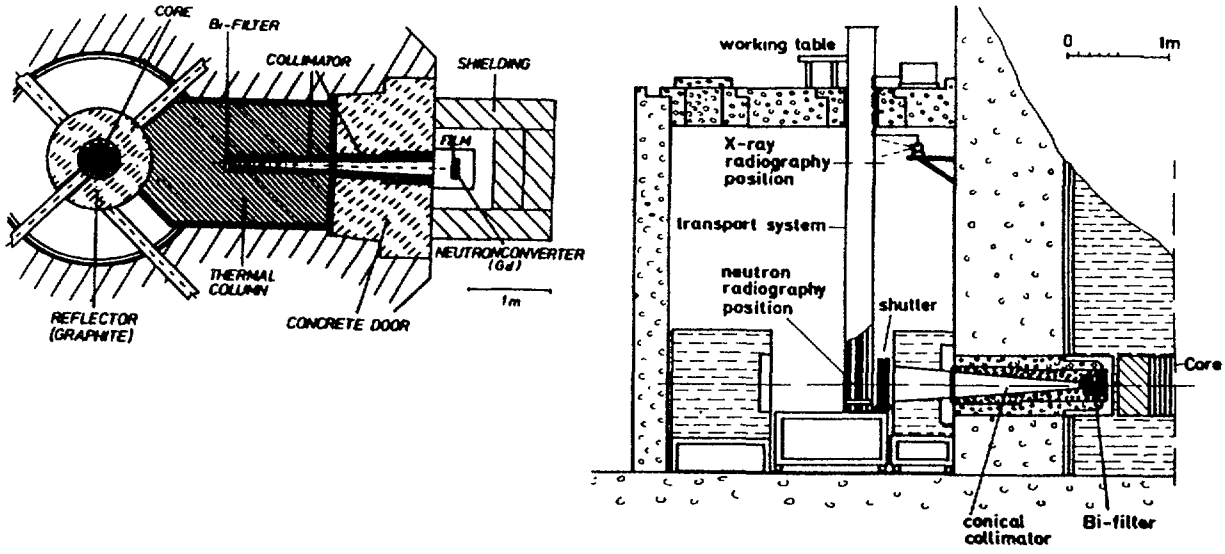

Fig. 2: Cross-sectional view of the two radiagraphy facilities.

The exposure times are of the order of 10-20 minutes. The darkening of the radiographs obtained by the usual gadolinium converter film technique due to the gamma background is always less than 20%.

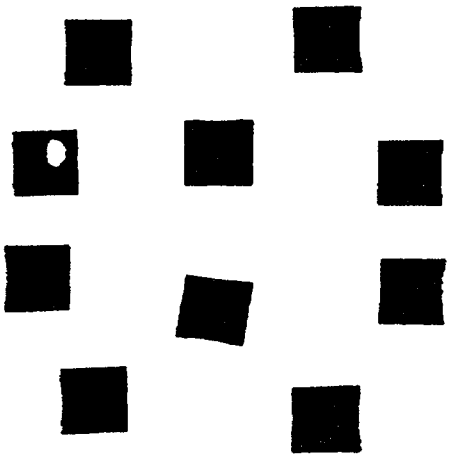

Fig. 3: Radiogaphs of electronic capacitors revealing some structural defects of the cast epoxy resin.

As a typical result obtained with system 1 Fig. 3 shows radiograpic images of a set of electronic multilayer capacitors. These capacitors were indended for the German "Meteosat" weather-satellite project and we had to be investigated for eventual structural defects. In some of these capacitors air bubble inclusion is clearly seen on the radiographs, which with high degree of probably would have led to a malfunction of the satellite electronics at the orbit. Two sets each consisting of 250 capacitors were checked, almost 40% of the first set were found to have some obvious defects. The second set was found to be 100% free of defects. One of the more recent projects was the investigation of ^{3}H and ^{3}He bubbles in various metals containing tritium, which is interesting in particular with respect to the embrittlement of structure materials of future nuclear fusion reactors [1]. There the radiographic films subsequently were evaluated more quantitatively by microdensitometric scanning. The achieved spatial resolution was about 16 μm. On the other hand one of the typical applications of the large scale radiography facility was the study of the humidity transport in bricks and other building materials.

3. Materials Irradiation

It is well known that the physical properties of many materials change upon neutron irradiation. In general this is of disadvantage but there are also some examples of profitable

use of this effect, in particular for semiconductors and superconducing materials. Controlled neutron irradiation of semiconductors can be used to modify their doping profiles, whereas irradiated superconductors exhibit inreased critical current densities compared to their initial state. In many cases this increase of the critical current, which is still one weak point of presently available ceramic HTC superconductors, reaches more than one order of magnitude. There neutron irradiation at different temperatures produces different types of pinning centers, which cause in turn a variety of changes of the superconducting properties that can be either stable or exhibit relaxational behaviour. In any case such investigations can help in our basic understanding of ceramic superconductors, but are also of importance for increasing their performance. The low temperature group of our institute has spezialized to this kind of irradiation investigations of superconductors, which are also of relevance for the development of superconductors sustaining the extremely high neutron radiation dose rates in fusion reactors. An example of the increase of the critical current of a HTC superconductor by neutron irradition is given in Fig. 4, which also shows for comparison the complete independence of the critical current of a sputtered NbN thin-film sample on the neutron fluence [2].

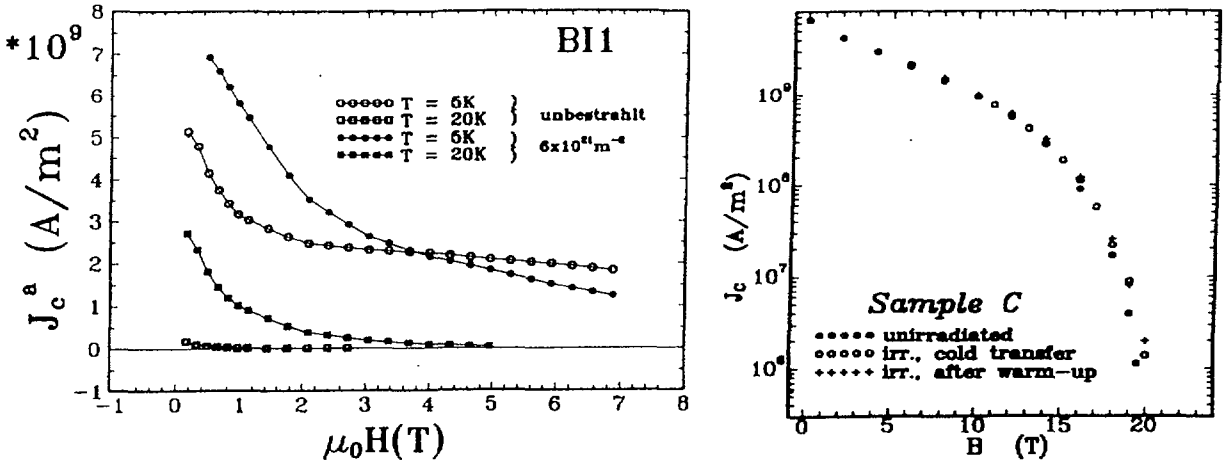

Fig. 4: (*l*) Increase of the critical current of a HTC sample upon neutron irradiation. (*r*) Irradiation independence of J_C of a NbN film.

4. Neutron Small-Angle Scattering

Standard neutron small-angle scattering (SANS) facilities are large instruments, which often are installed at the end positions of cold neutron beam guides of high and medium flux reactors, which usually are equipped with a Cold Source providing long wavelength neutrons. On the other hand, with the small-angle camera based on the non-dispersive arrangement of two perfect crystals, which is shown schematically in Fig. 5, one can achieve with thermal neutrons an extremely high angular resolution of a few seconds of arc, corresponding to momentum transfers of about 10^{-4} Å^{-1} which is about two orders of magnitude better than that of standard SANS instruments [3]. Since the angular resolution of such a small-angle camera is completely decoupled from the divergence of the incident beam

a rather divergent beam can be used, providing sufficient intensity at medium and even low flux reactors. To suppress the disturbing side tails of the resolution function by multiple reflection we have equipped the camera with two "channel-cut" perfect silicon crystals as indicated in Fig. 5. The compactness of this kind of small-angle camera allows to perform real-time experiments to investigate for instance ageing and relaxation effects in the samples under investigation. This technique allows to study the formation of any kind of inhomogeneities, as e.g. preciptitates or cracks, with spatial dimensions of up to about 1 μm. Measurements at different temperatures, different surrounding atmospheres, with external magnetic and/or electric fields, different pressures or tensile stresses etc. can easily be

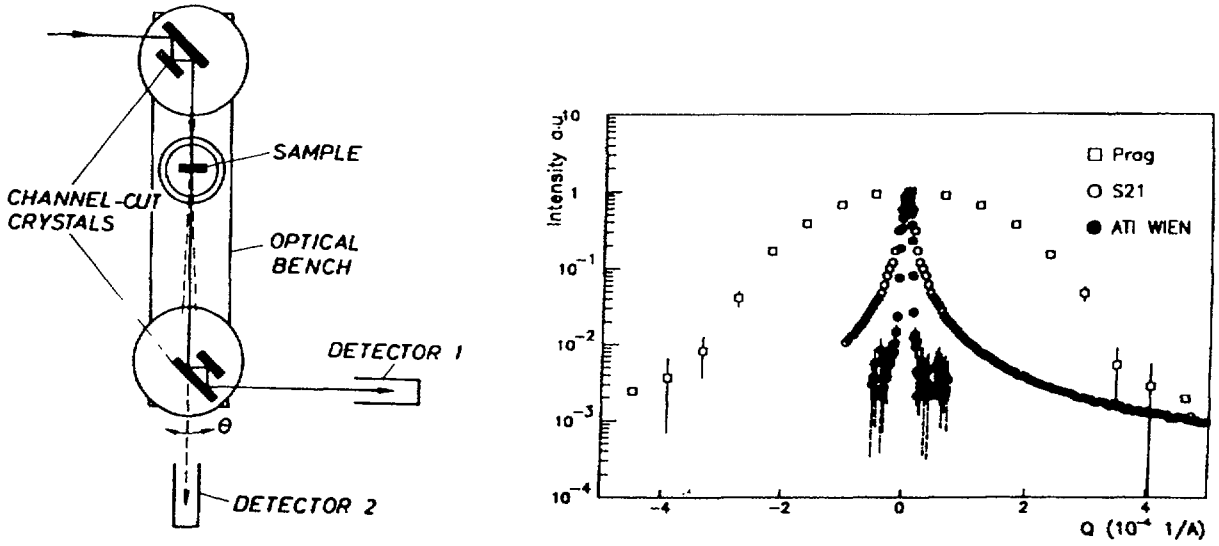

Fig. 5: Sketch of our perfect crystal SANS camera and comparison of its momentum resolution with that of two similar systems in Prague and Grenoble.

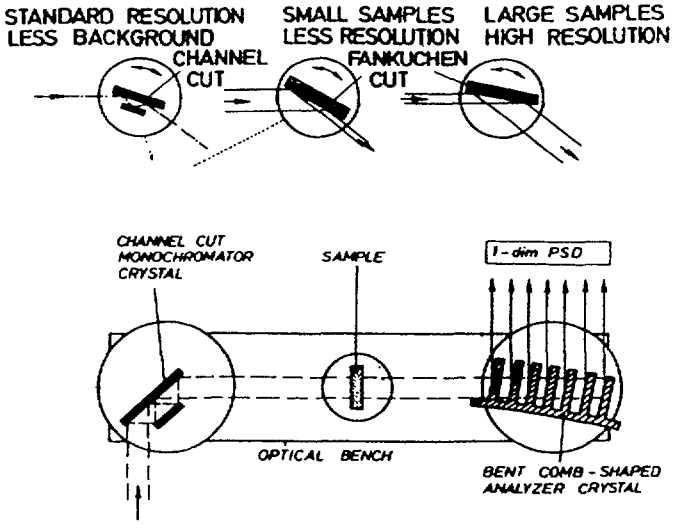

Fig. 6: Possible modifications of the standard small-angle geometry for more efficient use of the available neutron intensity.

accomplished and thus offer an almost unlimited field of applications both for applied materials testing and basic research. As indicated in Fig. 6 one could modify the scattering geometry of this camera according to the special requirements of the sample system. And by replacing the standard channel-cut analyzer by a bent comb-shaped crystal it should be possible in combination with a position sensitive detector to measure the angular distrubution of the transmitted beam simultaneously without resorting to the usual rocking curve measurement [4].

5. Neutron Activation Analysis

Besides of production of short-lived radionuclides partly for commercial purposes and partly for other scientific institutions the main application of the Vienna reactor in the field of radiochemistry is the neutron activation analysis of a broad variety of sample systems. Applications reach from the detection of environmental pollution, the analysis of filter dusts of fossile power plants, trace element analysis of geologic materials in search of mineral deposits to - for instance - the authenticity check of antique objects. An example of this latter application is shown in Fig. 7, where it can be seen that in the falsification of an antique copper coin in contrast to the genuine one, dating back to the ancient Roman empire, the alloy contains no noble metals. Two pneumatic transfer systems have been installed at our reactor for the purpose of neutron activation analysis. A slow one with transfer times of several seconds and a fast one with a transfer time of the sample from the reactor core to the measurement position of only 120 ms, which allows for the detection of radionuclides with short decay times of just a few milliseconds. Whereas the slow standard transfer system is easy to implement at any reactor the fast one deserves the development of special electronic equipment to handle the extremely high gamma-count rates associated with such short-lived radionuclides [5].

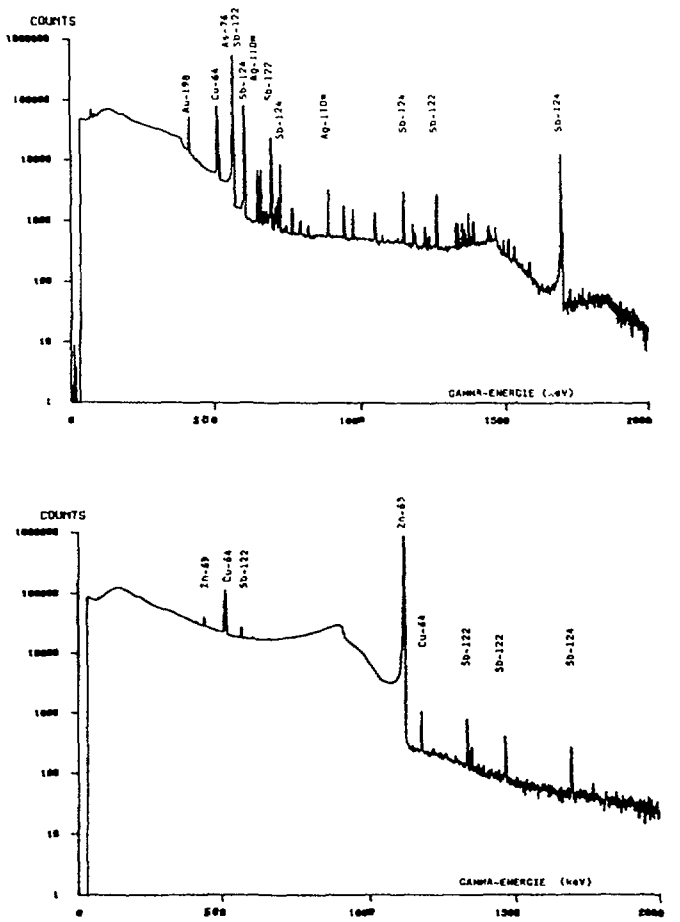

Fig. 7: Authencity check of an antique coin. In the falsificate (*below*) the noble metals are missing.

6. Neutron Depolarization

Its origin dating back in the early 'fourties neutron depolarization is a quite well established technique to study the domain structure of magnetically ordered materials [6]. Since it is essentially a transmission method it can be successfully applied even at relatively weak neutron sources, although the production of polarized neutron beams inevitably implies some loss of intensity. Without going deeply into details Fig. 8 shows the main components of the three-dimensional neutron depolarization setup which was installed at a tangential beam tube of the Vienna university reactor a couple of years ago and which since then was

used routinely for a variety of investigations of different classes of magnetic materials [7]. By this facility it is possible to reveal the influence of temperature, applied magnetic field and/or tensile stress on the mean size and orientation of the ensemble of magnetic domains within the sample. Relaxation effects of the domain structure after sudden variation of the external field or the applied stress can be investigated on a timescale ranging from about 100 μs up to several hours. Its function can be briefly described as follows:

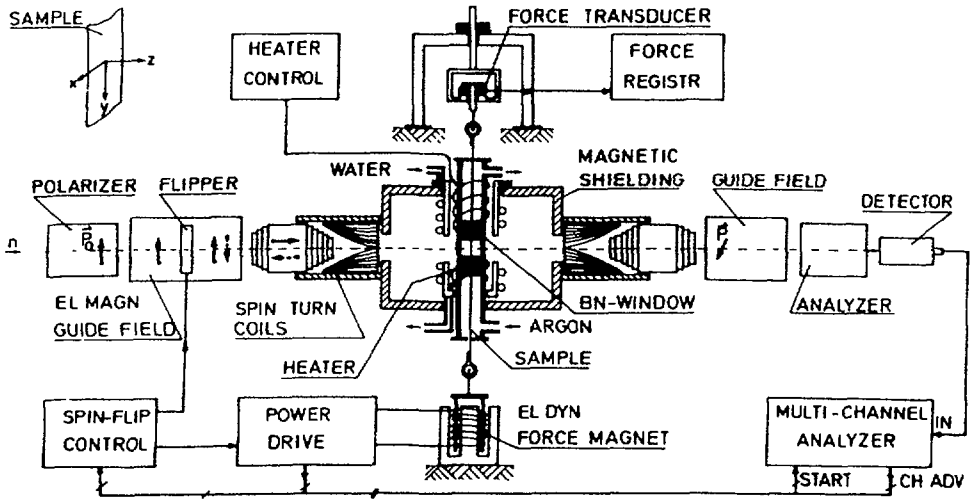

Fig. 8: Schematic sketch of the 3-dimensional neutron depolarization setup.

The incident beam is monochromatized (λ = 1.5Å) and polarized by Bragg reflection at a magnetically saturated Heusler crystal ($P_o = |\mathbf{P}_o| \approx 0.95$). On transmission through the sample, which is mounted within a magnetically shielding soft iron box and whose temperature can be varied between 4.2 K and about 800 K by means of a either a cryostat or a quartz furnace, the polarization $\mathbf{P}_o$ of the incident beam changes to the final state $\mathbf{P}_e$ according to the relation

$$\mathbf{P}_e = D\,\mathbf{P}_o \quad ,$$

where the so-called "depolarization matrix" D depends on the details of the magnetic domain structure within the sample. Two spin rotation systems, which consist of specially designed coil combinations described in detail in the literature [8], are attached to the shielding box. They serve to orient the incident polarization vector successively in any of the three spatial directions and to project any of the three components of the final polarization onto the direction of the analyzing crystal in front of the detector. In combination with a spinflip device which allows for a sudden reversal of the incident polarization state one is then able to measure all 9 elements of the (3 × 3) depolarization matrix D. By means of mathematical inversion algorithms it is then possible to derive the essential parameters of the domain structure, namely the mean size of the domains, the mean square direction cosines of the domain magnetizations, and also to obtain at least qualitative information about eventual correlations between neighbouring domains. Fig. 9 summarizes the action of a set of homogeneously magnetized domains on the transmitted neutron polarization vector.

From the many experiments we have performed with this neutron depolarization setup Fig. 10 shows the result of an analysis of the influence of tensile stress on the domain structure of amorphous ribbons as measured with neutron depolarization and the electro-optical Kerr-effect. Again neutrons offer the advantage to "see" inside of the samples, not just their surface. One should notice the extreme sensitivity of the depolarization method, as follows from a comparison of the depolarization matrix calculated from a theoretical model (Fig. 10, middle) with the experimentally measured one. Even minute variations of the model parameters lead to a significant deviation of the theoretical from the experimental data at least for one of the nine matrix elements.

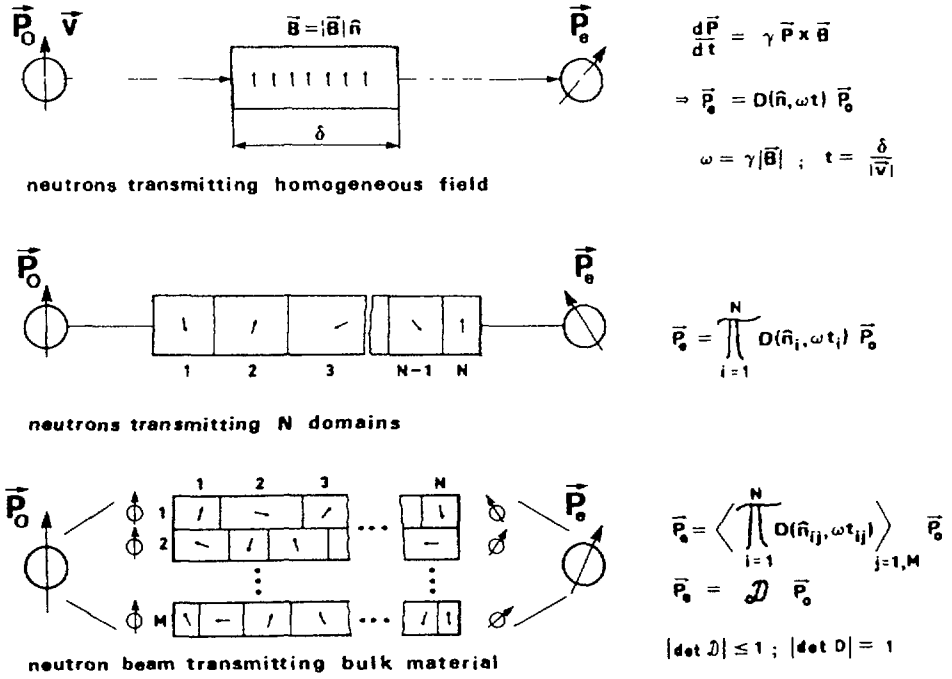

Fig. 9: The principle of neutron depolarization on transmission through ferromagnetic domains.

Another application of the neutron depolarization method which is of interest for the industrial development and production of modern permanent magnets, is the investigation of the magnetic anisotropy of hardmagnetic Nd-Fe-B specimens [9]. It turns out that valuable information can be extracted from the depolarization results which is hard or impossible to gain by conventional magnetization techniques. Very recently we have started a joint research project with a group of Sao Paulo university to develop a new setup allowing for investigation of magnetic relaxation effects simultaneously on one and the same sample by neutron depolarization and magnetization measurements.

For completeness we mention that the a similar setup has been used to demonstrate for the first time that geometric phase shifts of quantum mechanical systems, known as the so-called "Berry phase" [10], can be observed even for incomplete evolutions of the environment of that particular system, in our case a spin-½ particle. To realize this experiment instead of the sample a rotating magnetic field was established along the flight

path between the two spin turn devices of the depolarization facility [11]. But such a highly specialized experiment, belonging to fundamental quantum mechanical research, is surely not adequate as a "beginners" experiment on reactors in developing countries.

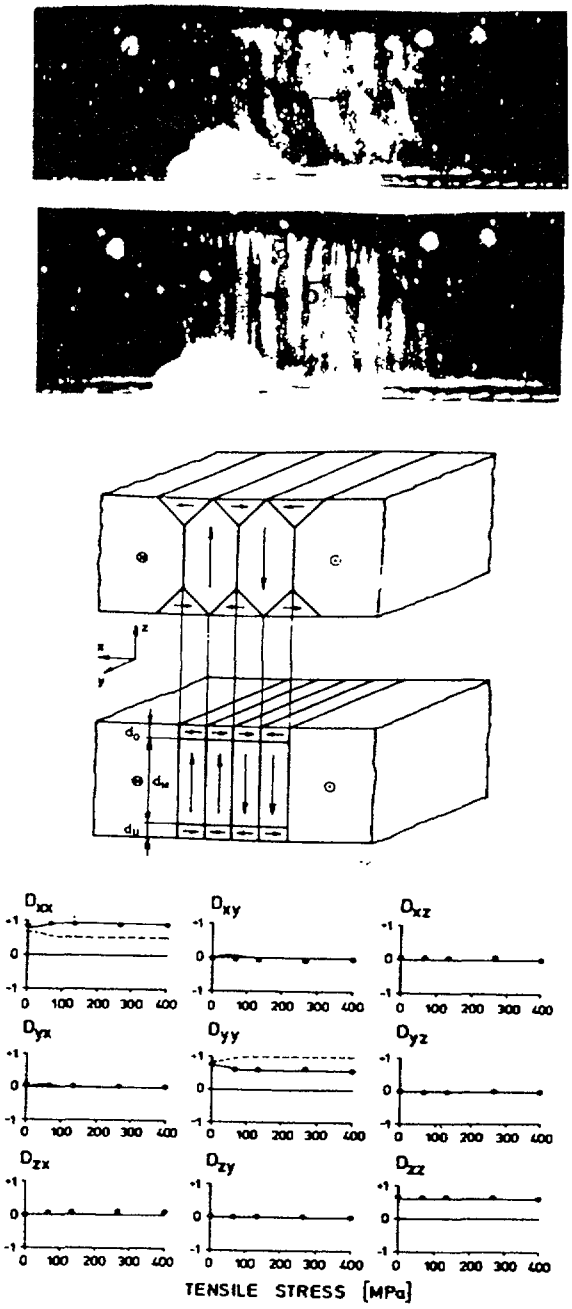

Fig. 10: Kerr-effect and neutron depolarization studies of the domain structure of amorphous ribbons (see text).

7. Neutron Diffraction

Neutron diffraction is a tremendously broad field of research performed at almost all scientific neutron sources around the world. Nevertheless, to do competitive work at a relatively low flux reactor one has to treat problems where it is not so essential to have a higher flux. One obvious situation of this kind refers to the case of structural relaxation effects in any kind of solid matter with time constants larger than about several hours, so that in a real time experiment a statistically sufficient number of neutrons diffracted by the sample hits the detector. Clearly there would be no essential gain of information if the neutron intensity increases further. Another possibility is to chose ambiental conditions at the sample position which can be established periodically but always for short times only. If, as it is the case with our TRIGA reactor, the neutron source can be pulsed synchronously with this periodic variation of the sample environment, so that the whole neutron flux is concentrated at short periodic time intervals and thus correspondingly increased during them, one can study the atomic or magnetic structure of the sample under the chosen external conditions very efficiently.

According to this considerations we have developed a pulsed field-neutron diffractometer, which allows to study magnetic structures with atomic resolution in magnetic fields of up to 20 Tesla [12]. Fig. 11 shows a schematic sketch of this diffractometer which is installed at one of the two radial beam tubes of our reactor. A graphite monochromator selects a wavelength of about 1.1 Å out of the thermal reactor spectrum. The sample can be cooled to liquid helium temperatures by a stainless steel cryostat of only 17 mm outer diameter. This is sufficiently small to fit into the central bore (ϕ = 18 mm) of a split-coil magnet which is connected to a 8 mF/2500 V

capacitor bank. Discharging of this capacitor bank via a high power thyristor circuit produces a field of up to 20 T for typical time intervals between 5 ms and 15 ms, depending on the chosen wiring of the discharging circuit. This fits not too bad to the minimum pulse width of approximately 40 ms of the TRIGA reactor in its pulsed mode of operation, which allows to increase the reactor power and hence the available neutron flux by a factor of about 1200 at a maximum repetition rate of about 5 pulses per hour. Fig. 12 shows that at least with single crystalline samples it is possible to measure Bragg peaks even with a single non-repititive "shot" of the reactor. The idea behind this kind of experiment is to detect the field-induced breaking up of the spin structure of antiferromagnetic materials via the associated change of the structure factors. However, the pulsed mode of operation of the reactor causes a thermal and mechanical stress of the fuel elements and it is hence risky in particular with old fuel rods. Therefore one has to be very careful in the preparation of such an experiment to keep the number of necessary pulses as small as possible. Because of this fact and since the experimental requirements are rather sophisticated this kind of research is not generally applicable even if the respective reactors are principially able to operate in a pulsed mode. As indicated in Fig. 11 the pulsed magnet equipment can be used also for depolarization studies. There the high field is essential to saturate extremely hardmagnetic materials like Nd-Fe-B. Their subsequent magnetic relaxation and domain formation can then conveniently be studied by observing the behaviour of the transmitted neutron spins.

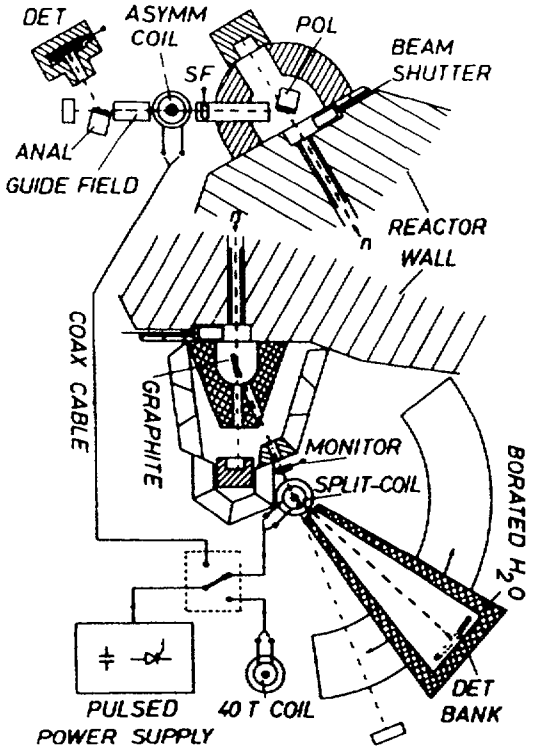

Fig. 11: The pulsed field neutron diffractometer.

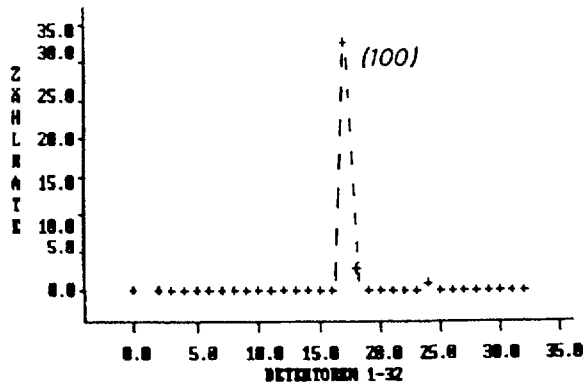

Fig. 12: Antiferromagnetic (100)-reflection of a MnF_2 single crystal measured in 1 ms.

8. Neutron Interferometry

In 1974 the first interferometer for matter waves with macroscopic spatial separation of the interfering beams has been successfully developed and installed at our reactor [13]. Since that time it was used fèr a series of quite spectacular fundamental quantum mechanical

experiments [14]. Although the overwhelming majority of this experiments had to be performed on the high flux reactor of the Institute Laue-Langevin Grenoble simply because of intensity reasons, it was essential to conceive and to prepare them at our reactor as well as to be able to test the functionality of the various components of the final setup. And not to forget, we and our involved students could accumulate sufficient know-how to finally realize the experiments at the high flux reactor.

Again, facilities like a neutron interferometer definitely represent one of the most advanced technologies in the field of neutron scattering and from that reasoning an unexperienced staff at a low flux neutron source should not at all start with such a difficult topic. Because of its complexity we will also not discuss it further here. Even only an introductory review would go far beyond the scope of the present report. But on the other hand it is an outstanding example of the importance of small reactors for the development and realization of new experiments and techniques, by giving young scientists the opportunity to improve their experience and by giving them a chance to follow unconventional ideas and concepts, which is often not possible at larger reactor facilities with their ultimate need of cost effective exploitation of the available beam time.

9. Concluding Remarks

We have shown that even at very small research reactors with low neutron fluxes a variety of valuable possibilities exist both for applied and fundamental research. Besides the topics we have presented her one should not forget two other important activities which are absolutely necessary to operate any nuclear reactor, namely radiation protection and reactor technology. For both of them qualified staff must be available, which in general can hold its standard only if it is not only involved in running the reactor but is also engaged in state-of-the-art research. Furthermore we have also not treated here the possibility to develop and to test new techniques and facilities at a small research reactor which are intended for later use at high flux neutron sources. Always during the last three decades this was a major field of effort at our institute, the most outstanding example being, of course, the successful development of the first neutron interferometer. And, once again, we finally want to stress the importance of small reactors for the education of young scientists and for the preparation of experiments at large high flux facilities.

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USE OF RESEARCH REACTORS FOR SOLID STATE PHYSICS STUDIES AT BHABHA ATOMIC RESEARCH CENTRE, INDIA

K.R. RAO
Solid State Physics Division,
Bhabha Atomic Research Centre,
Trombay, Bombay, India

Abstract

Three research reactors, - Apsara, Cirus and Dhruva have been utilised for over three decades in India for carrying out a variety of Solid State Studies. In this paper, we review several aspects of these studies especially related to the efforts during the past ten years. Plans were drawn around 1975 to design the neutron beam facilities of the then proposed DHRUVA ($\sim 2 \times 10^{14}$ ns/cm^2-sec) research reactor to cater to solid state physics studies. Beginning 1982, comprehensive actions were taken to design the neutron spectrometers, detectors, guides and hot and cold moderators and fabricate them locally. The reactor has been operating at high power levels since 1988 and the indegeneously developed neutron instruments have provided excellent facilities for solid state studies. Details of these plans, developments and results will be presented.

1. Introduction

Apsara, Cirus and Dhruva are three research reactors which have catered to research and development programs at Trombay, particularly for solid state studies during the past four decades. Their characteristics are summarised in Table I .

Apsara provided very useful means of training scientists in the initial phases of neutron beam research programmes. Currently, it is being used for isotope production, neutron radiography and neutron detector testing. There are proposals to convert this reactor into multipurpose higher flux research

Table I : Research Reactors for Solid State Research at Trombay

Reactor	Type	Max. Power	Max.Flux in core	Criticality date
Apsara	Light water enriched U-metal fuel	1 MW	~10^{12}n/cm^2-sec	Aug.4,1956
Cirus	D_2O moderated H_2O cooled nat. U-metal fuel	40 MW	~6×10^{13}n/cm^2-sec	July 10,1960
Dhruva	D_2O moderated D_2O cooled nat. U-metal fuel	100MW	~2×10^{14}n/cm^2-sec	Aug.8,1985

reactor that can operate upto 10MW and provide fluxes of nearly 10^{14}ns/cm^2-sec. Feasibility studies are under way.

Cirus reactor was the main research facility for nearly two decades (1962-1985) for Solid State Physics. A large number and varity of neutron spectrometers were built and operated at this reactor to investigate a variety of Condensed Matter problems like structure and hydrogen-bonds in hydrated crystals and amino acids, magnetic structure of several mixed ferrites, heusler alloys, dilute magnetic systems to map out magnetic electron density distributions, structure of simple liquids like Zn, Ga, CCl_4 etc., dynamics of atomic motions in hexagonal crystals Mg, Zn & Be, and molecular motions in NH_4-halides, liquids CH_4, Cl_4, NH_3 and ND_3 etc. A variety of new concepts in neutron beam instrumentation like window filter, multi-axis (analysis) spectrometer, temperature gradient based high resolution window analyser, data acquisition and spectrometer control systems like fully automated computer controlled diffractometer, microprocessor based control systems were designed, built and commissioned for improved utilisation of the reactor. Table II lists the spectrometers that were operating at this reactor around 1980 and the results of main investigations.

Table II – Spectrometers in Use at Cirus Reactor– Circa 1980

Spectrometer	Use related to	Main investigations
Powder Diffracto-meter	-Powder diffraction -unpolarised neutron diffraction	-Characterisation -magnetic structure of ferrites included canted spin structure
Single Crystal Diffractometer	Crystal structure determination	-Structure of hydrated crystals to understand hydrogen bond interaction, -structure of six amino acids, -structure variation with temperature eg. of $LiKSO_4$
Polarised Neutron Spectrometer	Magnetic structure	Mapping magnetic moment distribution in ferrites & dilute magnetic alloys like Ni–Ru systems
Triple Axis Spectrometer	Lattice dynamics & molecular motions	-Phonon measurements in Mg,Zn,Be,CaO & KNO_3 -elastic diffraction from ammonium halides
Multi–axis Inelastic Spectrometer	------- do -------	
Filter Detector Spectrometer	For inelastic spectral studies from powders	Motion of water molecules in hydrated crystals & simple biological molecules
Rotating Crystal Spectrometer	Study of reorientational motions in solids & liquids	-Dynamics of liquids CH_4,CD_4,NH_3,ND_3 -Dynamics of NH_4^+ & CH_3^+ in ammonium and methyl salts

A large number of visiting scientists from countries like Indonesia, Phillipines, Thailand, Korea, Egypt, Poland and Iraq worked at this reactor and some of them stayed on for long enough periods for doctoral degrees based on work carried out at Trombay. Indian scientists also took part in the Agency's Regional Cooperation Agreement Programme for 5 years at Manilla, Phillipines to train scientists and initiate neutron scattering programmes in many of the countries in the region. Several spectrometers or neutron scattering related apparatus were fabricated at Trombay and supplied to the neighbouring countries.

Cirus reactor has continued to operate in its 33rd year and has been a very useful reactor for our many programmes. Currently a small angle neutron spectrometer, a neutron interferometer, a position-sensitive detector test facility and a neutron spectrometer for demonstration and training purposes have replaced some of the existing spectrometers but other spectrometers have continued to be used for research.

Dhruva reactor has been serving as an additional more powerful source of neutrons since it went into full power operation in 1988. As already stated, solid state physicists were involved in the planning stage of this reactor unlike the case with Apsara and Cirus. This helped us in overcoming and reducing limitations of low neutron flux which would have been a bane otherwise. We shall detail some of these aspects in the following section.

2. Means for overcoming or reducing limitations of neutron flux at Dhruva reactor

Discussions related to planning Dhruva reactor were taking place in the early seventees, when several medium flux and high flux European facilities had already gone into operation with their very sophisticated neutron beam instrumentation that included neutron guides, hot and cold moderators, guide halls, multi-detectors etc. The flux of these reactors were in the range of 10^{14} - 10^{15} ns/cm^2-sec. Hence, it needed courage and tenacity to embark on design and development of a medium flux neutron beam research facility at the then proposed DHRUVA reactor whose characteristics were largely determined by the use of natural uranium metal fuel in a heavy water moderator, coolant and reflector environment. This limited the maximum flux of the reactor to be < 2×10^{14} ns/cm^2-sec and also it was to be a large

volume reactor like Cirus. Inspite of these limitations the solid state physicists at Trombay ventured to plan their R&D facilities with due consideration paid to installation of proper in-pile beam features as well as out-of-pile spectrometer characteristics. Some of the options examined and implemented at Dhruva Reactor are given in Table III.

Table III In-pile beam options and out-of-pile features

(i) In-pile Beam Features
- Cut-aways in-pile shielding backed by high density shielding
- in-pile cavities for installing monochromators to use pile shielding as part of monochromator shielding
- installation of a through tubes containing a section of D_2O to reduce fast neutron and gamma radiation in the neutron beam
- tangential beam tubes to reduce fast neutron component
- provision to instal guides, cold source and hot source

(ii) Out-of-pile Features
- large monochromator-drums backed with yokes to reduce background in hall to minimum
- guides to transport beams to an adjoining laboratory
- focussing monochromators
- position sensitive detectors
- hot and cold neutron sources

Except for the hot and cold neutron sources, all other aspects have been implemented and the throughput of the six spectrometers in operation are at least 10 times and sometimes about 25 times that of their counterparts at Cirus; going by the ratio of fluxes alone at the centre of the pile, the throughput improvement should have been only about 3 at the maximum power levels. Efforts to build the hot and cold neutron sources as well as a 2D-detector SANS are underway.

Table IV lists the spectrometers currently in operation and the principle and new types of investigations which we have been able to carry out at the Dhruva reactor.

Table IV – Neutron Spectrometers at Dhruva and some typical Solid State Studies (March,1993)

Spectrometers	Solid State Studies
1. Profile Analysis Powder Diffractometer	– Thermodiffractograms of mixed ferrites to monitor magnetic phase changes – In-situ loading of PdD – Study of hydrides
2. Single Crystal Diffractometer	– Used mostly for study of some 50 different samples of Hi-Tc materials – Trial texture studies of heat treated stainless steel components
3. Triple Axis Spectrometer	– Phonon density of states of Tetracyanoethylene, YBCO, TlCaBaCuO, Resorcinol
4. Polarised Neutron Analysis Spectrometer	– Paramagnetic phase studies of $CeF_{1.7}$
5. Hi-Q diffractometer	– Amorphous $Ge_{1-x}Se_x$, H_2O-D_2O mixtures and glasses used for waste retention
6. Filter Detector Spectrometer	– Under installation
7. Small Angle Spectrometer	– At Cirus : – of micelles of CTAB at various concentrations, pH values and temperatures – ferrofluids
8. Medium Resolution Inelastic Spectrometer	– Quasielastic scattering in NH_4 salts (trial runs)
9. Neutron Interferometer	– At Cirus : Under relocation
10.Spin-echo spectrometer	– Under development

It is because of increased throuhputs we are able now to measure data like the phonon density of states, structure factors of liquids and amorphous solids, polarised neutron analysis and high resolution quasi-ealstic scattering.

3. Means of improving performance of spectrometers etc.

Performance of spectrometers can be improved by a variety of means as has been documented in various publications. One can resort to multi detectors, multi analysers, focussing guides/collimators, focussing monochromators bent vertically and horizontally as are already well known. But these demand high technology and large finances. The other avenue open is by

increased use of the rather ubiquitious commercially available rather cheap data acquisition, spectrometer control cum analysis systems based on personal computers. The spectrometers operating at Dhruva are all connected to dedicated indegeniously developed on-line microcomputers which can control the instruments as well as as carry out data acquisition and analysis. Conventional as well multi-channel analyser data acquisitions are available. Analysis such as peak-hunt, Rietveld analysis and display can be routinely carried out using these systems. In some special situations like phonon measurements, one may like to assess effect of resolution, which varies with spectrometer's various angular parameters, even before measurements are carried out. We have recently developed such a package in order to carry out time-effective measurements on our triple axis spectrometer. Comparative data displays are a must to ensure accurate normalised data acqusition in many day-to-day situations especially if one were studying samples of slightly different compositions. Stable electronic systems are essential ingredients of PSD based systems. Development of such systems based on anologue or digital approaches using rise-time descrimination, charge division or delay time methods are adopted at Dhruva. Recently our electronics engineers have designed and developed special ADCs which can act as ratio circuits also in a multiplexed manner to overcome difficult matching situations.

4. Optimum exploitation of indigenous resources

Neutron beam research program at Trombay has served very many useful purposes and has influenced other activities in physics research in general in our country. It was realised very early in our development that a meaningful R&D program in solid state studies via neutron scattering can be sustained if

complimentary developments and studies were also initiated and expertise developed.

X-ray diffraction, Mossbauer spectroscopy and Laser Raman Spectroscopy were initiated within the research group; they have blossomed into their own complete research activities over the years. The protein crystallography activity which got started with meagre x-ray diffraction facilities has turned out to be one of the most advanced schools in India. Thanks to the extensive computer and graphics support available, the group has solved recently a protein-structure that of human carboxyanylase-based on several activities involving isolation of protein from human blood samples, protein crystallisation, synchrotron x-ray diffraction data acquisition and analysis based on computer modelling. The one and only low temperature high magnetic field Mossbauer spectrometer in the country designed and developed at BARC is being routinely used for understanding magnetism of Hi-Tc materials. The Raman spectroscopy group has recently commissioned a triple-monochromator based optical multi-channel system for investigation of binary and ternary semiconductors, hi-pressure Raman spectroscopy etc.

Detector fabrication, instrument design and development, low temperature accessories were all undertaken as a part of intentional indegeneous instrumentation activity. The aim was to maximise indegeneous resource involvement and to minimise, if not eliminate, external inputs. This approach has paid very rich dividends in terms of developing expertise in design, manufacture, machining, testing and commissioning not only all the neutron spectrometers and related equipments but also other equally important equipments like detectors, monochromators, guides, cryostats, magnets, power supplies, RF units, control

systems, data handling systems etc. The fairly large variety of infrastructure available in BARC (in various disciplines) and free flow of cross-disciplinary collaboration amongst various disciplines has played a crucial role in developing proto-types and even finished goods internally if necessary. Over the years, some of the finished raw materials and some of the equipments have been got developed by commercial industries and other public sector and private sector companies in the country also. The expertise developed helps in undertaking rather complicated activities which result in technological fall outs in addition to generate the capacity to operate and maintain our own systems. We are also able to offer indegenous products like boron carbide, boron plastics, berylium blocks, control and data acquisition systems and neutron spectrometers on commercial basis to other countries; we have done so bilaterally to Korea and UK and through the IAEA to Bangladesh recently.

As illustrations during the presentation of this paper I intend to show via slides our activities related to manufacture of spectrometers, development of guides and cold source.

Therefore, indegenisation has not been merely a slogan as far as we are concerned, it has become a reality which has enhanced our confidence to take on more difficult tasks.

Currently, installation of a spin-echo spectrometer and a 2D small angle neutron spectrometer have been occupying our interest but there is great enthusiasm amongst my colleagues to go for reflectometry, neutron topography and other applications related instrumentation. The only thing that is limiting our speed is lack of enough trained manpower. Hopefully, we will overcome this in the near future.

5. Requirements and Feasible Approach/es for Staff Training

Unlike in some of the advanced western laboratories where the large facilities are manned by large technical manpower running to hundreds sometimes and users also numbering a few hundred as they come from various universities and other institutions, our program is essentially based on in-house staff consisting of about 20 scientists and 10 technical assistants. This total staff undertakes design and development of equipment as well as operation and maintenance of the same as well as carry out research activities. The situation is somewhat like what used to exist in English or European Universities with younger students and senior professors.

Scientific staff are all M.Sc degree holders in physics and the technical staff, science degree holders qualified in various trades like electronics or vacuum technology or laboratory practices. The scientific staff have also undergone a one-year advanced course in physics in several topics not commonly covered in Universities and also exposed to several experimental projects in our Training School. Such young and fresh staff are attached to experimental groups carrying out solid state physics studies at the reactor and are expected to carry out research from then on. It is observed that, over a period of 3-4 years, most of them pick up sufficient experience to be able to carry out experiments reliably. They shape up as independent research workers over a period of 10-12 years. Opportunities are also created to enable them to proceed on post-doctoral or equivalent assignments to overseas laboratories in their assigned fields of specialisation to enlarge their expertise.

I have already referred to the small staff involved in the neutron beam research. Increasingly it is felt that there is a

need to augment this by suitably trained manpower particularly at the research level. An intentional effort was made about 4 years ago when Dhruva reactor was declared as a National Facility for Neutron Beam Research. It meant that the facility would be available for researchers from the Universities and other institutions and the BARC scientists would also take on the responsibility of Contact and Guiding Scientists for approved projects. The University Grants Commission and the Department of Atomic Energy created a consortium called Inter University Consortium for Department of Atomic Energy Facilities (IUC-DAEF) and one of the Centres of the Consortium to use Dhruva was founded. From then on, efforts have been made to train university students and staff in techniques of neutron beam research via workshops and hands-on practicals as well as encourage them to write proposals for experiments to be carried out at BARC. Four workshops have been conducted so far on : (i) general techniques (ii) powder diffraction (iii) liquids and amorphous systems and (iv) small angle scattering. The next workshop will be related to applications in texture, residual stress measurements and small angle scattering in metallurgy etc. The Consortium supports travel and local expenses for researchers, provides token support for chemicals and small equipments and expects that activities like sample preparation and characterisation by non-neutronic means are carried out at the Universities. It also encourages Universities to design and develop ancillary facilities that can be installed at Dhruva. It is expected that a few selected staff members of IUC-DAEF will also be stationed at Dhruva to coordinate, help and train the University researchers as well as carry out collaborative research programmes. They would be a very useful component as they can take over as Contact Scientists also releaving the local staff for other activities.

6. Regional and International Cooperation

We have already referred to the RCA activities in which India was involved as a part of the Regional Cooperation amongst countries in Asia. This activity in the field of Neutron Scattering has somewhat diminished compared to that in the sixties. However, India continues to support this by holding periodical workshops on topics related to neutron scattering. The next one is scheduled to take place during November-December,1993. Indian technical expertise can be made available for any activities in the region whether in design and fabrication of spectrometers or in providing research experts.

7. Possibilities of indegeneous and cost effective fabrication of spectrometric components, instrumentation and guides

The spectrometers installed at Dhruva are all based on indegeneous design and development. An optimum mix of shield materials and its shilding efficacy of the main monochromator drum and its yoke were determined by shielding calculations. Except for the 1.2 metre diameter bearing that carries the mobile monochromator drum load, all other components were fabricated out of materials commercially available in the country to tolerences specified. Rigid quality control to meet acceptability criteria and international standards were maintained at every stage of manufacture and assembly. In-house workshops as well as industrial machine-shops were involved in the manufacture.

Electronics counting systems were obtained from the Electronic Corporation of India Ltd., and on-line computer control systems from a state-owned commercial concern KELTRON. Detectors were fabricated in the division.

We have developed the guides indegeneously and it may be considered as a major developmental effort. Float glass plates of required size (100x25x1 cm^3) were imported and these were coated with nickel using a 3m dia high vacuum coating plant situated at optical observatory at Kavaloor, India. 0.5mm dia nickel wire was drawn to our specifications by Midhani, tungsten filaments designed at BARC were manufactured by Central Electronics Limited and other mechanical fixtures made at our BARC Workshop. The tungsten filaments had to be replaced after each coating run as they would break because of brittleness as a result of alloy formation. The nickel coated plates were assembled into 1m long rectangular guides using precision jigs fabricated at BARC. The fixtures to align the guides, vacuum enclosure, mechanical support etc., were all obtained from indigenous sources, vacuum tested and installed in position using optical instruments.

8. Commercial and Industrial Applications

We have not carried out any studies of commercial or industrial applications so far. However, we are in the process of developing techniques of texture and residual stress measurements on our diffractometers for testing mechanical components used in our reactor systems. We had carried out texture determination some time earlier to assess quality of uranium metal fuel rods at the stage of developing the process.

SOLID STATE PHYSICS AROUND PAKISTAN RESEARCH REACTOR AT PINSTECH

N.M. BUTT, J. BASHIR, M. NASIR KHAN
Pakistan Institute of Nuclear Science and Technology,
Islamabad, Pakistan

Abstract

The role of PINSTECH has been to conduct research and development as necessitated by the programmes of the Pakistan Atomic Energy Commission. This assignment was entrusted to PINSTECH from its very inception in early 1960's. The setup of PINSTECH and its facilities were planned at this time and the first major research facility in the form of 5MW swimming pool research reactor was established in 1965. This reactor went critical in December 1965 and attained full power in June 1966. The emphasis of course was on the utilization of the research reactor for the studies of nuclear physics, solid state physics, activation analysis and for the production of radioisotopes. The main facility for the study of solid state physics is the triple axis neutron spectrometer. In this presentation, after giving a brief description of the experimental facilities in and around the reactor, the utilization of the reactor for a variety of research projects in the field of solid state physics is described. A brief description of the recent upgradation of the reactor power to 10 MW is also given. At the end, planned future activities suited to the available thermal neutron flux (about $8x10^{13}$ n /cm^2 sec) are reported.

INTRODUCTION

Neutron scattering is by now a well established technique and with the advent of nuclear reactors as a source of neutrons a wide variety of phenomenon have been studied. The usefulness of thermal neutrons arises from its special properties like its lack of charge, and its magnetic moment. As the wavelength of thermal neutrons is comparable with the interatomic distances in a solid, neutrons diffracted from the crystals provide information on their structure. Also the energy of the neutrons is comparable to that of phonons, the inelastic scattering of neutrons provides an accurate method of determining the energy of phonons and hence the frequency of lattice vibrations. Its lack of charge implies that neutrons can penetrate several centimeters deep into the material and hence bulk of a material can be studied.

The Pakistan Research Reactor (PARR-I) was established in 1965 with the aim to promote the technical know-how that is necessary for the introduction of nuclear power in the country. With the establishment of 5MW research reactor, a neutron diffraction facility was indispensable. Hence a triple axis neutron spectrometer was installed at PARR-I. With this medium flux reactor and a simple triple axis spectrometer, a variety of projects such as phase transitions, texture, lattice dynamics, crystal structure, and measurement of temperature factors of materials have been completed. The utilization of this reactor in a developing country with limited instrumental facilities leading to research publications of international recognition is a source of encouragement for other developing countries of the region having similar situations.

This instrument operated successfully until recently when its was decided to upgrade the reactor power from 5MW to a nominal power of 10MW. With the

upgradation of the reactor, it is planned to install new neutron diffraction facilities so that the reactor can be utilized more effectively.

NEUTRON DIFFRACTION STUDIES

For the neutron diffraction studies a triple axis neutron spectrometer (TAS) was acquired from Poland. The spectrometer has been installed at the beam port 3 and moves on rails such that it can be moved up to 25 feet away from the reactor wall.

The TAS has 5cm x 5cm beam aperture. Using the soller collimators the collimation at the monochromator and analyser system can be varied between 10' to 60'. The overall neutron background using a 60 cm long, 5 cm diameter BF_3 detector is about 2 counts/minute. The monochromated neutron flux at the sample position is about 10^5 n/cm^2/sec at a wavelength of 1A· The main features of the spectrometer are given below.

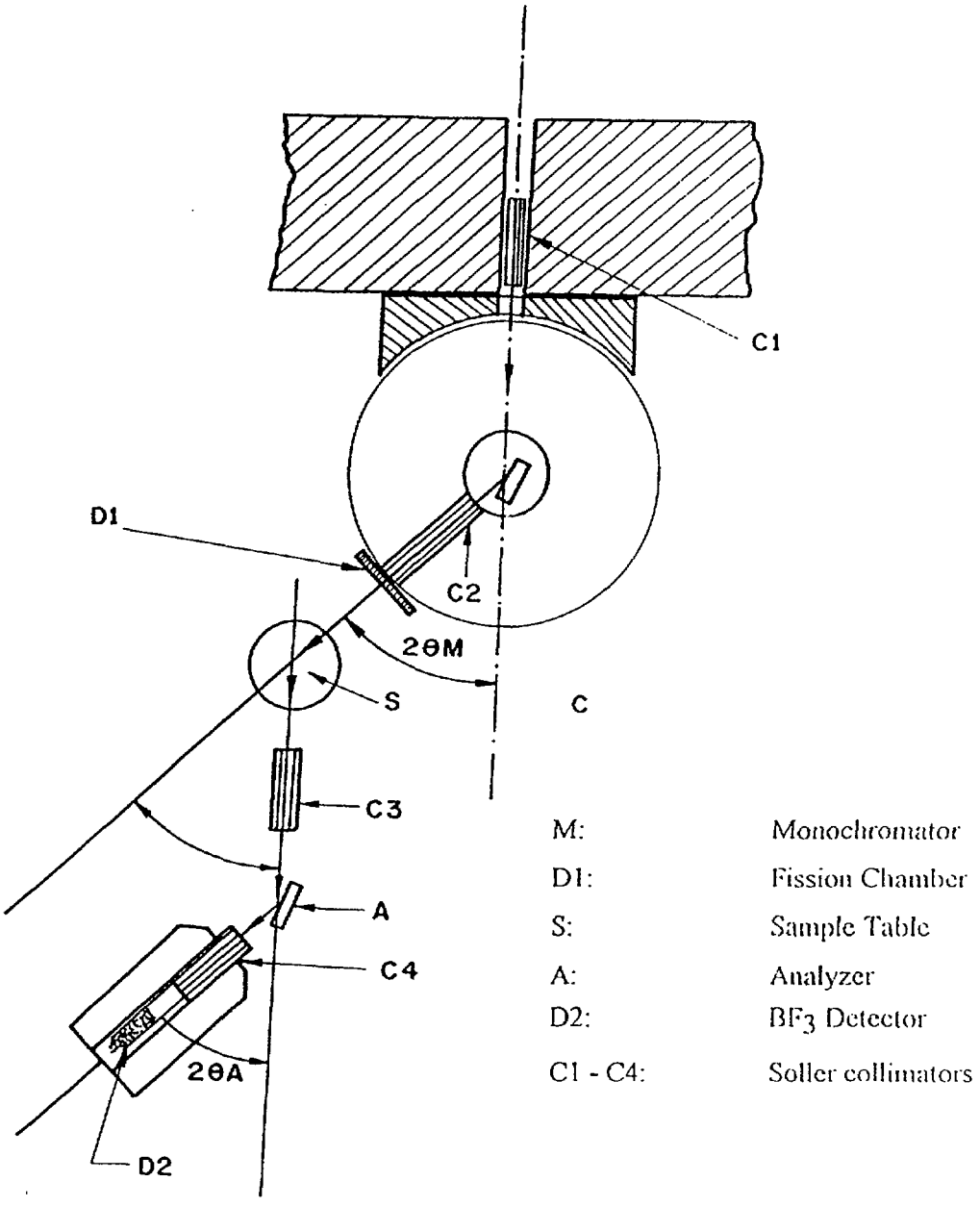


Fig.1 Layout of the Triple-axis Neutron Spectrometer TKSN-400 at PARR-I

MAIN FEATURES OF TRIPLE AXIS NEUTRON SPECTROMETER TKSN-400

The layout of the triple axis spectrometer is shown in Fig. 1. The mechanical parts consist of the first-axis where a single-crystal monochromators (M) is placed. The crystal is surrounded by a very heavy monochromatic shield of about 3 feet radial thickness of borated water. The crystal rotation table and the monochromator arm are coupled through gears in the ratio 1:2. The table has an angular range of rotation of 360° while the monochromator arm (or shield) can rotate over a range of -15° to +90°. The accuracy of the angle setting is 1' and the backlash in gear coupling is about 2'. The second-axis system, the sample table (S) and the sample arm are placed on the monochromator arm. The sample table has no gear-coupling with the sample arm and both can be rotated independently. The angular ranges of the two are 360° and 90° respectively. The third-axis, analyser table (A) and the analyser arm are placed on the sample arm. The angular range of the table is 360° while that of the analyser arm is 160°. All the angles of all the axes can be set automatically by a programmed paper tape. This spectrometer has been used for the following studies:-

(a) ELASTIC NEUTRON DIFFRACTION

(1) DEBYE-WALLER FACTORS OF MATERIALS

The intensities of x - rays or neutrons scattered elastically into Bragg diffraction peaks by crystals is proportional to a Debye - Waller factor ***exp(-2M)*** which accounts for the thermal disorder of the atomic motion.Therefore, the observed intensity, I_o, of a Bragg peak is written as

$$I_o = I_c \, exp(-2M)$$

For a monoatomic cubic crystal ***M*** is related to the thermal parameter ***B*** by

$$2M = 2B(\sin\theta/\lambda)^2 \qquad (1)$$

where λ is the x- ray or neutron wavelength, θ is the Bragg angle. The ***B*** parameter is related to the mean square amplitude of atomic vibration $\langle u^2 \rangle$ and the Debye temperature Θ of the crystal through the relations

$$B = 8\pi^2 \langle u^2 \rangle \qquad (2)$$

$$= (6h^2T/mk\Theta^2)[\Phi(x) + x/4] \qquad (3)$$

where the symbols have their usual meanings (James 1967).

MEASUREMENT PROCEDURE

There are two different methods of determining the B values of the crystals, viz.:

1) From the variation of integrated intensities with Bragg angle (Wilson plot)
2) Rietveld refinement procedure

WILSON PLOT

In this method, intensities of a large number of Bragg reflections are measured at a fixed temperature. The integrated intensity for each reflection is then determined

by summing the counts, point by point, across the Bragg peak and subtracting the background on either side. These integrated intensities (I_{hkl}) are converted to the structure factors (F_{hkl})using the standard relation

$$I_{hkl} = C\, J_{hkl} L_{hkl} F_{hkl}$$

with C is the scale factor, J_{hkl} the multiplicity of the reflection, L_{hkl} its Lorentz factor of the (hkl) reflection. The temperature parameter ***B*** is then determined from the least square fit to the line

$$\ln(F_o/F_c) = \text{Constant} - B\,(\sin\theta/\lambda)^2 \qquad (4)$$

The slope of the line [Fig. 2] gives **B** at the temperature at which the diffraction pattern [Fig. 3] is measured and the mean square amplitude of vibration $\langle u^2 \rangle$ and the Debye temperature Θ are calculated from equations (2) and (3).

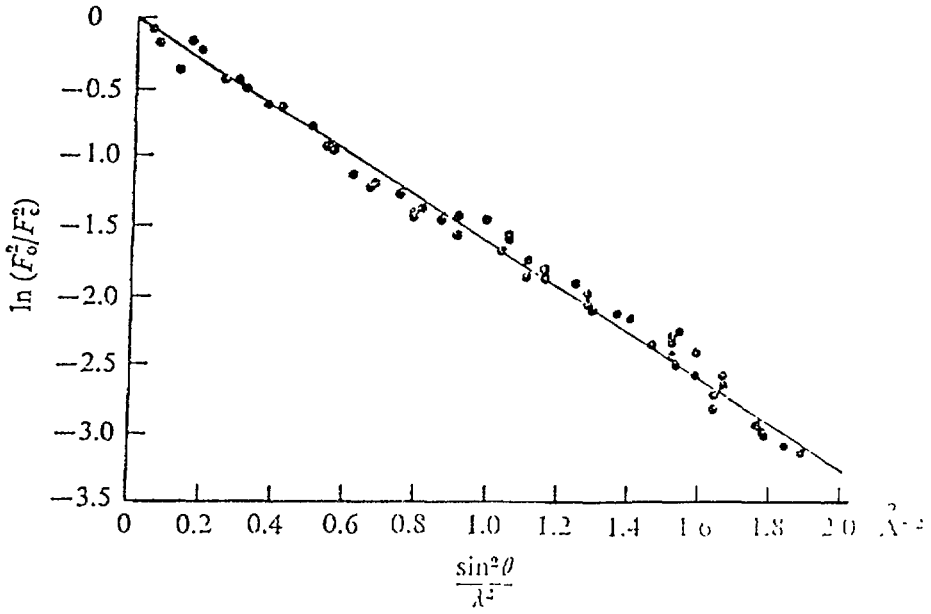


Fig.2 Wilson Plot for Al (X-ray diffraction data).

In case of cubic compounds, there are two atoms and hence two different B-values, B^+ for cation and B^- for anion. In this case, B determined from (4) gives the average B value which is the mass weighted average of B^+ and B^-.

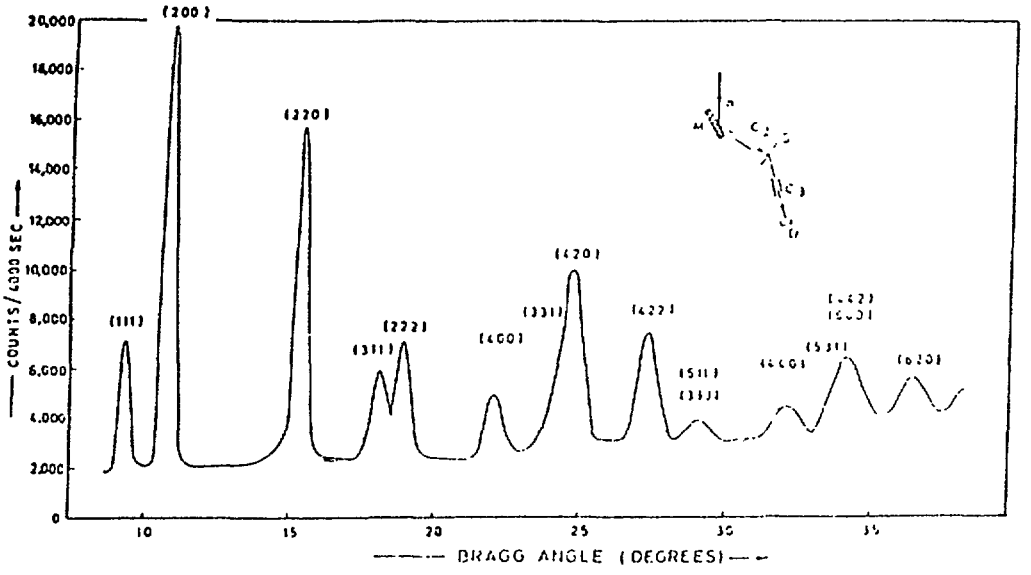
It is also possible to determine the individual temperature factors of cation and anion from the diffraction experiment. For example, in case of ZnTe which has zinc blende structure (space group F43m), the structure factors can be expressed in three forms of the sum of h, k and l related to 4n, 4n±1 or 4n±2, n being an integer. With Zn atoms at (000) and equivalent positions, and Te atoms at (1/4,1/4,1/4) and equivalent positions, the structure factors are given by

$$F^2_{4n} = 16\,[\,b_{Zn}\exp(-M_{Zn}) + b_{Te}\exp(-M_{Te})]^2 \qquad \text{for } h+k+l = 4n \quad (5)$$

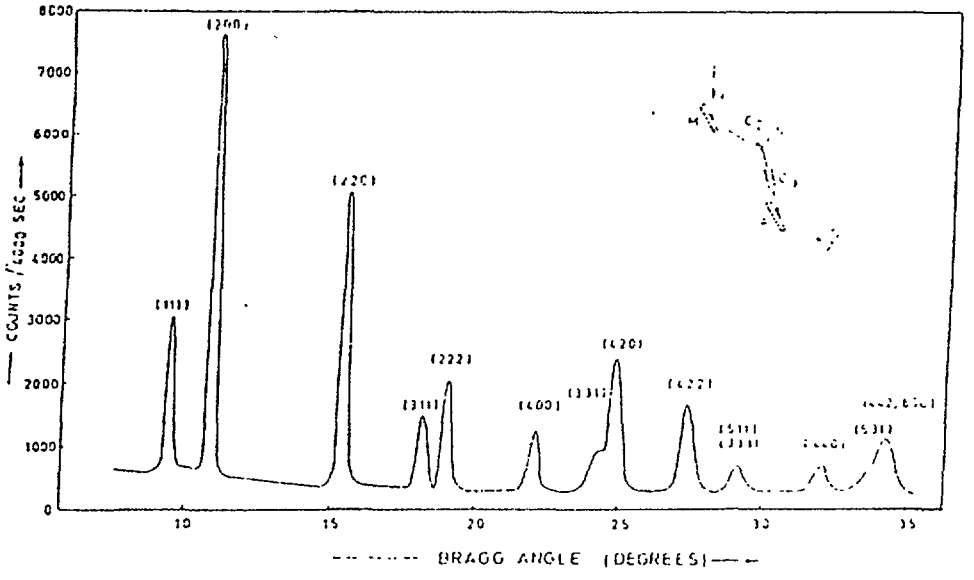
$$F^2_{4n\pm1} = 16\,[\,b_{Zn}\exp(-M_{Zn})]^2 + 16\,[b_{Te}\exp(-M_{Te})]^2 \qquad \text{for } h+k+l = 4n \pm 1 \quad (6)$$

$$F^2_{4n\pm2} = 16\,[\,b_{Zn}\exp(-M_{Zn}) - b_{Te}\exp(-M_{Te})]^2 \qquad \text{for } h+k+l = 4n \pm 2 \quad (7)$$

where b_{Zn} and b_{Te} are the neutron scattering lengths of Zn and Te and n is positive integer. From (5) and (6) we can write (Butt et al 1978)



Double-axis neutron diffraction pattern of KCl powder.



Triple-axis neutron diffraction pattern of KCl powder.

Fig.3 A typical Neutron Diffraction pattern in Double axis and Triple axis mode.

$$\ln(\alpha+\beta) = \text{Constant} - B_{Zn} (\sin\theta/\lambda)^2 \quad (8)$$

$$\ln(\alpha-\beta) = \text{Constant} - B_{Te} (\sin\theta/\lambda)^2 \quad (9)$$

where

$$\alpha = [F^2_{4n}]^{1/2} \quad (10)$$

$$\beta = [2F^2_{4n\pm1} - F^2_{4n}]^{1/2} \quad (11)$$

Thus the plot of $\ln(\alpha+\beta)$ and $\ln(\alpha-\beta)$ vs $(\sin\theta/\lambda)^2$ will be straight lines; the slopes of these two straight lines yield B_{Zn} and B_{Te} respectively.

CORRECTION FACTORS

The correction factors include the contributions of thermal diffuse scattering(TDS) and absorption.

THERMAL DIFFUSE SCATTERING (TDS)

In the diffraction experiment, the scattered intensity consists of both elastic and inelastic components. In fact the elastically scattered component is measured on a background of inelastically scattered radiation consisting of a Compton - scattered component which has a slow variation with the scattering angle, and a phonon - scattered component which is known to be peaked around the Bragg position and the principal part of which is proportional to *M*. The peaked part of the inelastic scattering cannot be completely resolved from the elastic Bragg peak owing to insufficient energy resolution of the experiments. The inclusion of this part of the phonon scattered radiation in the measurement of the Bragg peak intensity thus results in a systematic error in the estimates of the temperature factors.

This systematic error can partly be corrected by the use of triple axis spectrometer whereby the resolution is improved as compared to the two axis spectrometer. In the triple axis spectrometer, a second single crystal is placed after the sample. This single crystal, the so called analyser crystal diffracts only those neutrons to the detector which have the same energy as those of incident neutrons. In this way, inelastic contribution to the Bragg reflection is considerably reduced and therefore more accurate B values can be determined.

The TDS contribution to the diffracted intensity can be estimated theoretically [Nilsson (1957), Warren(1953), Chipman & Paskin (1959), Willis(1969)]. If I_{obs} is the observed integrated intensity, the TDS corrected intensity is given by

$$I_{obs} = I_{Bragg}(1+\sigma) \quad (12)$$

where

$$\sigma = (8\pi/3)\,(Q^2 q_{max} K_b T/3mv_z)\left[1/V_l^2 + 2/V_t^2\right] \quad (13)$$

where the symbols have their usual meanings (Willis 1969). Eq. (13) shows that σ is proportional to Q (for a fixed value of q_{max}). If σ is sufficiently small, then $(1+\sigma)$ can be replaced by e^{σ} then the effect of including TDS in the estimate of the Bragg intensity is to decrease artificially the overall B-value. This decrease ΔB_{TDS}, is given by

$$\Delta B_{TDS} = (64\pi^3/9)\,(q_{max} K_b T/mv_z)\left[1/V_l^2 + 2/V_t^2\right] \quad (14)$$

Willis (1969) also pointed out that the first order TDS correction for the faster than sound neutrons can be estimated from the same formulae as for x-rays, and that, to first approximation, there is no TDS correction for the scattering of slower than sound neutrons.

ABSORPTION CORRECTION

For materials with high linear absorption coefficient, the change in B due to absorption is (Hewat 1979)

$$\Delta B_{absor} = \lambda^2 \left[d_1(\mu r) + d_2(\mu r)^2\right] \qquad (15)$$

where μ is the linear absorption coefficient and r is the radius of the sample. The values of the two constants d_1 and d_2 for the cylindrical sample are -0.0368 and -0.03750 respectively.

RIETVELD REFINEMENT METHOD

In the Rietveld method of refining powder diffraction data, the profile of the entire diffraction pattern is calculated and fitted to the experimental profile. There is no need to extract integrated intensities first and so patterns can be analysed containing many overlapping Bragg peaks. The method was applied originally by Rietveld (1967, 1969) to the refinement of neutron intensities recorded at a fixed wavelength. Subsequently, it has been successfully used for analysing powder data from all four categories of experimental technique, with neutrons or X-rays as the primary radiation and with measurement of the scattered radiation at a fixed wavelength (and variable angle) or at a fixed angle (and variable wavelength).

A structural refinement based on powder data is always likely to be inferior to one using good single-crystal data. In cases where it may not be possible to obtain a single crystal sample, with a powder sample twinning, absorption and extinction may be neglected. The Rietveld method is suitable for refining structures with up to 200 parameters: for a review of successful refinement, see Cheetham and Taylor (1977) and Hewat(1985).

The function M_p which is minimized by least squares is

$$M_p = \Sigma_i W_i \left[\{y_i(obs)-b_i\} - y_i(calc)\right]^2 \qquad (16)$$

Where $y_i(obs)$ is the intensity measured at a point i in the diffraction pattern, w_i is its weight and b_i is the contribution from the background. If the variance of the background is arbitrarily set to zero, and if the only source of error is that from counting statistics, then

$$W_i = [y_i(obs)]^{-1}$$

$y_i(calc)$ is the calculated intensity and is given by

$$y_i(calc) = \Sigma I_k G_{ik} \qquad (17)$$

in which I_k is the integrated intensity of the kth reflection whose peak shape is described by the function G_{ik} which is normalized so as to give a value of unity when summed over the whole peak. The summation in (17) includes just those reflections, making a significant contribution to $y_i(calc)$; the summation in (16) is over the number, N, of points used in the refinement.

In the fixed wavelength neutron technique, the intensity is measured at different scattering angles $2\theta_i$. The integrated intensity I_k is then:

$$I_k = C\, J_k L_k F_k \qquad (18)$$

with C is the scale factor, J_k the multiplicity of the reflection, L_k its Lorentz factor and F_k the structure factor which is calculated from a starting model of the structure; for magnetic structures there is both nuclear and magnetic scattering, and so (for unpolarised neutrons) F_k is given by

$$[F_k]^2 = [F_{nk}]^2 + [F_{mk}]^2 \qquad (19)$$

where F_{nk} and F_{mk} are the nuclear and magnetic structure factors respectively. The peak-shape function is approximated by a Gaussian

$$G_{ik} = [H_k]^{-1} \exp [-4\ln 2(2\theta_i - 2\theta_k)^2/H^2_k] \qquad (20)$$

and H_k is the full width at half maximum and varies with the Bragg angle 2θ in accordance with

$$H_k^2 = U\tan^2\theta + V\tan\theta + W \qquad (21)$$

where U, V, W are constants and depend on the collimators and mosaic spread of the monochromator(Caglioti et al, 1958). The contribution of the reflection k to $y_i(calc)$ effectively drops to zero when $(2\theta_i - 2\theta_k)$ exceeds 1.5 H_k.

From the least-squares refinement, information about the thermal parameters can be obtained.

We have measured, the Debye-Waller factors of various elements and alkali halide materials. Furthermore, concentration dependence of the Debye temperature in mixed alkali halide systems was studied. For these systems, it was observed that concentration dependence of Debye temperature is not linear. The results also indicated that the values of mean square displacement of atoms obtained using the diffraction data were composed of two factors. One due to the dynamical factor based on lattice vibrations and the other due to the static variation in atomic positions. The static variation would occur due to different sizes of the constituent ions. Knowledge of these factors is important and one has to be careful in evaluating various constants like specific heat, elastic anisotropy, shear and Young's moduli for these materials. Table 1 summarizes the results of these investigations.

Reliable values of temperature factors are required in the calculations of TDS, EXAFS, LEED, impurity scattering and band structure. These are further useful for reactor moderator studies and for the test of lattice dynamical models used to understand the binding forces in the solids.

Table 1: List of materials for which thermal parameter B has been determined at PINSTECH by the powder neutron diffraction method.

Material	$B^+(A^2)$	$B^-(A^2)$	$B(A^2)$	Θ(K)	Reference
Mo			0.25(1)	385(7)	Bashir et (1992)
$K_{0.5}Rb_{0.5}Cl$			2.39(12)	172(10)	Bashir et al (1992)
Si			0.45(2)	531(11)	Beisheng et al (1990)
RbCl			2.43(20)	153(7)	Ghazi et al(1989)
Nb			0.55(5)	262(12)	Bashir et al (1987)
KF			1.23(11)	312(14)	Beg et al(1981)
RbF			1.40(25)	216(19)	Beg et al (1981)
$K_{0.5}Rb_{0.5}F$			1.84(20)	214(12)	Beg et al (1981)
TlCl			3.07(22)	97(4)	Mahmood et al(1980)
RbI			3.19(11)	101(2)	Beg et al (1979)
KI			3.06(16)	117(3)	Beg et al (1979)
$K_{0.7}Rb_{0.3}I$			3.10(24)	112(4)	Beg et al (1979)
$K_{0.5}Rb_{0.5}I$			3.52(17)	102(3)	Beg et al (1979)
$K_{0.3}Rb_{0.7}I$			3.14(11)	105(2)	Beg et al (1979)
ZnTe	1.26(5)	0.74(5)	0.91(5)	198(5)	Bashir et al (1988)
UO_2	0.23(77)	0.43(7)	0.25(9)	396(70)	Ahmed et al (1979)
KBr	2.55(7)	2.20(4)	2.33(30)	158(4)	Butt et al (1976)

Although temperature factor data for materials is available in the literature, but in some cases large variation in B-values are observed. A typical example is that of GaP where average temperature factor ranges from 0.38(3) to 0.74(3) A^2. Therefore in order to provide reliable values of temperature factors, the same were compiled and experimental data was evaluated. From the evaluated data, recommended values of temperature factors of 22 cubic elements [Butt et al, 1988] and 52 cubic compounds [Butt et al, 1993] were published. Furthermore using these data, interesting correlations between the thermal parameter B with various thermal properties [such as melting point (Fig. 4 & 5), coefficient of thermal expansion (Fig. 6)], mechanical properties [such as Young's modulus (Fig. 7) and compressibility (Fig. 8)] and cohesive energy (Fig. 9) of cubic elements have been established (Butt et al 1993). Similar relations have been observed in case of cubic compounds (Fig. 10 & 11).

(2) NEUTRON DIFFRACTION STUDIES OF THE UNIT CELL OF CELLULOSE - I, CELLULOSE - II AND DEUTRATED CELLULOSE

The unit cell of cellulose-I has been a subject of study for about 50 years using x-ray and electron diffraction techniques. The most widely favoured structure of cellulose-I has been the structure of Meyer and Misch (1937) who gave a=8.35A, b=10.3A c=7.9A and $\beta = 84^o$ based on their x-ray work. However, it was pointed out by many workers (Jones 1958, 1960) that the structure was not completely satisfactory. They argued that agreement between intensities calculated on the basis of this structure and observed intensities is not good. Furthermore, it is not possible to

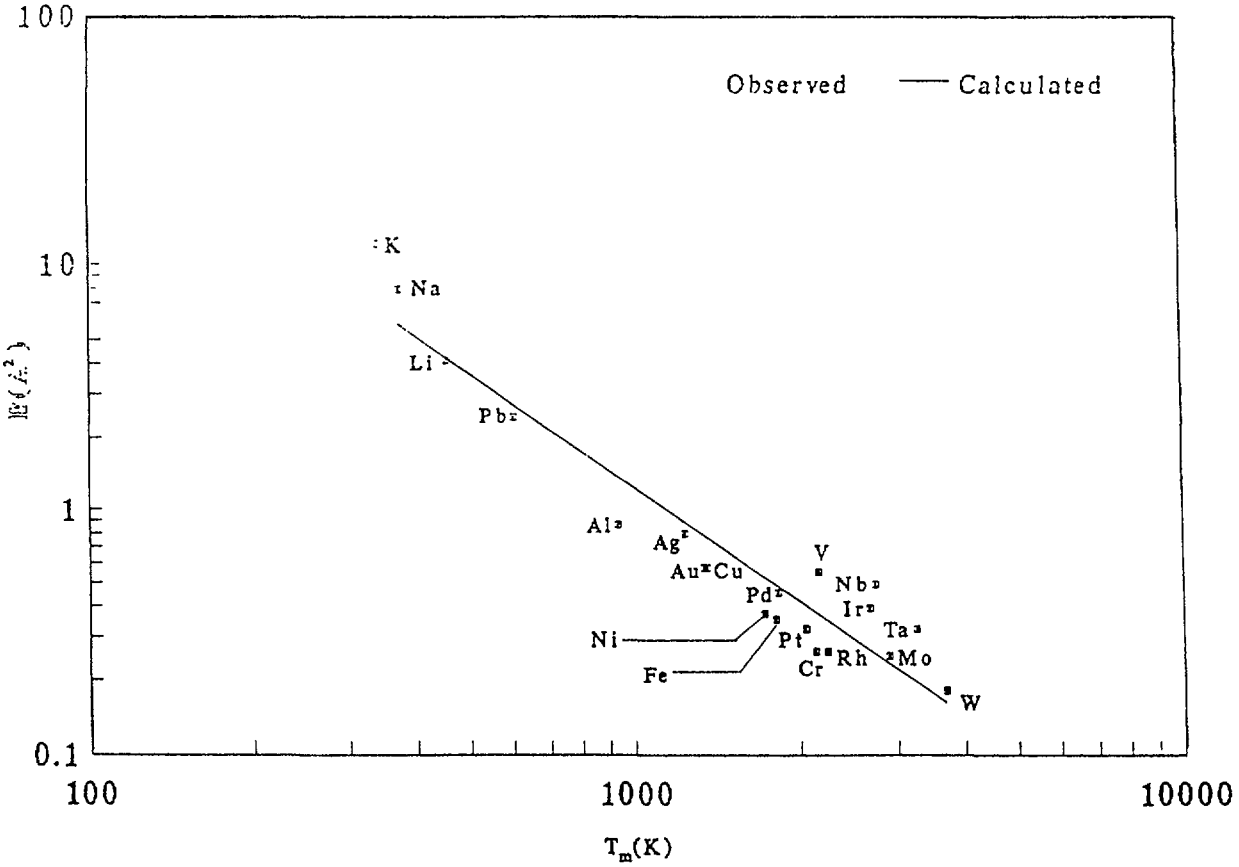


Fig.4 Correlation of Thermal parameter with Melting Point T_m. (Cubic Elements)

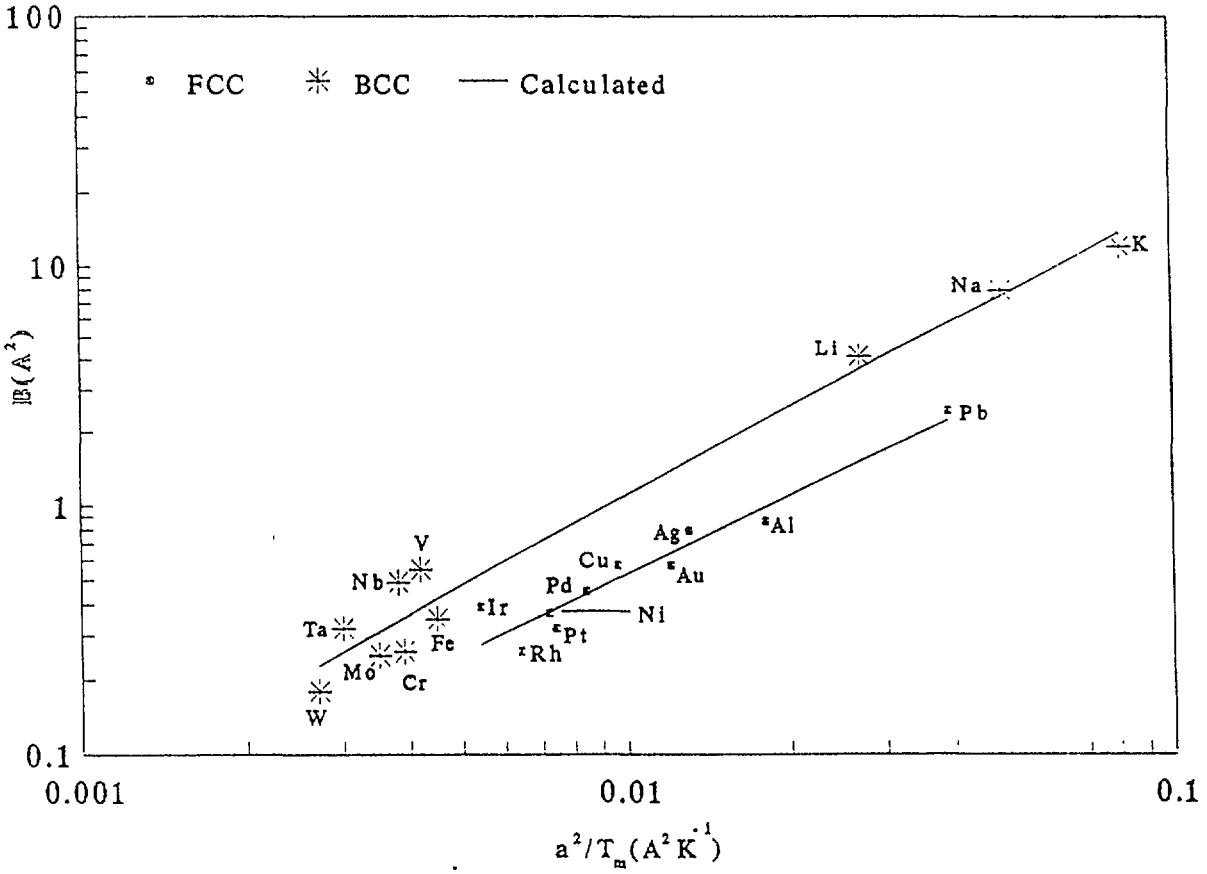


Fig.5 Correlation of Thermal parameter with a^2/T_m. (Cubic Element).

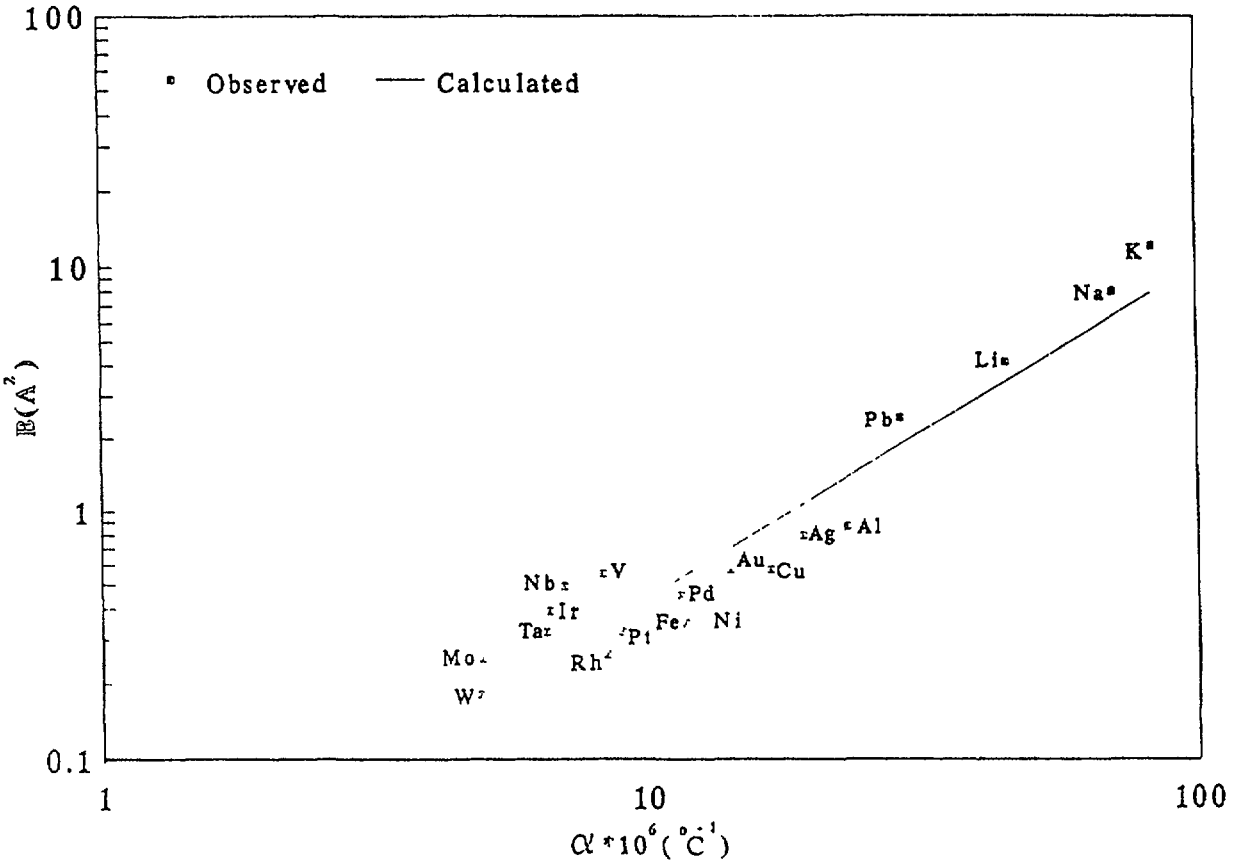


Fig.6 Correlation of Thermal parameter with coefficient of Thermal expansion (Cubic Elements)

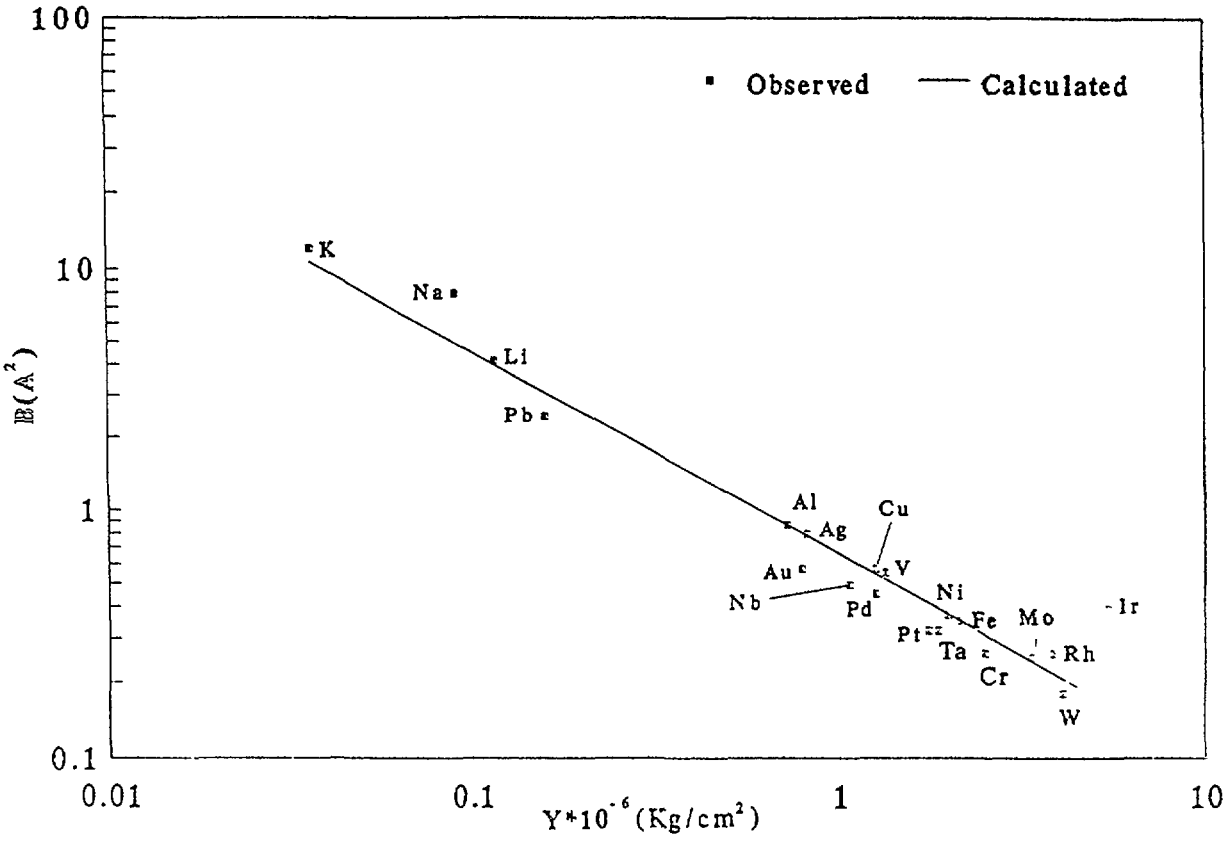


Fig.7 Correlation of Thermal parameter with Young's Modulus. (Cubic Element).

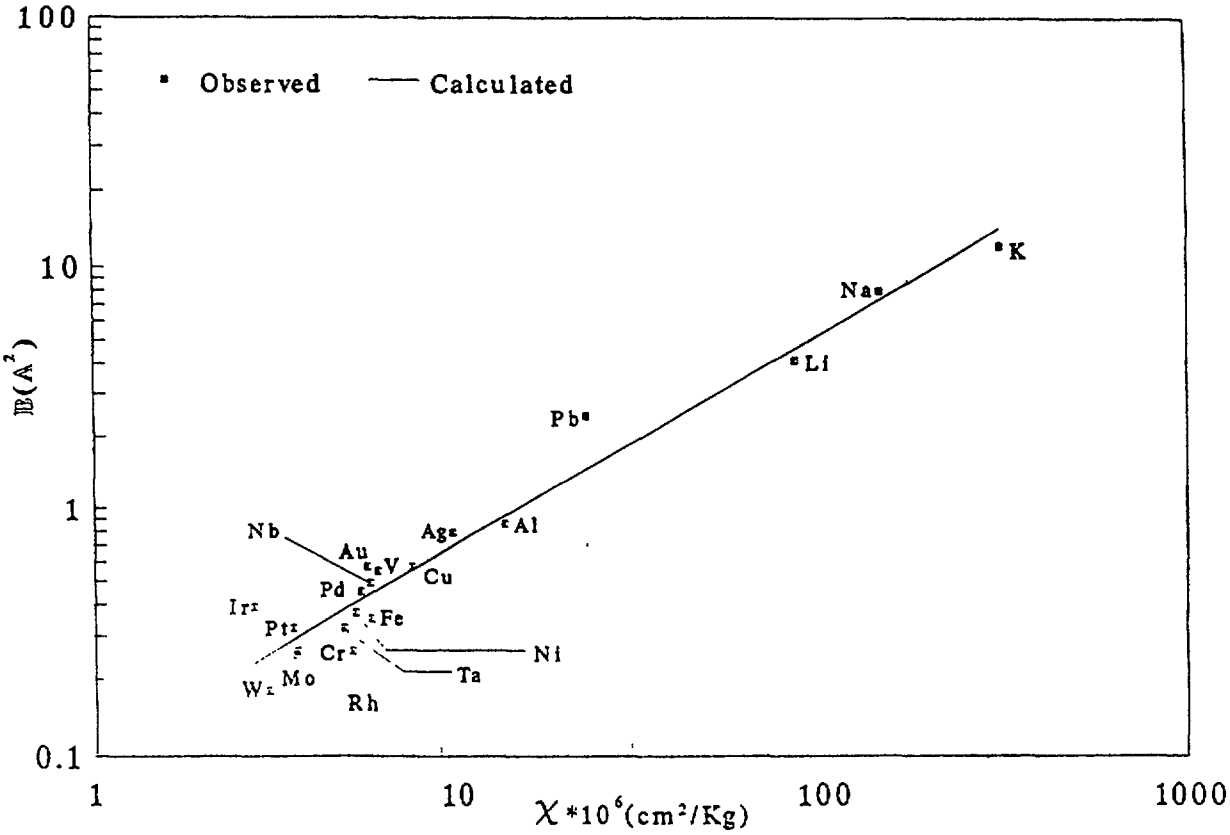


Fig.8 Correlation of Thermal parameter with compressibility. (Cubic Elements).

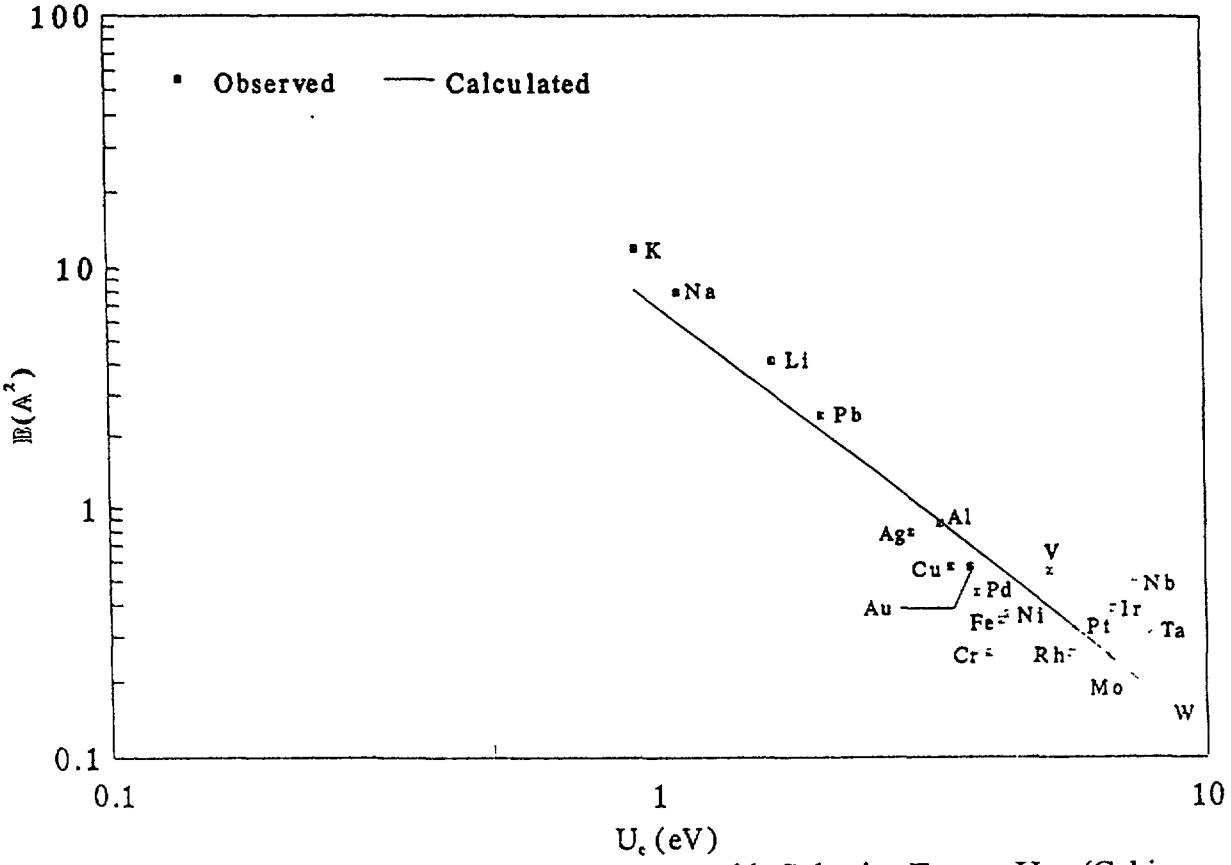


Fig.9 Correlation of Thermal parameter with Cohesive Energy U_C . (Cubic Elements).

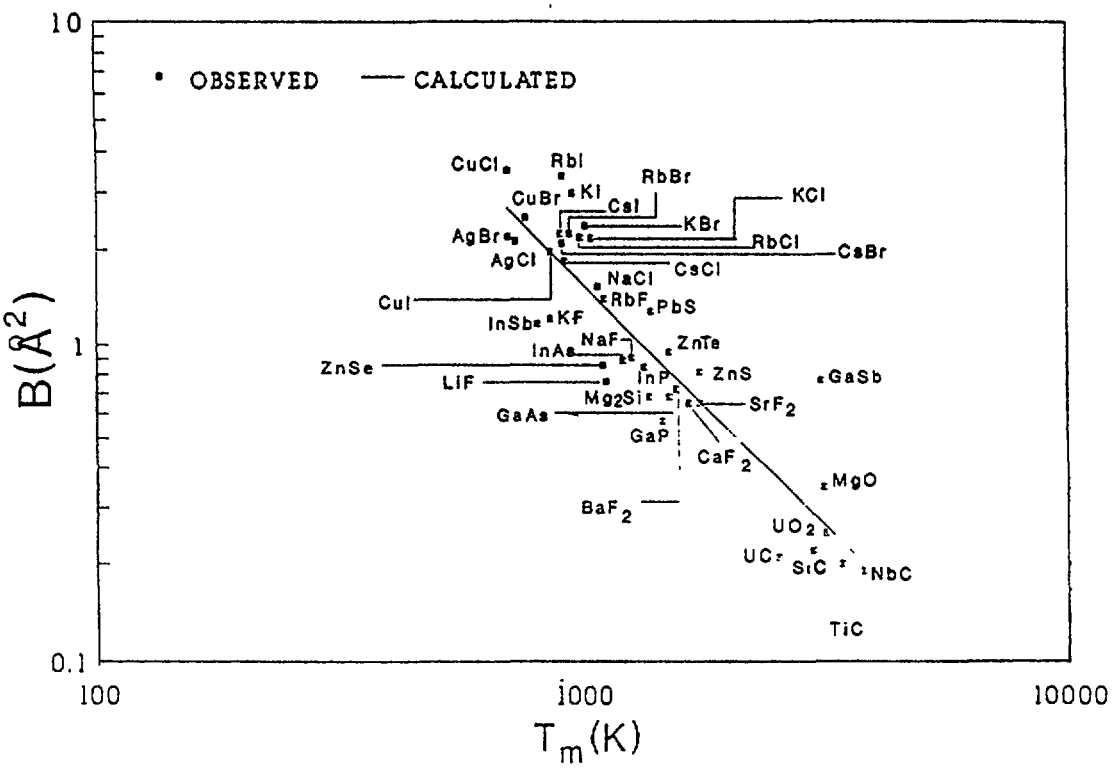


Fig.10 Correlation of Thermal of Thermal parameter with Melting Point (Cubic Compounds).

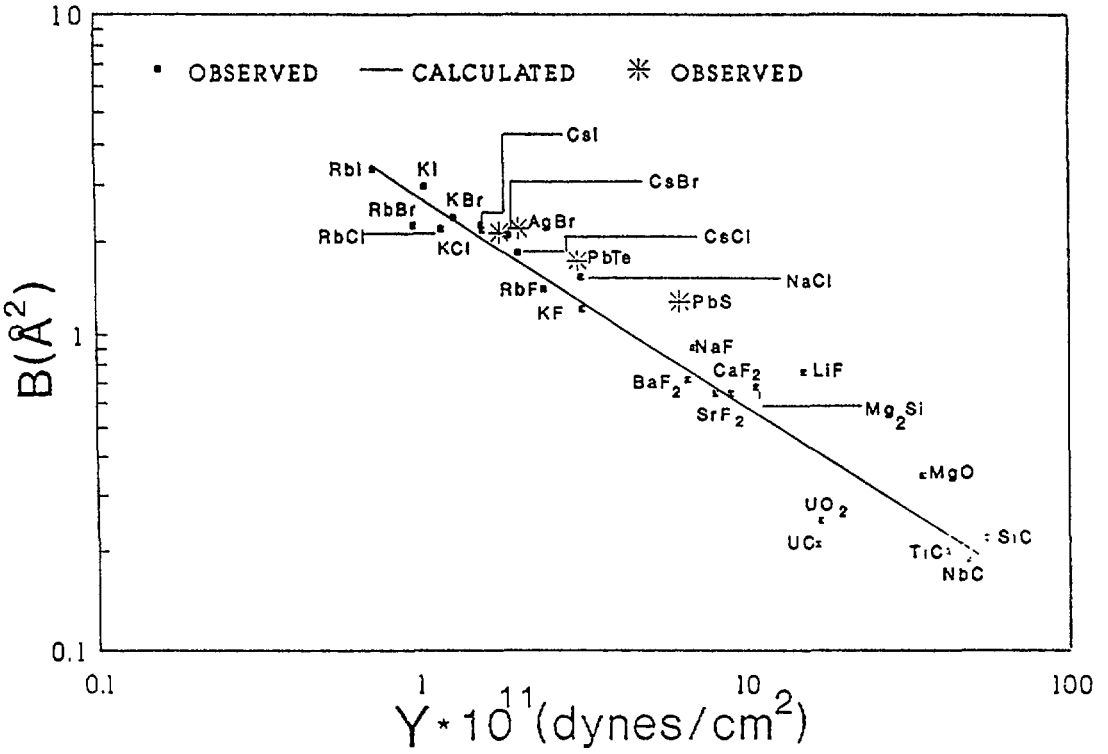


Fig.11 Correlation of Thermal parameters with Youngs Modulus (Cubic Compounds).

index all the reflections on the basis of Meyer and Misch unit cell. Honjo & Watanabe (1958) carried out the examination of cellulose - I by low temperature electron diffraction technique and reported that a number of reflections can be indexed, not by Meyer and Misch unit cell, but by a cell having *a* and *c* dimensions twice as long as those suggested by Meyer & Misch.[Fig.12]

Neutron diffraction studies conducted at PINSTECH gave the unit cell for cellulose - I as $a = 16.78$A, $b = 10.3$A, $c = 15.88$A and $\beta = 82^{\circ}$ [Beg et al 1974]and hence we are able to confirm the results of Honjo & Watanabe. We were also able to observe the diffraction peaks which were not observed in the X - diffraction pattern. The studies were latter extended to cellulose-II [Fig.13]. These experiments were performed with plate as well as cylindrical samples to avoid the preferred orientations. The cell dimensions were again found to be larger than those given by x - ray diffraction technique and were $a = 15.7$A, $b = 10.3$A, $c = 18.4$A and $\beta = 63^{\circ}$ [Ahmed et al 1976].

(3) ORDER-DISORDER PHASE TRANSITION STUDIES IN IRON BASED ALLOYS

In iron rich Fe-Al alloys, two ordered structures with stiochometric composition FeAl and Fe_3Al can be formed from the disordered bcc alpha - phase. The FeAl phase is formed by ordering among the nearest neighbour atoms of the alpha phase to form the CsCl type structure with iron atoms at the cube corners and at the body centers. A further ordering among the second neighbour atoms produces the Fe_3Al structure with a unit cell having twice the lattice parameter of the phase.

The order-disorder transition in FeAl alloys was first reported in 1932 by Bradley & Jay. Since then, much effort has been put into understanding the order-disorder transformation in different systems particularly in the FeAl system. However there is some disagreement with regard to the type of transition from disordered to FeAl, Fe_3Al ordered phases. According to some workers, this transition is a first order transition, while according to others it is of a continuous nature.

The neutron diffraction technique provides a direct way of investigating the order-disorder transition by studying the intensities of the superlattice peaks [(111) and (200) in the present case] in the diffraction pattern. The intensity of peaks depends upon the degree of the order and on the difference between the scattering lengths of the atoms involved.

We studied the order - disorder phase transition in Fe Al alloy for compositions 20.24, 24.15, 28.06, and 31.45 at. % Al.[Ahmed et al 1982] We confirmed that the phase transition from the disordered alpha phase to ordered FeAl and from FeAl to Fe_3Al is of continuous nature. The value of critical index β, the order parameter, was found to be 0.302(9) thereby indicating that the transition is the first order transition. The phase transition in sample with 24.15 at.% Al is shown in Fig. 14.

(4) TEXTURE STUDIES IN SHEETS OF COPPER AND ALUMINIUM

The great majority of technologically applied materials have a polycrystalline structure. In such materials the texture describes statistically the orientation distribution

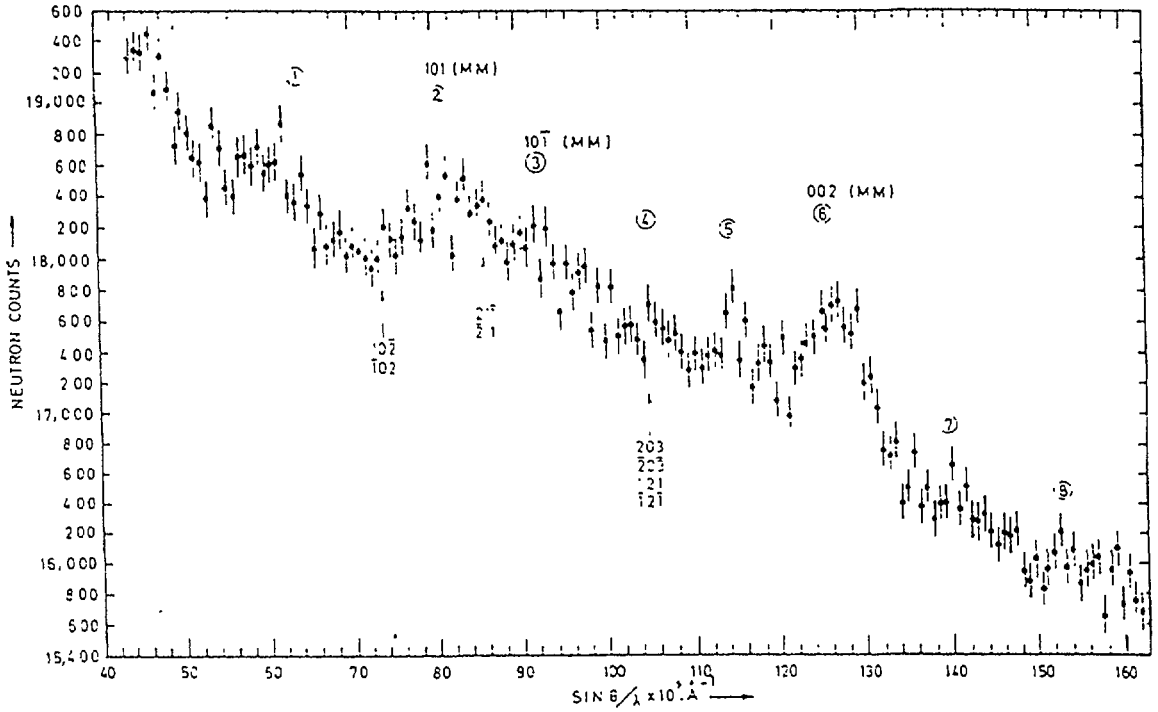


Fig.12 Double-axis Neutron Diffraction pattern of Cellulose-I.

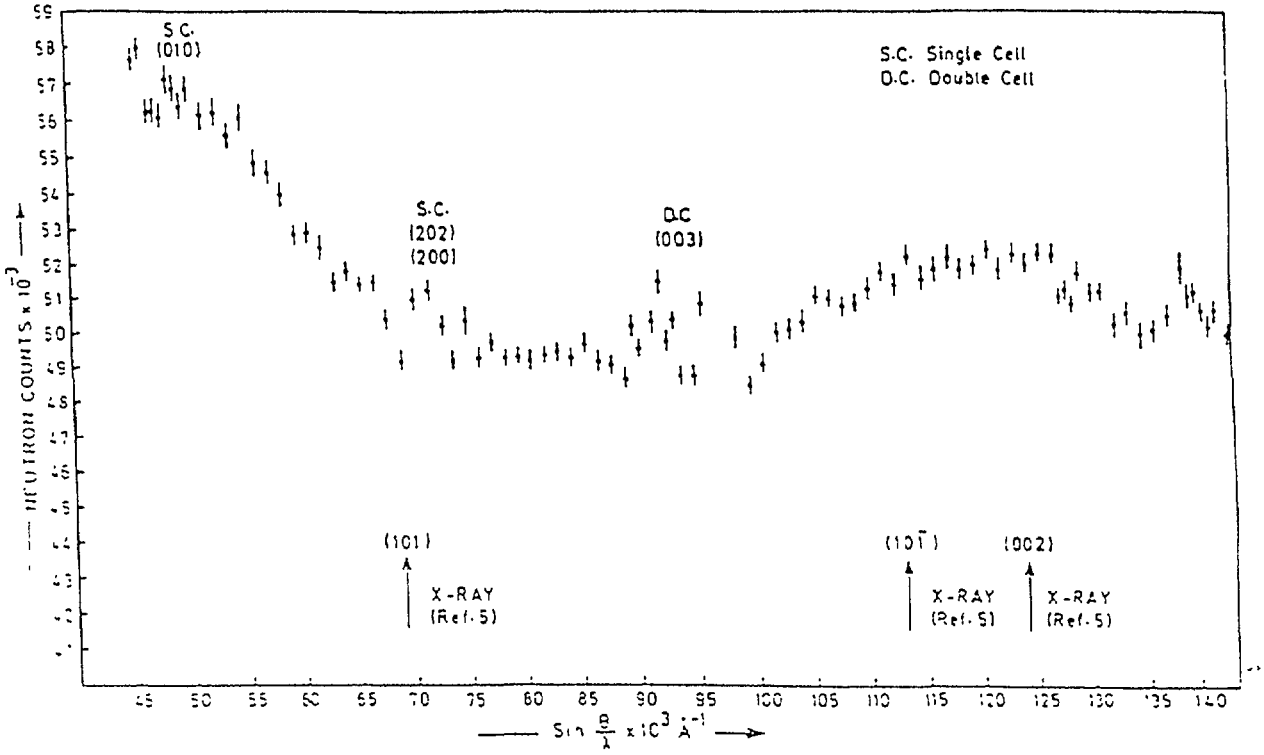


Fig.13 Neutron Diffraction Pattern of Cellulose-II.

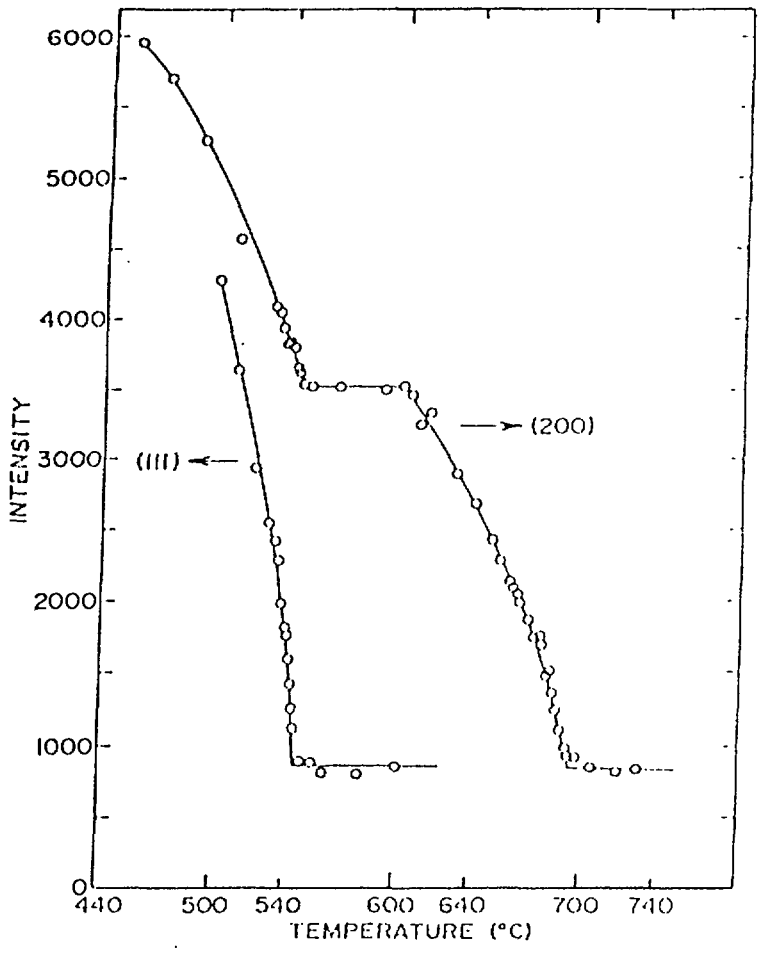


Fig.14 Temperature dependence of the (111) and (200) superlattice peak intensity in Fe-Al (24.50% Al).

of crystallographic planes of the grains (crystallites) with respect to set of macroscopic samples axis i.e. the texture gives information about how large a volume fraction of the material having a specific crystallographic orientation but no information about where in the material these different grains are positioned. When the texture is strong,, most of the grains have almost identical orientations, whereas, when the texture is weak, the grain orientations are almost random.

Texture may have large effects on materials properties, for example on strength, anisotropy, ductility, fracture behaviour, fatigue resistance, and magnetic properties. In characterization of metallic uranium fuels the texture plays an important role. It is important to know the dimensional changes due to prefered of these changes on the type of orientation in the uranium sample. From an industrial viewpoint it is therefore, important to have a precise knowledge of the texture. Furthermore, texture is of fundamental interest as it can be used as a probe to study metallurgical processes: most metallurgical processes, like plastic deformation, recrystalization and grain growth are orientation dependent, which means that texture commonly changes during these processes. By measuring the change in texture, information about the crystallographic processes taking place in the bulk material is obtained. This type of data is not directly accessible through other measuring techniques for example by x-ray diffraction.

At PINSTECH sheets of 99.999% aluminium and copper were cold rolled to 92% reduction in thickness for texture studies by neutron diffraction. The experimental setup is shown in Fig 15. A piece of copper single crystal was also rolled. The results showed that all samples of f.c.c. metals did not attain standard texture. One of the aluminum samples ended up in [200] (002) texture (Fig.16), whereas the sheet made from copper single crystal followed the orientations of the original crystal. Only the specimen prepared from a billet which was carefully heat treated and mechanically worked gave standard orientations [Beg et al 1985].

(5) STUDY OF SUPERIONIC CONDUCTORS

Superionic conductors are materials of latest interest for their possible use in future electric (vehicles) batteries. Superionic materials are the materials which under certain conditions have higher ionic conductivity than those of molten salts and electrolyte solutions. In CaF_2 type materials, the transition from insulating to conducting state starts gradually with temperature. A large proportion of halogen atoms leave their regular sites and assume interstitial positions. The mobile atoms are responsible for high conductivity of the material. The phenomena can be studied with neutron diffraction temperature whereby physics of the continuous order-disorder

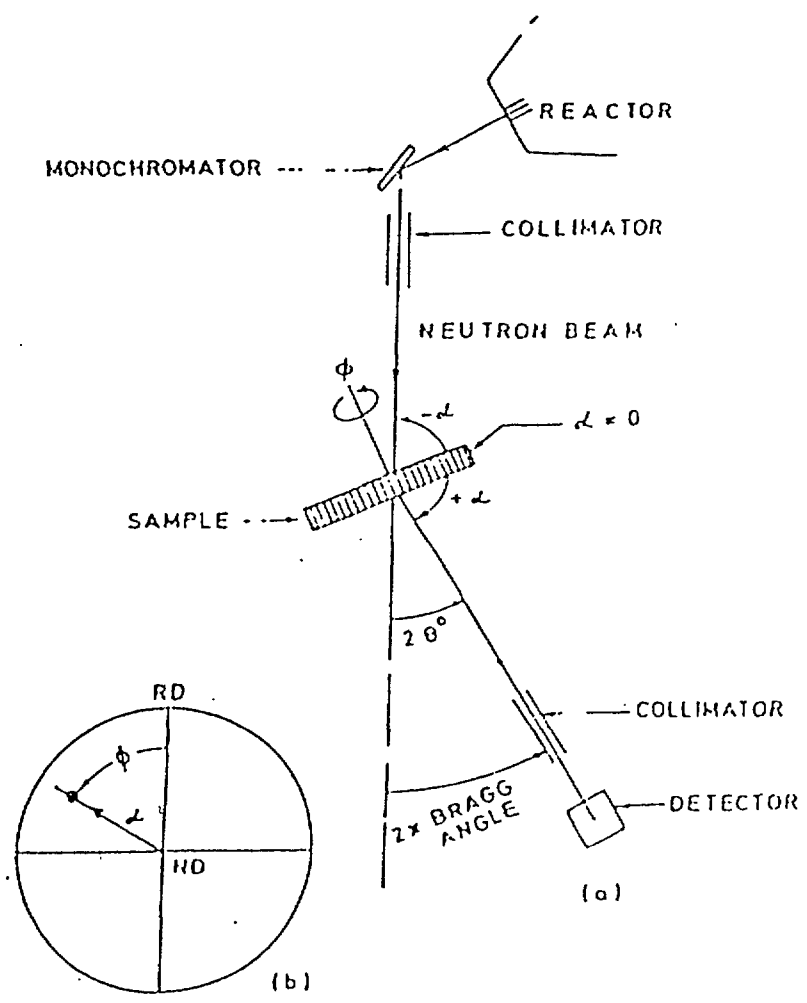
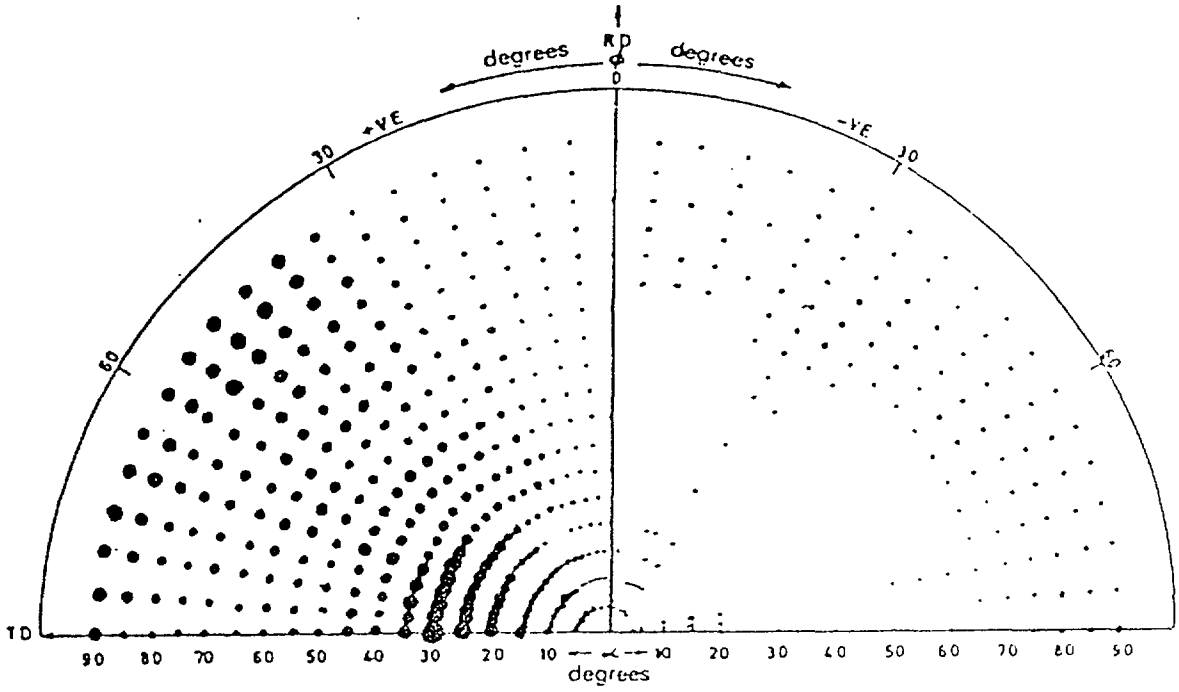
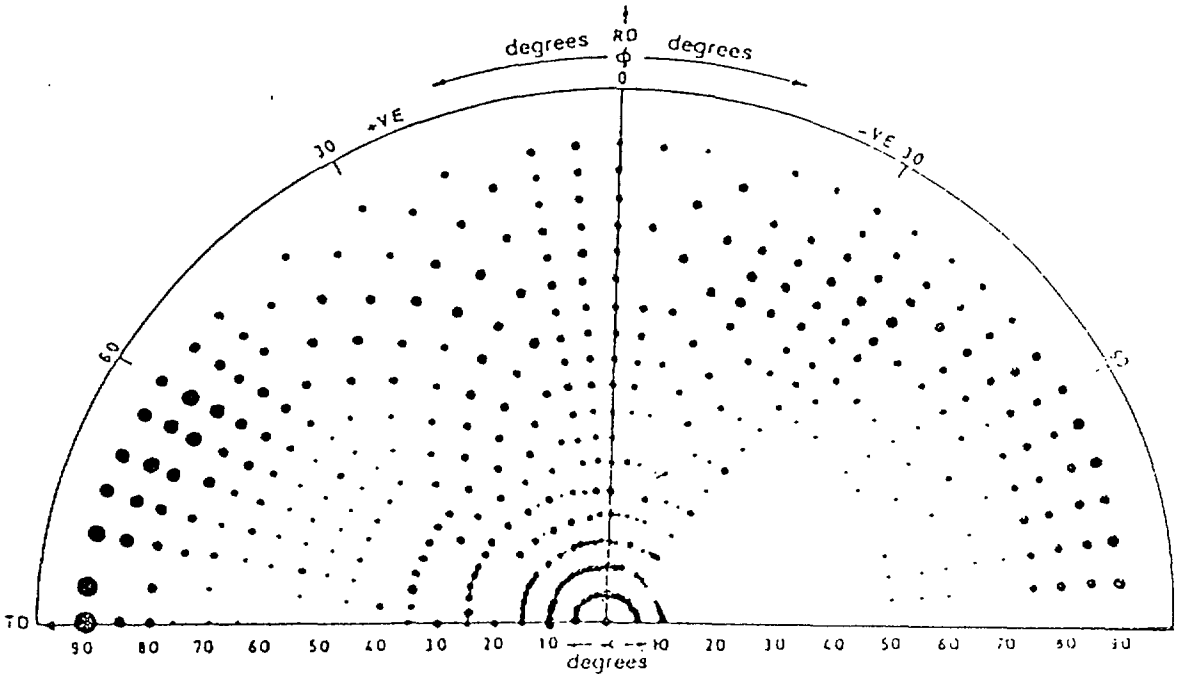


Fig.15 Schematic diagram of Texture. Studies of Rolled sheets by Neutron Diffraction.

transformation can be studied. We have studied the variation of (111), (220) and (200) diffraction peak intensities in $SrCl_2$ up to 800°C. A continuous transition is observed between 650 °C to 700 °C.



(200) pole figure of cold-rolled aluminium sheet (sample 11).



(200) pole figure of cold-rolled copper sheets (sample 11, 87% reduction).

Fig.16 Pole Figures of cold rolled copper sheets.

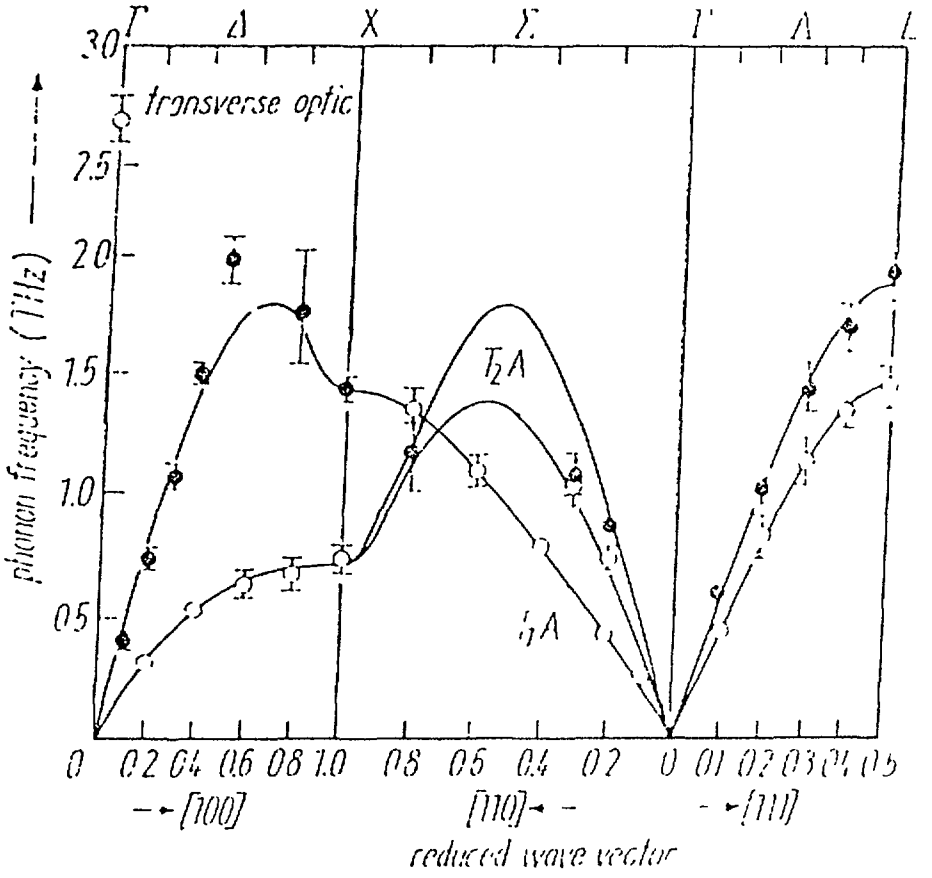


Fig.17 Phonon dispersion in mixed crystal $K_{0.5}Rb_{0.5}I$.

(b) INELASTIC NEUTRON SCATTERING

LATTICE DYNAMICS OF $K_{0.5}Rb_{0.5}I$, $K_{0.5}Rb_{0.5}Cl$ AND COPPER NICKEL ALLOYS

Studies of mixed materials and alloys are very important in the fields of solid state physics and metallurgy. Alkali halides are ionic salts and they are easier to alloy. These materials are also easier to handle mathematically. First detailed lattice dynamical study of mixed alkali halides was carried out at PINSTECH. Acoustic phonon branches, both longitudinal and transverse were measured in a single crystal of $K_{0.5}Rb_{0.5}I$. (Aslam et al 1976). The research on these materials gave the following important results:-

(i) Interatomic force constants and effective ionic charges are reduced on mixing.

(ii) The mixtures are elastically more anisotropic than the constituents.

(iii) All mixed alkali halides have split or double phonons which disperse together. This is a direct result of mass disorder. The present work clarified many misconceptions, about mixed materials, based on the infrared work. Fig.17 gives the dispersion relations and the shell model fit for $K_{0.5}Rb_{0.5}I$. The work was extended to a copper-nickel alloy single crystal. The above effects were also observed for this alloy.

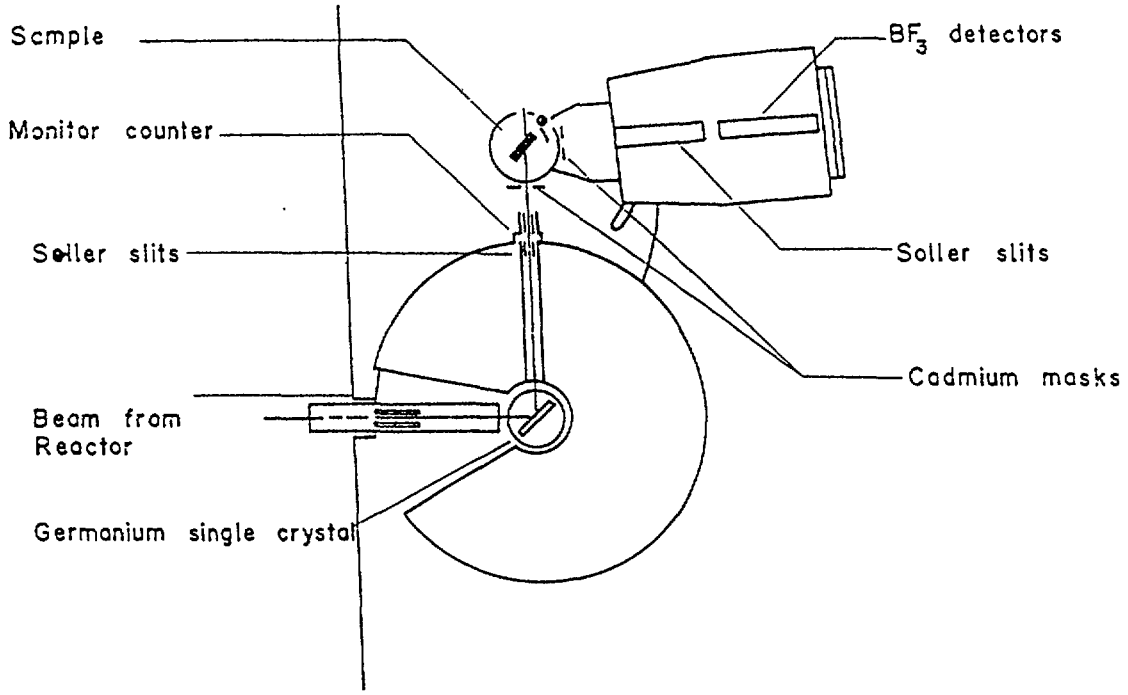


Fig.18 Lay out of a High Resolution Neutron Powder diffractometer.

FUTURE

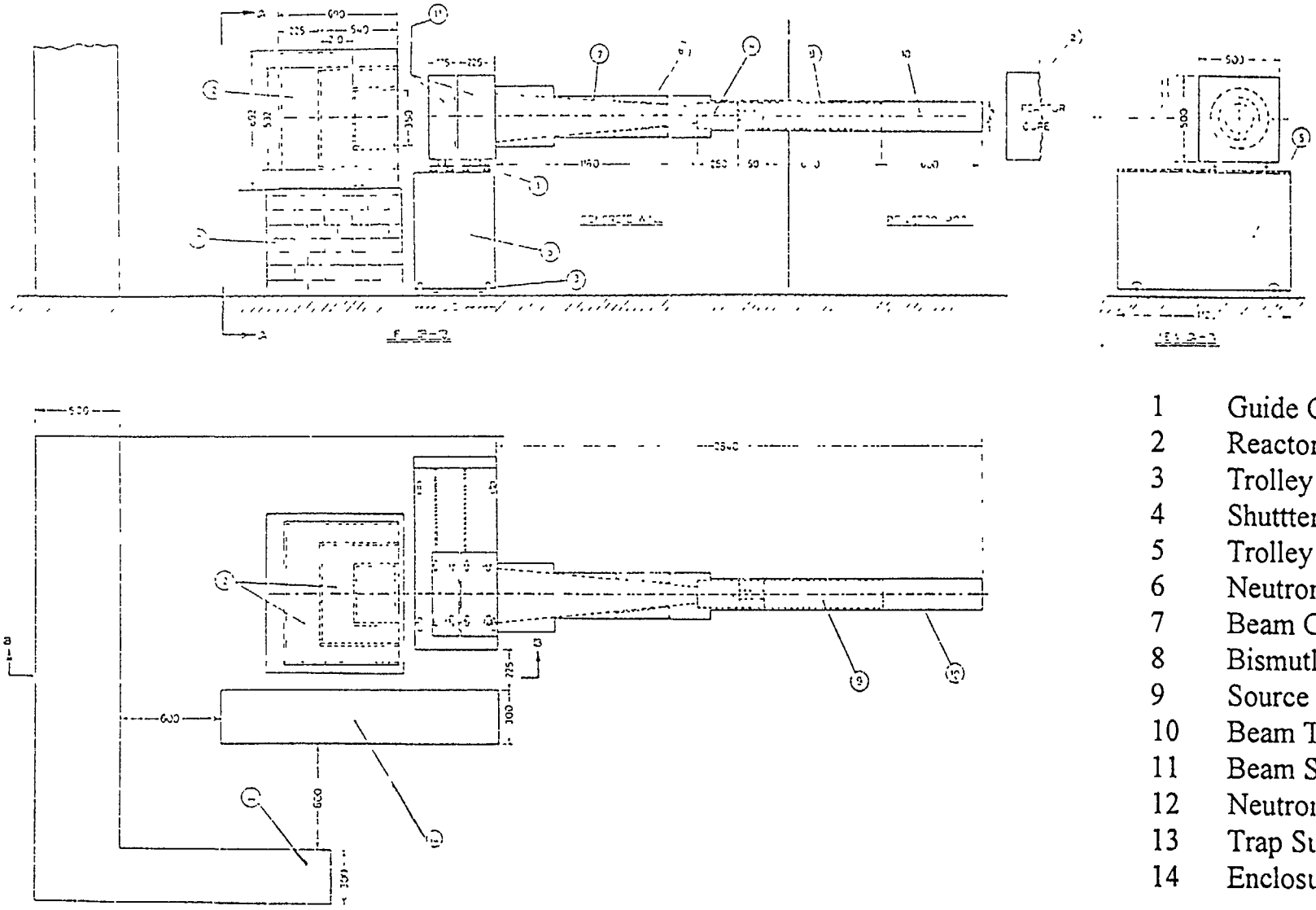
So far we have discussed the facilities available and the physics research that has been done when the reactor power was limited to 5MW. In 1992, the reactor power was upgraded to about 10MW. This would result in approximately doubling the flux that was available before. This would help the experiments to be completed in less time and with more accuracy. In future it is purposed to established the following new facilities around the reactor.

HIGH RESOLUTION POWDER DIFFRACTOMETER

This instrument will be used for the measurement of residual stresses in the materials and will be installed at the beam tube No.4 of PARR-I. The layout of the diffractometer is shown in Fig.18. Disk shaped Ge single crystal will be used as monochromator. Ge crystal is especially needed when second order contamination must be avoided. It will be adopted in the strain scanning mode by the addition of automatic sample scanner giving three orthogonal x, y, z translations and a rotation about a vertical axis. The shape of the gauge volume will be defined by cadmium masks. Positioning of the samples will be achieved using fixed theodolites.

NEUTRON RADIOGRAPHY FACILITY

Neutron radiography technique is based on the principle that the workpiece (sample) is exposed to a beam of thermal neutrons extracted either from the reactor beam port or from a neutron source. The transmitted intensity of neutron beam through the sample exposes the converter screens/foil which then emit radiation sensitive to a detector (Fig. 19). The detector could be a 1) X-ray film, 2) SSNTD detector or 3) video camera with TV screen. The shadow image formed on the screen/foil is then



1	Guide Channel
2	Reactor Core
3	Trolley Wheels
4	Shuttter Wheels
5	Trolley
6	Neutron Streaming Stop
7	Beam Collimators
8	Bismuth Filter
9	Source Block
10	Beam Tube No. 6
11	Beam Shutter
12	Neutron Beam Trap
13	Trap Support Structure
14	Enclosure Walls

Fig.19 Lay out of a Neutron Radiography apparatus at PINSTECH Reactor.

transferred to X-ray film by placing the film in close contact with the screen/foil. The exposed films are then processed to reveal defects and flaws present in the sample.

This technique has an advantage over other techniques because of the random behaviour of the neutron cross - sections for different materials. The attenuation coefficient of different materials increases with increase of the atomic number but neutron cross - sections show a random behaviour. Due to this behaviour of neutron cross - sections the neutron radiography technique is being used to examine both low and high atomic number materials. It can also be applied to active as well as non active nuclear fuels and components. Further advantage is the large penetration power of neutrons in such materials which enables non-destructive testing of several centimeter thick samples.

An increase in the reactor power would almost double the thermal neutron flux available at the sample. This would help reduce the exposure times.

This technique has applications in

1) Nuclear industry
2) Aerospace
3) Explosives
4) Archeology
5) Materials analysis

SILICON TRANSMUTATION DOPING

The use of silicon as a semiconductor material has increased rapidly and the present world wide consumption is now excess of 6000 tons per annum (Meese 1983). Silicon is used in a wide range of electrical and electronic devices and the applications range from high voltage thyristors to power diodes and transistors in conventional electronics and as the base material for integrated circuits used for computer systems and microprocessors.

The most commonly used dopant is phosphorous for n-type silicon. Phosphorous doping can be achieved by the incorporation of the dopant in the molten stage used during the float-zone method of crystal formation. However, this method leads to an inhomogeneous distribution of the dopant and resistivity variations are as high as ±30%.

The application of a neutron transmutation doping process which shows much smaller variations has been of interest to silicon manufactures.

By the capture of a thermal neutron an atom of Si can be transmutated into the unstable isotope Si which decays with a half life of 2.62 hours by the emission of beta-particle to the stable isotope P.

$$^{30}Si\ (n,\gamma)^{31}Si \longrightarrow {}^{31}P$$

By means of this reaction, phosphorous atoms which act as electron donors can be produced in the silicon crystal lattice. Thus the technique results in uniformally doped n-type silicon with accurately predetermined resistivity values.

SMALL ANGLE NEUTRON SPECTROMETER

Normally small angle neutron spectrometer (SANS) use cold neutron sources and high flux reactors for the investigation of defect structures in materials. A new concept using thermal neutrons instead of cold neutrons has been developed recently

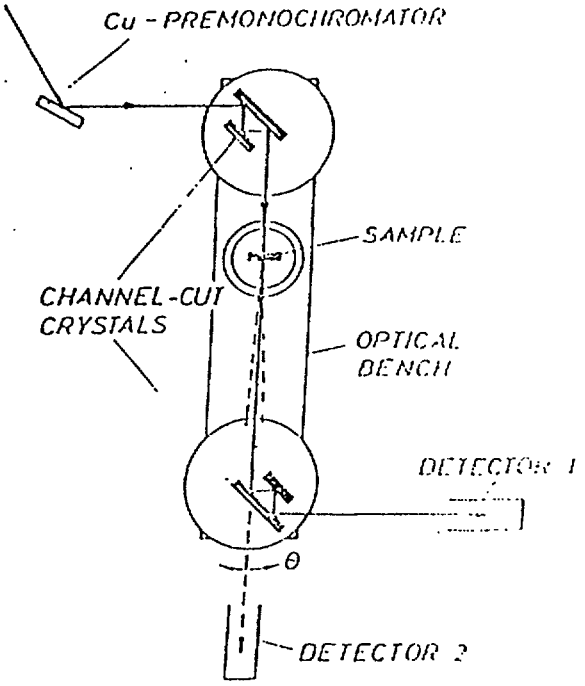


Fig.20 Lay out of a Small Angle Neutron Spectrometer (SANS) using thermal neutrons.

(Rauch 1991). This small angle spectrometer [Fig. 20] is equipped with two monolithic channel crystals increasing the angular resolution almost two orders of magnitude better than for other small angle instruments. In this way the resolution is decoupled from the divergence of the incident beam. Therefore a divergent beam can be used on the sample thereby providing sufficient thermal neutron intensity available at the small and medium flux reactors. This instrument is of versatile use, for example, in the study of; ageing and fatigue effects, precipitates, cracks, and other inhomogeneous ties in materials. The instrument has promising use in the study of materials science and will enable the opening of another type of studies of materials using neutrons.

ENGINEERING STRAIN MEASUREMENTS BY NEUTRON DIFFRACTION TECHNIQUE

Heat treatment, welding and plastic deformation during fabrication can leave strong residual stresses locked in within components. These stresses can affect, the component life in service as they may add to applied loads causing fatigue and failure.

The presence of residual stress in engineering components can affect their mechanical properties and structural integrity. Neutron diffraction is the only measuring technique which can provide spatially resolved non-destructive strain measurements in the interior of the component. By recording the change in interplaner spacings elastic strains can be measured for individual lattice reflections.

Engineering strain measurement necessitates positioning of samples to accuracies of 0.1 mm relative to precisely defined neutron beams. Different positions are examined by translating the specimen through a defined volume. The strain in different directions is measured by re-orienting the specimen with respect to the detectors. Thus the main requirements are a multi-axis manipulation system and precise, repeatable collimation of both the incident and collimated beams.

The diffraction principles for the determination of strains are essentially the same for both X-rays and neutrons when a beam of incident neutrons or X-rays is incident upon a polycrystalline material whereby both scattering and absorption occur. The scattering is not isotropic but is concentrated in "Coherent Bragg reflections" in directions defined by the Bragg equation

$$\lambda = 2d \sin \theta \qquad (22)$$

where 2θ is angle through which the beam has been scattered, d is the interplaner spacing and λ is the wave length of scattered radiation. A change in lattice spacing δd will, for a fixed wavelength produce a corresponding change $\delta\theta$ in the Bragg angle θ, is given by

$$\delta\theta = -\tan\theta \ \delta d/d \qquad (23)$$

where $\delta d/d$ represents the lattice strain ε. It may be expressed as

$$\varepsilon = \delta d/d = -\delta\theta \cot\theta \qquad (24)$$

where the strain measured in that in a direction perpendicular to the reflecting planes and parallel to the scattering vector Q which bisects the angle between the incoming and scattered beams. Strain distribution through out a component may thus in principle be determined by measuring $\delta\theta$ for neutrons scattered from different locations within the component.

STRUCTURE DETERMINATION OF HIGH T_C SUPERCONDUCTORS

Superconductivity is one of the most exotic phenomena observed in some solids. It is the state of matter in which electrical resistance of a material goes to Zero abruptly below a certain temperature known as critical temperature T_c, which is the characteristic of the material. After the discovery of superconductivity in ceramic materials by Bednorz & Muller (1986), over past few years, tremendous efforts have been devoted to the study of physical properties of high Tc superconductors and their non superconducting parent compounds. Numerous experimental studies of the superconductors, in both superconducting and normal states, have revealed many properties that are substantially different from the characteristic of the conventional superconductors. This in turn raises a serious question regarding whether high temperature superconductivity derives from the same pairing mechanism known to be responsible for the conventional superconductors

In order to understand the phenomena of this high temperature superconductivity poly crystalline sample of rare earth oxides and bismuth oxides superconductors doped with Pb Nb, Sb or V will be studied by the neutron diffraction techniques. The objectives are to find the structural and lattice dynamics parameters by elastic and inelastic neutron diffraction techniques. From structural information exact positions of the various parents and substituted dopants and oxygen atoms will be determined. The Rietveld method will be used for the refinement of crystal structure parameters. The structural information will be correlated to zero resistance critical temperature which is an important parameter for the high temperature superconductors.

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FRAMEWORK FOR A SUSTAINABLE DEVELOPMENT OF NEUTRON BEAM WORK IN THE SMALLER RESEARCH REACTORS

F.G. CARVALHO, F.M.A. MARGAÇA
Physics Department,
ICEN/INETI,
Sacavém, Portugal

Abstract

The authors analyse the present situation of research reactors for neutron beam work in the light of the changes that took place in the nuclear field during the last decades.

Trends in supply and demand of neutron beam time in view of the specific requirements of the techniques and of the user's community are outlined. It is argued that resources, both human and material, should be considered in a global perspective, encompassing the national, regional and international levels, where national facilities, mostly low flux research reactors, should be looked upon as a valuable component of a commonwealth of resources to be usefully exploited for the benefit of the neutron user's community at large. The importance of international cooperation to develop a higher level of research reactor utilization is emphasized while suggestions concerning the role of IAEA are made, particularly, to promote the mobility of scientists and engineers directed from developed to less developed countries (LDC's) where research reactors are in operation.

The potential of small research reactors in LDC's as an instrument of the country's general scientific and technological development is pointed out as well as difficulties commonly experienced and essential requirements of a successful performance with emphasis on the importance of establishing close links with the national scientific community and especially with university groups.

The scientific and technological relevance of neutron scattering techniques is discussed. Reference is made to the techniques best suited to modest research reactor facilities as well as to the importance of developing a local competence in instrument design, optimization and construction.

INTRODUCTION

1. At the heart of the problem set proposed to the Advisory Group lie the present and the future of nuclear reactors for R&D work. Both appear to bear in many ways the marks of the origin of these complex facilities.

   Around the world a number of nuclear research reactors of varied designs were built and have been operated under very diferent conditions and with diferent degrees of success, during the last 35 years.

   In Europe (East and West) the number of operable research reactors with a capability for neutron beam work is estimated at 20–30.

   Nuclear R&D reactors, including MTR reactors, can be classified according to the maximum nominal thermal neutron flux at the core as low, medium or high flux reactors, the three classes corresponding to less than 5×10^{13}, less than 5×10^{14} or more than 5×10^{14} neutrons per cm^2 and second.

   To give a few examples: the reactors at Delft and Lisbon are in the first category; those at Risø and Saclay (LLB) are in the second category; the Grenoble reactor is the third category. [1] [2].

2. The majority of these nuclear reactors were built in the sixties, either out of a genuine necessity to foster the progress of nuclear energy (the case of the industrialized nations) or as prestige symbols associated with vague aspirations of power politics. The superpowers played a role in this since in some cases they presented nuclear research reactor installations as a gift to countries in their respective areas of influence.

   Reactors specially designed as radiation sources for non-energy applications, particularly for neutron beam work, generally came at a later time and are few in number.

   Profound changes in the situation of the nuclear power industry have occurred. The effects of these changes together with those of the economic recession of recent years bear upon the nuclear ou nuclear-related scientific and technical community.

   On the other hand the progress in methods and instruments, particularly, radiometric techniques using neutron beams, and the slow but progressive spreading among researchers of the awareness of their specific advantages and of expertise in their utilisation, apparently leads to a steady growth of user demand for access to neutron beam facilities, notably in Europe.

   We are thus facing a situation where several existing sources are reaching the end of their useful life (for physical or political reasons); where pressing problems are posed by the reprocessing and storage of spent fuel where "expertise (in nuclear engineering) in human terms is a declining resource" [1], affecting both design and operation capabilities; while the neutron user's community is probably growing more rapidly than at any time in the past.

3. The user's group in both the developed and the less developed countries, is composed largely of researchers who wish to carry out basic research, mostly oriented and "far from the market". A demand for services in very special areas also exists.Two examples are the aerospace industry (neutron imaging) and health care (neutron capture therapy).

   Quite often neutron techniques are used alongside with other techniques to characterize specific sample and material properties. This means that it is seldom possible or practical to address a scientific problem using a single experimental technique and that the availability of neutron radiometric techniques will not by itself satisfy the requirements of a research project. Cooperation between research groups, normally requiring a certain degree of human mobility, can be the solution to this type of technical limitations. Also, the evaluation of neutron data can be a time-consuming task, this being another reason for the user of neutron techniques to divide his or her time between different work places.

The development of the application of neutron beam techniques cannot be separated from the development of the techniques themselves. This is a kind of work that requires the actual or potential availibility of neutron beams at the researcher's home institution — whatever that means, given the current practice of highly "flexible" employment.

4. The situation of demand and supply that has been outlined, in view of the specific requirements of the techniques and of the user's community, recommends that existing resources (both human and material) and resource development be considered in a global perspective, encompassing the national, regional and international levels. On the one hand, and anticipating a major shortfall of neutron sources at the dawn of the 21st century, imediate efforts should be made to ensure that a limited number (5 – 6) of specially designed state-of-the art high flux neutron sources (of the reactor and/or the accelerator type) would be available world-wide around the year 2000. Access to these facilities should be open on a fair basis to the international scientific community, through adequate schemes of cooperation — an end that should be easier to attain in the present post-cold war world than in the past. On the other hand, measures should be taken to guarantee a more efficient use of existing facilities.

   In the case of medium and high flux reactors in the scientific and technically more advanced countries it would seem unreasonable to increase reactor power or to implement other modifications that may require new safety assessment procedures. One should preferably concentrate on improving the quality and increasing the number of neutron beam-tube facilities, including new and upgraded instrumentation and optimally designed spectroscopy equipment. An increase of available beam--time and a reduction of spare capacity should be the result. Such a course of action would require, of course, a certain amount of capital investment and more funds for current operation expenses.

5. Also, national facilities in the less developed countries — mostly to be classified in the category of low flux research reactors — should be looked upon as a valuable component of a commonwealth of resources to be usefully exploited for the benefit of the neutron user's community at large.

   This would represent a major step towards improving the utilisation of beam port facilities which in several cases have been there for many years without adequate use or no use at all as a consequence of the lack of trained personnel and lack of funds but also due to organizational problems and insufficient motivation at various levels – political and scientific.

From our own experience we know well the catalytic effect of external inputs, material and also imaterial, on the availability of funds and general support for the definition and execution of programmes and projects in land. They can be used as arguments to satisfy political decision makers and they do work more often than not. From this point of view, but also from the point of view of the effective momentum they can communicate to projects, the forms of the visiting scientist and of the scientific mission are particularly relevant forms of cooperation.

If national facilities are perceived as an integral part of an international whole of resources that are complementary in the pursuit of common goals — namely the adaptation of supply and demand for the utilization of neutron beams — that perception will constitute an incentive to cooperate. The question is to what extent is this actually true.

We believe it is true and that the importance of local facilities (as opposed to centralised large instalations) is being recognized under the pressure of circumstances: "(...) apart from their training capabilities, they provide neutron beams for instrument development, monochromator and detector improvements and the testing of components. In addition they allow good science and technology to be performed for those areas where the highest fluxes are not required" [1].

RESEARCH REACTORS IN DEVELOPING COUNTRIES

1. The role of a small research reactor in a developing country deserves some thought. In the first place it is not an easy task to define a developing country. For the present purpose a good indicator is probably the percentage of GNP that is spent in R&D activities.

   If we place the limit at 1% then we will have included Portugal, Spain and Greece in the group of developing countries.

   It is probably true to say that the decision to built a research reactor in a developing country was taken at the time for a combination of reasons: aspiration for a nuclear power programme, more or less vague military ambition, prestige. At the political level the urge to develop general purpose scientific and technological capabilities has probably played a minor role in that decision.

   Typically, as time went by, the original intentions were largely frustrated. Also, since it is generally easier to make decisions to invest capital than to finance current expenses the facilities lived a stagnant life, until, eventually the original justifications having disappeared, a fight for survival set in.

   Notwithstanding this long and sometimes despairing evolution, one should contend that there is a real value in research reactors as tools for carrying out fundamental and applied research work in various disciplines and for the training of young

scientists and technicians in basic science and modern technology. In fact there is no hope for a developing country to assert itself in the modern world without a huge effort to raise the level of scientific and technical education and professional training of the younger generation. The development of R&D activities is an indispensable component of that effort. Also expertise in nuclear techniques has a wide horizontal range of applications in almost all spheres of social activity.

A successful activity in science and technology requires particularly demanding forms of organization and management of resources (human, material and financial) that are often beyond common standards in developing countries. This is the more so in the case of a nuclear reactor installation for reasons of complexity, safety requirements and sheer dimension — a research reactor has a good chance of being by far the largest scientific and technical single facility in a developing country.

2. In order to fulfil a useful function a research reactor should establish as many links as possible with the scientific and teaching community of the country and also with the international scientific community. Isolation at home should be fought with perseverance. This can be done especially by establishing contacts with university groups that are potential users of neutron techniques and by introducing this techniques to interested colleagues.

   There should be a disposition of the reactor group (or the neutron scattering group) to offer experiment design capabilities and support services to carry out experiments and to analyse data.

   To attract teachers and students of different levels some thought should be given to the preparation of demonstrative experiments.

   To perform these functions a small motivated team (4 – 5 people) of good scientific and technical level is necessary. This means permanent posts with good career prospects for the small group of scientists and engineers. The group should be involved in a few scientific projects of their own, that should include some form of partnership or cooperation with foreign groups.

   A significant part of the group's activity should be dedicated to the development, construction and improvement of instrumentation. This is important for the group's work and as a source of technical spin-offs to the community.

   The group would also provide the necessary technical and scientific basis to support forms of international cooperation to be carried out "in situ". The forms that appear to be the most interesting are the following:

   - visits of scientists and/or engineers to help design, build and install new or displaced instruments, with the right to become regular users of the instruments. Displaced instruments may be instruments from facilities that have been shut

down or also instruments from larger facilities (to be adapted locally or refurbished) for which new instruments have been substituted in their original working place.

- scientific visitors interested in having access to existing instruments or beam tube facilities where beam time is almost entirely free, to carry out "odd" experiments, to perform preliminary analysis of special samples and or for other purposes.

  In certain cases, the lucky visitor would be suprised to find locally exceptionally good people at the level of technicians and also bright young researchers that are unfortunately not adequately supported in their home institutions.

INSTRUMENT DESIGN

1. The importance of the development and application of new and advanced materials is well understood in the developed and in most developing countries. It is also common knowledge in materials science that relating bulk properties to microscopic structure is an essential step towards the development of new materials and new processing technologies. Namely, information is required on relations between structure, properties and performance and how they are affected by processing.
   Neutron scattering techniques have proved very fruitful in the investigation of structural properties of complex materials. As a tool for the characterization of materials, thermal neutrons offer several well–known and unique advantages over other common probes such as protons, electrons or X–Rays. Since they carry no electric charge, they are deeply penetrating. Because of their low energy they are non–destructive. Low energy also makes them ideal for inelastic scattering studies. However, inelastic scattering cross–sections are invariably very low and such studies are best left to high flux reactor facilities. On the contrary, in elastic scattering experiments a substantial fraction of the incident neutron flux is actually measured, and these studies can be successfully carried out at reactors with modest fluxes. Furthermore–and this is a key point–some of the neutron elastic scattering techniques are particularly well suited when it comes to tackle a few important problems of technological relevance on a level likely to lead to practical progress.
   Generally the techniques which are felt to be particularly well suited to modest research reactor facilities [3, 4] and which probe the microscopic structure of materials, fall into two broad categories depending upon the magnitude of the angle θ by which the neutron beam is scattered:

- Small Angle Neutron Scattering (SANS) ($0.01< \theta < 10°$) which probes homogeneity at a scale of 10–1000 Å, such as pores or precipitates in a matrix, macromolecules in solution, etc.

- Powder Diffraction, for larger angles ($\theta > 10°$) which explores structure on the interatomic length scale (1–10 Å).

- A third interesting technique is neutron reflectrometry for surface studies.

The techniques have different degrees of complexity and different budget requirements. They can be implemented at reactors with ambient sources and demand a minimum expertise and design capabilities in the fields of fine mechanics, radiation shielding, electronics and data accumulation and processing.
We have some experience in the implementation of these techniques. Our first instrument was a time–of–flight diffractometer for powder diffraction and the second, now under construction, is a SANS facility. In both cases we have learned that design optimization is the key to get the most out of the available reactor flux, through the instrument.

2. A neutron scattering facility is often thougth of as a standard black box (which can be installed in no matter what type of neutron source): a neutron beam goes in and data comes out. Such a facility when installed at a low or medium flux reactor would yield a count rate lower by one or two orders of magnitude, respectively, than the corresponding count rate when installed at a high flux reactor.
On this basis it is sometimes argued that it is pointless to try and install most state–of–the art scattering facilities at the smaller research reactors with ambient sources.
We have a different view. To start with, we reject the concept of the standard black box facility. We argue that each case is a case, meaning that a specific lay–out has to be studied and an optimized design carried out for each particular facility, to make the most efficient use of the available neutron source and minimize all possible sources of loss. That is, for each instument a particular design can be developed, optimized for the instrument's required performance and for the characteristics of the reactor installation. This has been demonstrated for different types of neutron scattering instruments. In our case we have studied the optimization of a diffractometer for stress measurements with the result that a count rate gain of one order of magnitude could be achieved relative to the use of a standard diffractometer for stress measurements [5]. Taking into account that the instrument design should be optimized for the actual installation characteristics, it is no longer true that the ratio of the count rates of two such instruments installed at two different reactors

is the ratio of the corresponding maximum nominal thermal neutron fluxes (just as the ratio of thermal neutron fluxes is not the ratio of the reactor thermal powers).

For the last four years, we have been studying the optimization of SANS instruments for different types of neutron sources (pulsed, steady–state); different collimation assemblies (guide tubes, single pin–hole and multiple pin–hole collimators), and different detector assemblies and sizes. The main results have been reported and discussed [6–11].

One result is that the design which had been considered for about 20 years to be the optimum one – the equal–flight–path design – does not lead to the highest count rate for a given measurement with fixed resolution. Its main handicap is the use of an effective neutron source area which is invariably a fraction of the total available area. The design requires a source area equal to twice that of the detector counting cell (around 2 cm^2) whereas the available source area at a beam port is about one order of magnitude larger.

We have shown that when the full source area available is used the count rate gain relative to the conventional design can go up by a factor equal in the limit to 4 or 16, for single and multiple pin–hole collimation, respectively. The actual gain factor depends on the physical constraints and the characteristics of the installation site as well as on the actual size of the source area.

Our SANS instrument optimization results have been experimentally confirmed at Risø National Laboratory [12]. We recognize that the implementation of a large source area design at a medium or high flux reactor is often very difficult because a large number of scattering instruments are usually crowded around the neutron source. This is not the case, in general, for the lower flux reactors. Wherever possible the use of the large source area design with multiple aperture collimation is recommend. Our neutron scattering group will be happy to collaborate in the design of SANS instruments tailored to match specific neutron source characteristics, space constraints and other installation room characteristics.

FINAL REMARKS

To develop a higher level of research reactor utilisation, particularly for neutron beam work, international cooperation is a decisive factor.

Increased mobility of scientists and engineers from developed to developing countries — that is, contrary to the normal trend — is essential. Also, existing capabilities in the less developed countries should not be underestimated. They can contribute beneficially not only to specific research activities in the developed countries but can also be used to enhance capabilities in other developing countries. It is probably true that up to a certain degree of complexity problems experienced by groups in certain less advanced

countries can be better solved — more economically and more efficiently — by groups working in other developing countries that have managed to attain a higher level of scientific and technical expertise.

Solved and unsolved problems in different developing countries often do not coincide. To take advantage of this an effort should be made to foster communication and diffusion of technical information between research groups and institutions in different developing countries.

The availability of good auxiliary services and infrastructures, mainly workshops, is an important asset. Related to this and equally important is expertise in design and construction of instruments, special parts and components.

The Agency could act as a vehicle to spread information on this capabilities, and eventually act as a match–maker and sponsor of contracts between centers in developing countries for the supply of certain specific types of equipment and services.

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BASIC RESEARCH IN SOLID STATE PHYSICS USING A SMALL RESEARCH REACTOR

V. DIMIC
Institut Jozef Stefan,
Ljubljana, Slovenia

Abstract

The 250 kw TRIGA Mark II reactor of the J. Stefan Institute in Ljubljana, Slovenia, is a light water reactor which is used for basic research in the following main fields: solid state physics, neutron radiography and autoradiography, neutron dosimetry, reaktor physics, neutron activation analyses, etc. In the field of solid state physics the rotating crystal time-of-flight spectrometer together with the cold neutron source using solid methane as a moderator has been used to measure incoherent inelastic neutron scattering spectra in order to study optical and acoustic vibrations, phase transitions, and diffusive motions in different materials. Nondestructive studies using neutrons are performed for inspection of internal distribution of elements in alloys, structure studies in polycrystalline materials, etc. For these studies the following methods are used: neutron radiography with thermal, resonance and fast neutrons, neutron induced autoradiography using solid state nuclear track detectors and radiography with back-diffused electrons.

Research reactors with the flux in the region of about 10^{13} n/cm^2 s may serve as an important research tool which is in many fields competitive with the reactors in the higher flux region. Use of low (or medium) flux reactors for research has often demanded a higher degree of ingenuity and a sense of pioneering, whic are important characteristics for scientific research of high quality. In order to achieve this, the choice of the reactor instrumentation has to be implemented which makes optimum use of this flux, the reactor needs a certain amount of auxiliary "background laboratories" and the link between a nuclear centre and a University. The acquisition and installation of nuclear reactor has mainly been justified on the following ground: to produce isotopes, to support basic (and applied) research in various fields and as a tool for training in the nuclear sciences. These objectives have been met with different degrees of success. As an example of how these objectives could be fulfilled rather successfully with a small research reactor like a 250 kW TRIGA Reactor the basic research programme in solid state physics carried out at the J. Stefan Institute in Ljubljana (Slovenia) will be discussed in detail.

1. TRIGA Mark II Research Reactor at the J. Stefan Institute

The 250 kW TRIGA Mark II reactor began operation in May 1966. It is a light water reactor, with solid fuel elements in which zirconium hydride moderator is homogeneously distributed between 20% or 70% enriched uranium. The maximum neutron flux in the central thimble is 1.10^{13} n/cm^2 s. A 40 position rotary specimen rack around the fuel elements, a pneumatic transfer rabbit system, as well as a central thimble are used for irradiation of samples. The thermal neutron flux at the rotary specimen rack is 1.4×10^{12} n/cm^2 s.

Other experimental facilities include two radial and two tangential beam tubes, a graphite thermal column and a thermalizing column.

2. Research in solid state physics by neutron scattering

Through neutron inelastic scattering experiments detailed information can be gained on phonon spectra and dispersion curves, phonon lifetimes, thermal diffusion of atoms, the time dependence of the case when a sample contains atoms with a high incoherent or coherent cross section for neutrons. For instance, neutron scattering by hydrogenous substances is almost entirely incoherent due to the large incoherent cross section of the proton. Therefore, when hydrogen atoms are present in the molecule, neutrons are scattered mainly by these atoms, providing relatively easy interpretation.

2.1. Rotating crystal time-of-flight spectrometer

Incoherent inelastic neutron scattering spectra can be measured by cold neutrons (energy gain method) with a neutron energy lower than 5 meV (a wavelength is higher than 4Å. The spectra of scattered neutrons are very often measured by a time-of-flight method. A pulsed beam of almost monoenergetic neutrons, produced by rotating single crystals of lead or copper, is scattered by a sample into a bank of neutron detectors arranged at different scattering angles to the incident beam direction at a distance 2- 3 m from the sample. The energy spectrum of the scattered neutrons is analysed by measuring the scattered neutron time-of-flight from the velocity selector to the neutron detector by means of a multichannel analyser.

Such a spectrometer, where the neutron spectra are measured at four scattering angles simultaneously with 4 banks of He-3 detectors, was built at our reactor. A cold neutron source using solid methane as a moderator was placed in the tangential beam hole. With this cold neutron facility it is possible to perform experiments which otherwise could not easily be carried out with a low flux reactor[1-3].

The rotating crystal time-of-flight spectrometer together with the cold neutron source represent very valuable experimental tool for research in the field of solid state physics by neutron inelastic and elastic scattering. At our reactor this method is used for the study of optical and acoustic lattice vibrations, phase transitions, and diffusive motions in [4-14]:

- ferroelectrics and antiferroelectrics:
  KH_2PO_4, $CO_2Sr(CH_3\text{-}COO)_6$, $PbCa_2(CH_3\text{-}COO)_6$, $NH_4H_2PO_4$
- liquid crystals: MBBA, PAA, Na-palmitate, Na-stearate, anisalazine, etc.
- biological samples: LiDNA, NaDNA
- hydratization of cement.

2.2. Liquid methane cold neutron moderator

Thermal and subthermal neutrons are a unique tool for research on condensed matter phenomena because their wavelengths are comparable to those of bound atoms. The

magnitude of the energy transfer involved in these phenomena is about 0.1 to 100 meV. In order to detect these small energy changes one is often forced to use very slow neutrons; 4 Å to 6 Å is a convenient wavelength region. Reactors used as slow neutron sources in these scattering experiments usually have moderator temperatures near 60°C. Thus, a Maxwellian spectrum of neutrons emerging from a reactor at these temperatures has a maximum around 1-2 Å and the flux falls off rapidly towards higher and lower energies. Therefore, cold neutrons represent only about 1% of the total spectrum. It is evident than an increase in the flux might be achieved if the reactor moderator temperature could be lowered. For neutrons of wavelength 4 Å, an increase of an order of magnitude is possible, and much higher increases are obtained at higher neutron wavelengths.

Many materials have been used as moderators, for example, liquid hydrogen, several liquid hydrogen/deuterium mixtures, liquid deuterium or heavy water, methane and methyl alcohol. Liquid hydrogen and methane have the best moderating properties because of their small-down length, long scattering cross section and because atoms are loosely bound, which gives a high density of states at low energies.

At our reactor, TRIGA Mark II, a time of flight spectrometer has been installed which is used for cold neutron scattering experiments. The flux of neutrons in the low energy tail of the thermal spectrum for the TRIGA reactor was inconveniently low. It was therefore decided that a cold source should be placed in the tangential beam hole in order to increase the cold neutron flux by a factor of five to six.

The loop was installed in the reactor and at first it was tested with methyl alcohol as the moderator; however, a large decomposition rate of methyl alcohol was found. Methyl alcohol was replaced by methane, with a boiling point at -161.5°C and freezing point at -148°C.

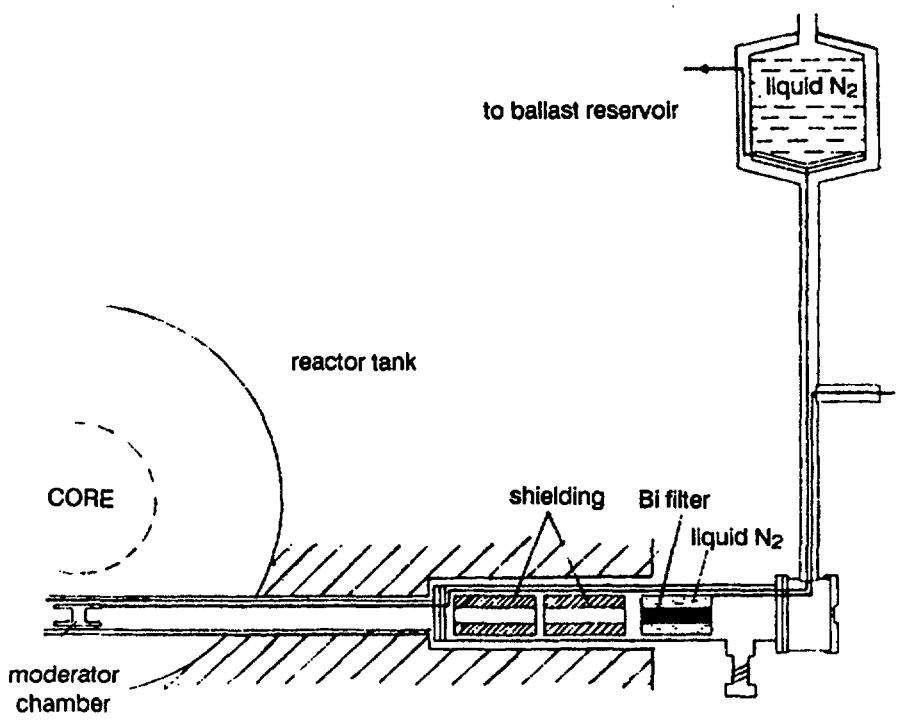

Fig. 1. General layout of cold neutron source facility.

The neutron cold source was inserted into the tangential beam tube 15.0 cm in diameter, traversing the graphite reflector (Fig. 1). The time of flight spectrometer is placed on one side of the beam tube and the service end of the cold source is on the other side. Vacuum pumps, inlet and outlet pipes for methane and liquid nitrogen, and the control system are situated at the service end of the cold source. The berillium filter with collimators is placed in another vacuum jacket. With this arrangement, it is possible to take out from the beam hole either the beryllium filter or the cold source. The vacuum is maintained by a diffusion pump and is better than 10^{-4} torr.

The optimum shape and position of a moderator chamber for our cold source has been carefully investigated[1]. We have found that the optimum moderator chamber should be shaped in a form of a black-body scatterer.

The spectra of neutrons, slowed down in the black body shaped moderator chamber using methyl alcohol and methane as moderators, were compared with the spectrum of cold neutrons produced by a graphite slab with a length of 15 cm and diameter of 13.8 cm and located at the centre of the reactor core. The results are given in Table 1. where the moderator efficiencies are tabulated.

A measurement taken with the moderator chamber filled with water at ambient temperature has been used as a reference point.

Table 1. Relative cold neutron output

Water T = 300 K	Graphite T = 300 K	Methyl alcohol T = 300 K	Graphite T = 100 K	Methyl alcohol T = 88 K	Metlane T = 88 K
1.0	0.62	0.93	0.66	4.3	5.1

We conclude that the construction and operation of a cold neutron source is a fairly straightforward matter and that, with a source such as that described, reliable and continuous operation can be readily achieved. During the operation normally lasting 100 h, no special checking is required. However, the gain of cold neutrons is quite considerable. With such a cold neutron facility it is possible to perform experiments which otherwise coud not easily be carried out.

3. Nondestructive studies using neutrons

In the last thirty years significant efforts have been devoted to developing different nondestructive methods using neutrons from the research reactors for inspection of internal distribution of elements in alloys, structure studies in polycrystalline materials and long range fluctuations of molecular or magnetic density in matter. The most useful methods in order to investigate all this phenomena are neutron radiography especially microneutronographic and neutron induced autoradiographic techniques, neutron diffraction and small angle scattering of neutrons. At the reactor in Ljubljana research in the fields of neutron radiography stimulated the development of related fields like microneutronography, neutron induced autoradiography and radiography with back-diffused electrons[15-23].

3.1. Neutron radiography

Neutron radiography is a valuable non-destructive method, which can be placed along side the more familiar and well- developed X-ray and γ-ray radiography. However, neutron radiography should not be regarded as a method competitive with X and γ-ray radiography, but has to be considered as an additional radiographic method, which provides complementary and, in some cases, unique information relative to other radiographic methods.

In general, there are two ways in which neutron radiography can be applied:

a. As a high resolution neutron radiographic method, capable of investigating in a qualitative and quantitative manner the microstructures and composition of a specimen. This technique is called **microneutronography**.

b. As a defectoscopic method capable of detecting on a macroscopic scale various detrimental defects in the objects being investigated.

3.1.1. Principles of microneutronography

Microneutronography is a high-resolution radiographic method for inspection of the internal distribution of highly neutron- attenuating elements in alloys. The method can be used either for viewing the internal microstructure in the sample or could possibly be applied in a quantitative manner for the determination of the internal composition of a specimen by means of microdensitometric readings. In microneutronography applied to viewing the internal microstructure an image is obtained by placing a specimen, usually thin (from about 100 μm and up to a few 1000 μm), in intimate contact with a suitable neutron image detector and exposing it to a collimated beam of neutrons. The primary image represents the spatial variations in the intensity of the transmitted neutron beam owing to different attenuation by the various phase in the specimen. Thus the chemical heterogeneity is revealed as a variation in photographic density. The microneutronograph is then obtained by enlarging the primary image by means of the light microscop. The resolution of the neutron-imaging technique must be adequate to allow the observations of internal microhetereogeneities at useful magnification of the primary neutron radiograph from at least 5 x up to 100 x. A resolution of about 5 μm can be achieved by using track-etching techniques together with boron-10 converters.

The aim of quantitative microneutronography is to determine the concentrations of strong neutron absorbing elements. Quantitative microneutronography can be applied either to obtain concentrations averaged over structural details in the area of the scanning window of the densitometer and throughout the specimen thickness, or concentration in a particular phase.

3.1.2. Neutron induced autoradiography

Microneutronography is a suitable method for the qualitative and quantitative investigations of the distribution of strong neutron-absorbing elements, especially of light elements (B, Li). However, the method is limited by its sensitivity and spacial resolution. The complementary method to microneutronography, with required sensitivity and spacial

resolution is neutron-induced autoradiography. In cases where a specimen contains elements like B, Li or U, which by themselves emit charged particles upon neutron absorption, the presence of these elements can be revealed without any intermediate converter screen directly by detection of reaction particles with a suitable detector. It should be noted that in this case the collimation of neutrons is no more required. In this method the specimen to be examined is polished and appropriate detector is placed against this surface. The autoradiograph represents the distribution of particular elements on the surface in contact with the detector. As a detector of charged particles could be used various Solid State Nuclear Track Detectors (SSNTD) where the track-etching is used.

4. Conclusions

This paper has briefly summarised the variety of experimental activities in solid state physics using a small (or medium) research reactor and which can easily be a subject of fruithful cooperation between laboratories in different countries. The research activities in this field have generated development of a variety of experimental facilities and other accessories to provide a self-sustaining growth in science. As to the choice of an eventual basic research programme one should try to consider the infrastructure available in the country and the connections to the already existing activities in the country.

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THE NEW SWISS SPALLATION NEUTRON SOURCE SINQ AND ITS PLANNED DAY-ONE INSTRUMENTS

Status as of spring 1993

G.S. BAUER
Paul Scherrer Institute,
Villigen, Switzerland

Abstract

The paper briefly reviews the concept and layout of a cw spallation neutron source for condensed matter studies, SINQ, under construction in Switzerland. Without involving fissile or fissionable material this technique allows to build a medium flux neutron source essentially with available technology. A target development program to make optimum use of the proton beam driving the facility, is described and performance estimates for the source are given. A carefully designed experiment support system inlcuding a cold moderator, super mirror coated neutron guides and beam shutters incorporated in the bulk shield will help to optimize the performance of the instruments. Six instruments under construction and planned for operation when the source will be commissioned are described briefly.

1. Introduction

This paper was prepared upon request by the organizers as one of the introductory talks at the IAEA-advisory group meeting on Uses of Research Reactors for Solid State Studies held at Vienna from March 28 to April 1, 1993 in order to outline current activities in a small industrialised country in the field. It is intended on the one hand to spread information on new developments, in particular on the implications of using spallation as an alternative method for neutron release from nuclei and on the other hand to serve as a starting point to discuss basic instrumentation needs at a low-to medium flux neutron source in order to implement a rather comprehensive research programme using neutron beams.

2. The incentive for a new neutron source in Switzerland

Since 1957 Switzerland has been operating and utilizing its light water pool reactor SAPHIR for irradiation and beam hole based research. After two upgrades which included the provision of beryllium reflector elements at the core position adjacent to the neutron beam tubes, the reactor was operated at a power level of 10 MW for several years. The effect of the use of beryllium elements between the fuel and the beam tubes was an increase in useful thermal flux and a decrease of fast neutron (and γ-radiation) in the beam ports. While not eliminating totally the need for filters in the beam, their thickness could be reduced. In this way the Beryllium elements are of double benefit to the neutron scattering instruments. Fig. 1 shows the core configuration of the reactor and the instruments arranged at the different beam tubes. It is remarkable that triple axis spectrometers have been used rather successfully on this reactor.

Based on these available instruments a neutron scattering tradition has developed which resulted in a successful research programme as documented by the large number of guest scientists and publications per year and the annual reports published regularly by the Laboratory for Neutron Scattering (LNS).

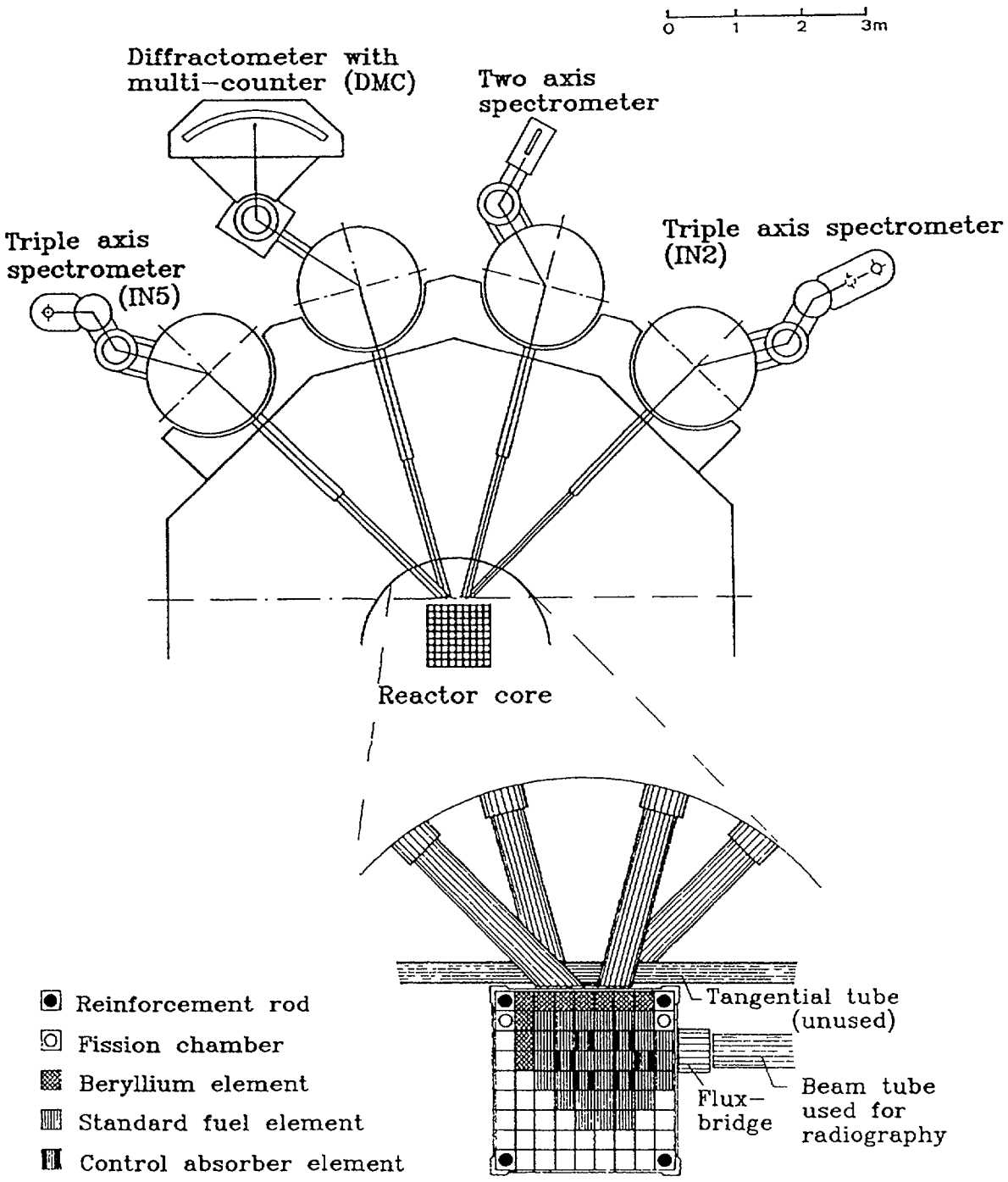

Figure 1: Core disposition and arrangement of neutron spectrometers at the PSI-reactor SAPHIR

In 1992 a review of the reactor safety under the rules now in effect suggested that the operational safety margin might not be sufficiently large at 10 MW and the reactor power was reduced to under 7 MW.

Independent of this setback it had been decided in 1987 that Switzerland was going to maintain and expand its neutron-based research activities on a longer term basis and to provide a new neutron source for its national and international community of neutron users. Advantage was to be taken of the planned upgrade of the isochronous proton cyclotron at the then SIN (Swiss Institute of Nuclear Research, now part of PSI). Since this accelerator had been developed for pion and muon production only, about 80% of the accelerated beam used to end up in a beam dump. The cyclotron was orginally designed for operation at 590 MeV, 100 μA.

Due to the very low loss level in the machine it was concluded that an upgrade to the regime around 1.5 mA was possible with a new 72 MeV injector, working on the same principle as the main ring. In order to make operation at this current level a real option, most of the beam transport line and the meson production targets had to be rebuilt with extra shielding and more intense cooling especially in those positions, where beam losses would be unavoidable. This task was completed in 1991. Since this time the upgrade of the ring accelerator itself is in progress, with a planned yearly increase in beam current by some 250 μA. Currently 500 μA can be delivered. The goal for 1993 is 750 μA. When operating at a higher current level, the usage of the accelerator could be expanded by adding a suitably designed target to create a medium flux neutron source. Under the given circumstances this would be a relatively cheap way to ensure the future availability of neutrons for the country. At the same time it seemed possible to meet the growing demand for long wavelength neutrons by incorporating a cold moderator in the design from the very beginning.

3. Brief review of the source concept

Neutron scatteres were involved in the decisions made early on in the conceptual planning stage and existing experience from other neutron sources was incorporated to the largest possible extent, subject of course to existing boundary conditions.

One of these boundary condition was that the PSI-cyclotron is operating at an rf-frequency of 51 MHz but apart from this has no macro pulse structure. Therefore, the concept of SINQ is that of a steady state neutron source and hence aims at providing the highest possible neutron flux that can be generated from the available beam power and at offering the maximum possible space for instruments. For this reason the proton beam is injected into the target from underneath, making 360 degrees around the target accessible for beam extraction. The concept provides for a multiple containment of all potentially volatile radioactivity produced inside the target block itself. For this purpose a double-walled steel tank with contained atmospheres will surround the target and the moderator tank. A perspective view of the facilities is shown in Figure 2. The target block, which is shown partly cut open to display the space in which the steel tank was inserted, will surround a 2 m diameter heavy water tank in whose axis the target with essentially cylindrical geometry is positioned (Fig. 3). The D_2O will guarantee a long life time of thermal neutrons and hence a high thermal flux. Two spectrum shifters (cold or hot moderators) can be located near the maximum of the thermal neutron flux in the D_2O-tank. One of them will be a 20 litre liquid D_2 moderator, feeding neutron guides and one pair of beam tubes. A bundle of 7 neutron guides will serve to transport cold neutrons to the neutron guide hall, where the majority of instruments will be located.

All beam tubes are designed as twin pairs to minimize the flux pertubation in the D_2O and still have a large number of possible instrument positions around the target block. More details can

be found e.g. in the status report delivered at ICANS XI [1]. The whole of the services building shown behind the target block will be located inside the target hall. Its construction is due to start early in April, 1993.

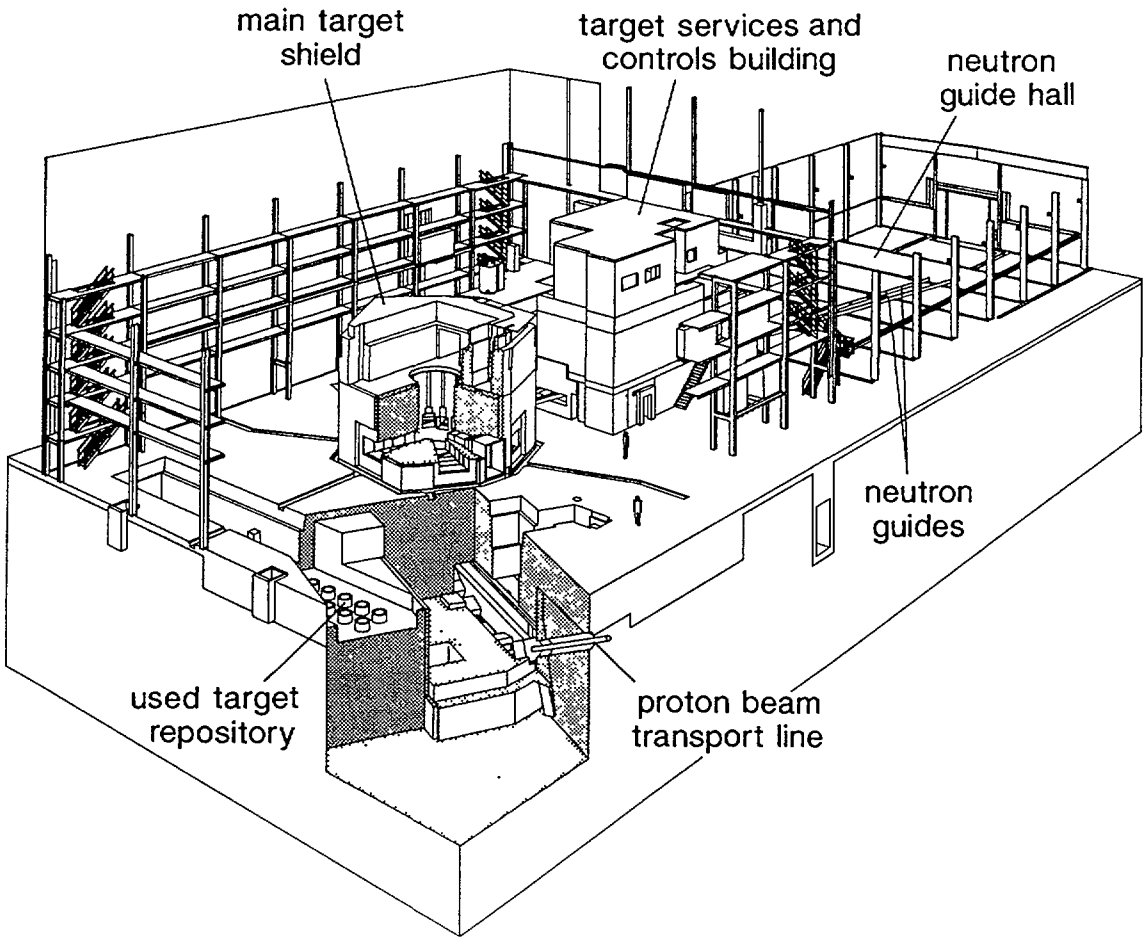

Figure 2: *Partial cut-away view of the SINQ facility located in the neutron target hall. The proton beam line partially shown in the foreground allows injection into the target from underneath. The target shielding is shown in partially cut open to display the space where the atmospheric containment was inserted and the penetrations for the neutron beam ports and cold source insertion. Adjucent to the target shielding and located above the neutron guide shielding cavern is the target services building, completely located inside the target hall. Seven neutron guides, two of which are visible, lead into the neutron guide hall.*

4. Target development programme

Together with the maximum neutron yield that can be obtained, safe and reliable operation of a target at the anticipated beam power level of nearly 1 MW is a prime concern in SINQ. The proton current density will be 25 $\mu A/cm^2$ under normal conditions and can go substantially higher for short times, if the upstream meson target fails or the beam is incidentally steered past it. Inititally we inteded to start operation with a liquid lead-bismuth target which has a number of attractive features. However, in view of the lack of experience world-wide with such targets, we decided on an approach which puts maximum emphasis on operational safety and hence availability of the source [2].

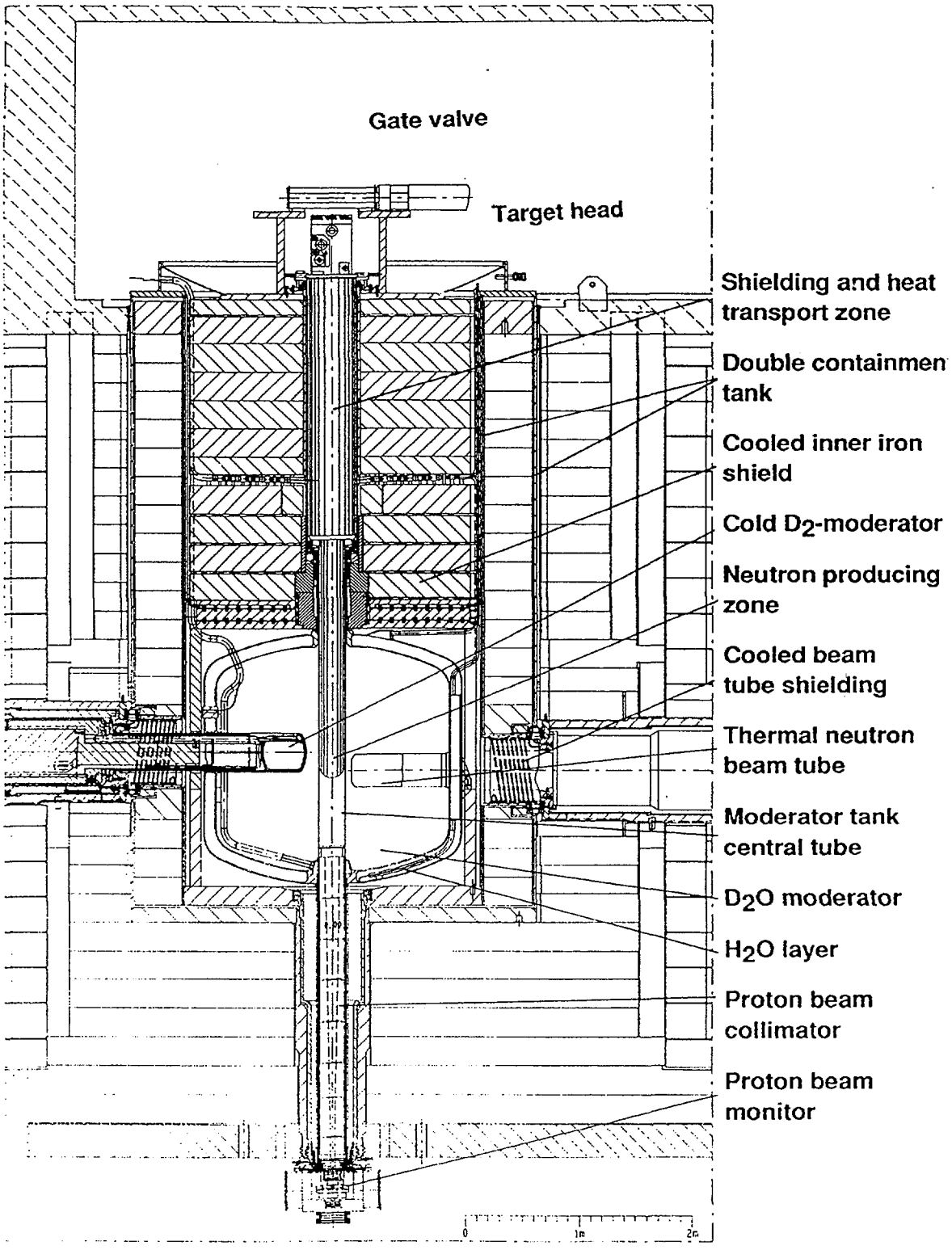

Figure 3: *The inner parts of the SINQ target block, showing the target and its surrounding D_2O moderator tank which are contained within two concentric steel tank structures with controlled atmospheres*

Starting with a relatively simple design of a target made up of zircaloy rods and directly cooled by D_2O, we plan to develop and demonstrate a target safety container which should be able to provide tight enclosure of any radioactivity even if something goes wrong in the target itself. In its present version this container is a double walled aluminium structure with water cooling which will surround the target proper. We will initially use it together with the zircaloy rod target and hope to proceed to a target with higher neutron yield within a year by replacing the zircaloy rods by lead filled zircaloy tubes. Comparative expected flux figures for different target materials and concepts are given in Table 1. So far, thermal cycling tests on the lead-filled zircaloy tubes showed no signs of shape change even after more than ten thousand transitions through the melting point of lead.

Table 1: Calculated flux levels of thermal neutrons for various target concepts for SINQ. The numbers are per mA of proton current on target.

Target System	Maximum thermal ($cm^{-2}s^{-1}$)	Thermal flux at 25 cm ($cm^{-2}s^{-1}$)	Loss factor at 25 cm radius
Pb with weakly absorbing container	2×10^{14}	1.3×10^{14}	1
Pb-Bi with steel-container	0.9×10^{14}	0.85×10^{14}	0.65
W-plates in Al-container	0.8×10^{14}	0.6×10^{14}	0.46
Ta-plates in Al-container	0.55×10^{14}	0.45×10^{14}	0.35
Pb-rods in zircaloy-tubes	1.5×10^{14}	1×10^{14}	0.70 (estimated)
Zircaloy	0.8×10^{14}	0.59×10^{14}	0.45

Engineering work on the mark 1 target is virtually complete and construction of the first model is in progress. Since, according to Table 1, more than a twofold flux gain can be expected from an optimized massive liquid metal target relative to the zircaloy target, development of this concept will remain a focus of future work for SINQ. Fig. 4 shows schematically the two target concepts.

5. Target services and ancilliary equiqment

The decision to go from a liquid metal targt with no cooling water in the proton beam to a solid target with D_2O coolant that will be exposed to the protons had a profound effect on the cooling circuits and other ancilliary equipment. Not only did we have to provide for a delay tank very near the exit of the coolant from the target, but the whole circuitry became

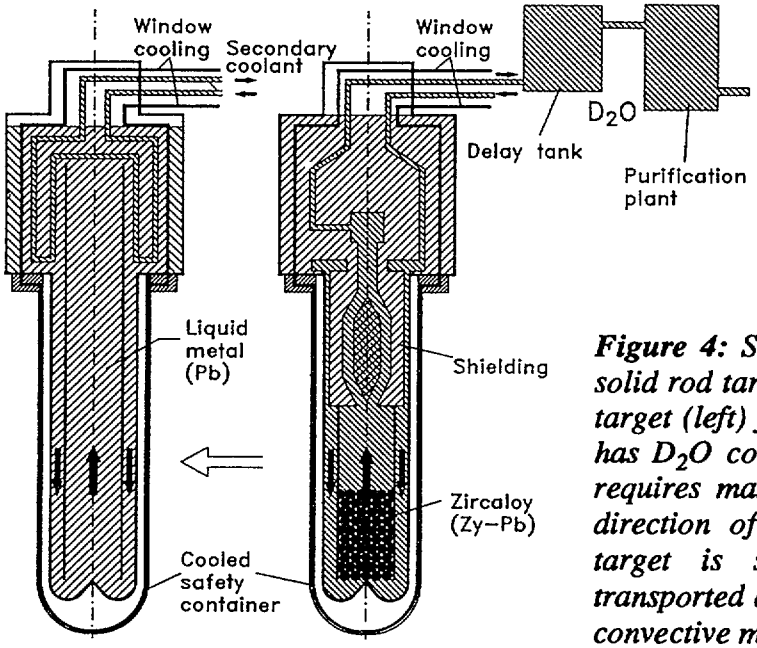

Figure 4: *Schematic representation of the solid rod target (right) and the liquid metal target (left) for SINQ. The solid rod target has D_2O coolant in the proton beam and requires massive shielding in the forward direction of the beam. The liquid metal target is self shielding and heat is transported away from the reaction zone by convective motion of the target material.*

significantly more complex and voluminous. With most of the basic engineering for the cooling circuits now at hand, we are presently establishing a complete 3-dimensional CAD-model of the whole system to ensure optimum placement of pipes and equipment throughout. Sufficient space must be allowed for maintenance and exchange where necessary, so work can be performed at minimum radiation exposure of personel. The requirements to the target transport flask have been specified and a conceptual desgin has been worked out. Detailed design is due to start soon.

Similarly, the concept of the control and safety system has been layed down and individual parts of the system are now being detailed.

All pertinent information is stored in a data base on the PSI central computer and is accessible form everywhere in the laboratory. The data base mangagement system used is ORACLE.

6. Expected neutronic performance

Neutronic performance predictions for SINQ are based to a large extent on model calculations [3], [4], [5]. They were benchmarked against exploratory experimental work done during the preparatory phase of the project. The expected distribution of unperturbed neutron flux in the D_2O moderator is shown in Fig. 5 for the case of the optimum target. For other targets the isoflux contours may not simply scale with the figures given in Table 1 due to a varying degree of neutron absorption in the target and resulting flux depression close to it. For the thermal and higher energy groups the flux is given as a function of the radial distance from the target centre in Fig. 6. Fig. 7 shows the expected energy spectrum at 30 cm from the target centre line. This spectrum ressembles very much that of a modern reactor, but it should be noted that it extends all the way up to 500 MeV, although with an increasingly steep drop.

The high energy component of the neutron spectrum is not only responsible for the massive target shielding but is also a matter of concern in the beam tubes of SINQ. Fig. 8 gives the variation of its calculated intensity in the beam tubes as a function of the angle of the beam tube relative to the direct line of sight on the target. Obviously the 90° viewing angle chosen for the beam tubes of SINQ is of great significance. Nevertheless we plan for rather massive monochromator shielding for the first two instruments in the target hall (see section 8).

7. Experiment infrastructure

In parallel to work on the target block and the target itself, great emphasis is put on those pieces of equipment, which we call the experiment infrastructure for the instruments. This includes in particular

- the cold D_2 moderator
- the neutron guide system
- the beam port inserts
- equipment needed to install and maintain these components

Since these systems will serve a large number of instruments over a long time, we consider it particularly important to provide a state-of-the-art standard, even if this limits the number of instruments we can provide for the start-up phase of the source.

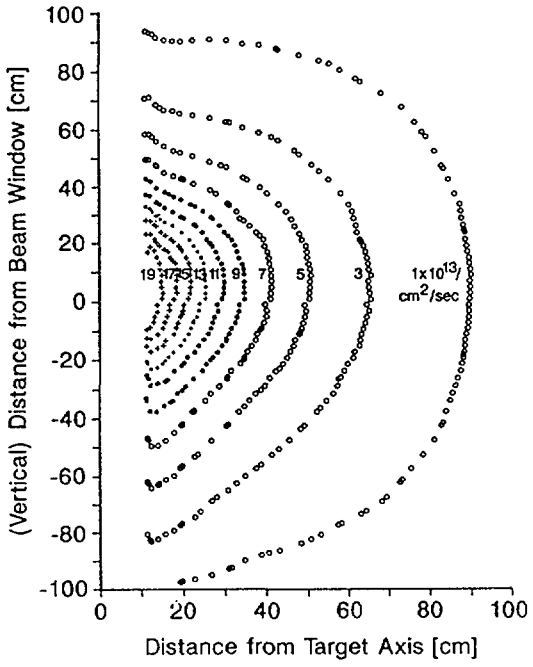

Figure 5: Contours of equal unperturbed thermal neutron flux in the SINQ-moderator tank at 1 mA proton current and for a massive lead target with low absorption container.

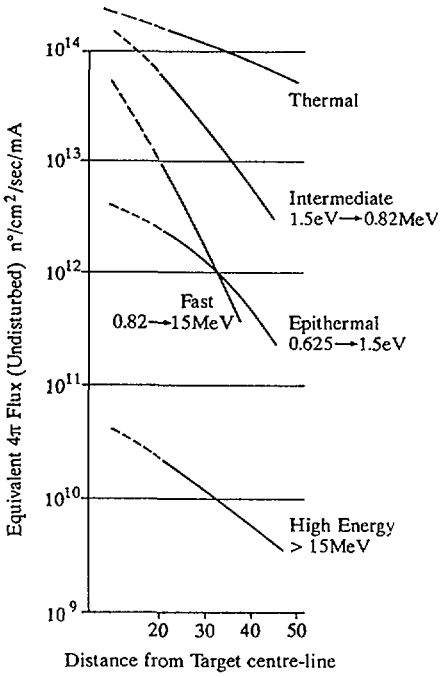

Figure 6: Radial dependence of the neutron flux for different energy groups in the moderator tank of SINQ (same target as in Fig. 5).

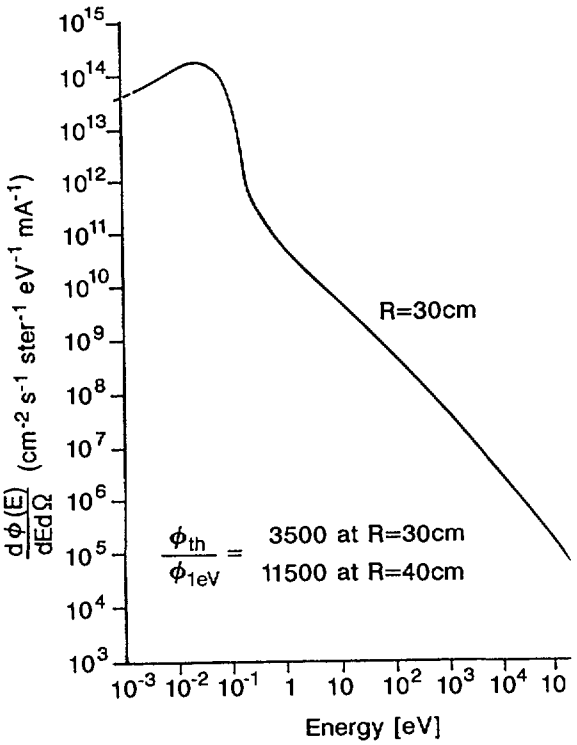

Figure 7: Expected energy spectrum in the SINQ moderator at 30 cm from the centre line of the target.

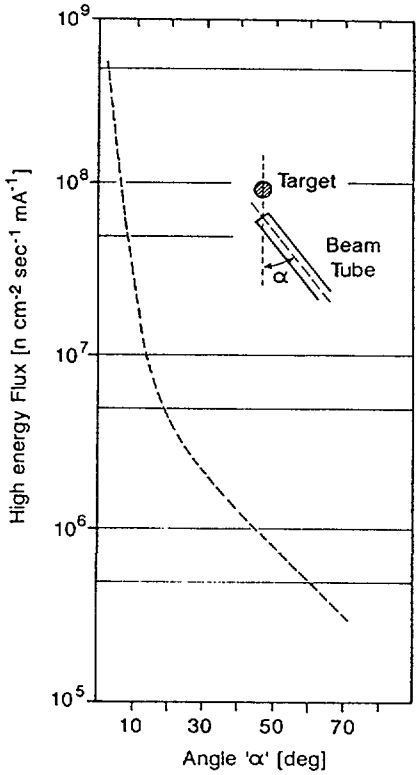

Figure 8: Variation of the high energy neutron component seen at the end of a beam tube with the angle of the beam tube relative to the direct line of sight onto the target.

The cold D_2 moderator system is presently being procured for an off-line test setup. The deuterium-helium heat exchanger is nearing completion, the cryogenic plant is being commissioned and the horizontal leg of the cold moderator system including the moderator vessel and the zircaloy pressure tube are being manufactured. We expect to start the test setup in the second half of this year. The planning of the necessary control system is progressing well.

Table 2: Specifications of neutron guides at SINQ. Guide coating ist with supermirrors unless indicated otherwise (NiC).

Guide designation	Anlge relative to centre line of bundle	Dimension to Width x Height (mm^2)	curved length L_{curv} (m)	Radius of curvature R_{curv} (m)	critical wave length λ* (Å)
1RNR11	-6°	50 x 120	20	1445	2.4
1RNR12	-4.8°	20 x 120	20	3612	1.7 (NiC)
1RNR13	-4.0°	30 x 120	20	2408	1.5
1RNR14	+3.2°	35 x 120	20	2063	1.7
1RNR15	+4°	30 x 120	20	2408	1.5
1RNR16	+6°	50 x 50	20	1445	4.2 (NiC)
1RNR17	+6°	two times 24.5 x 50s	24	1234	

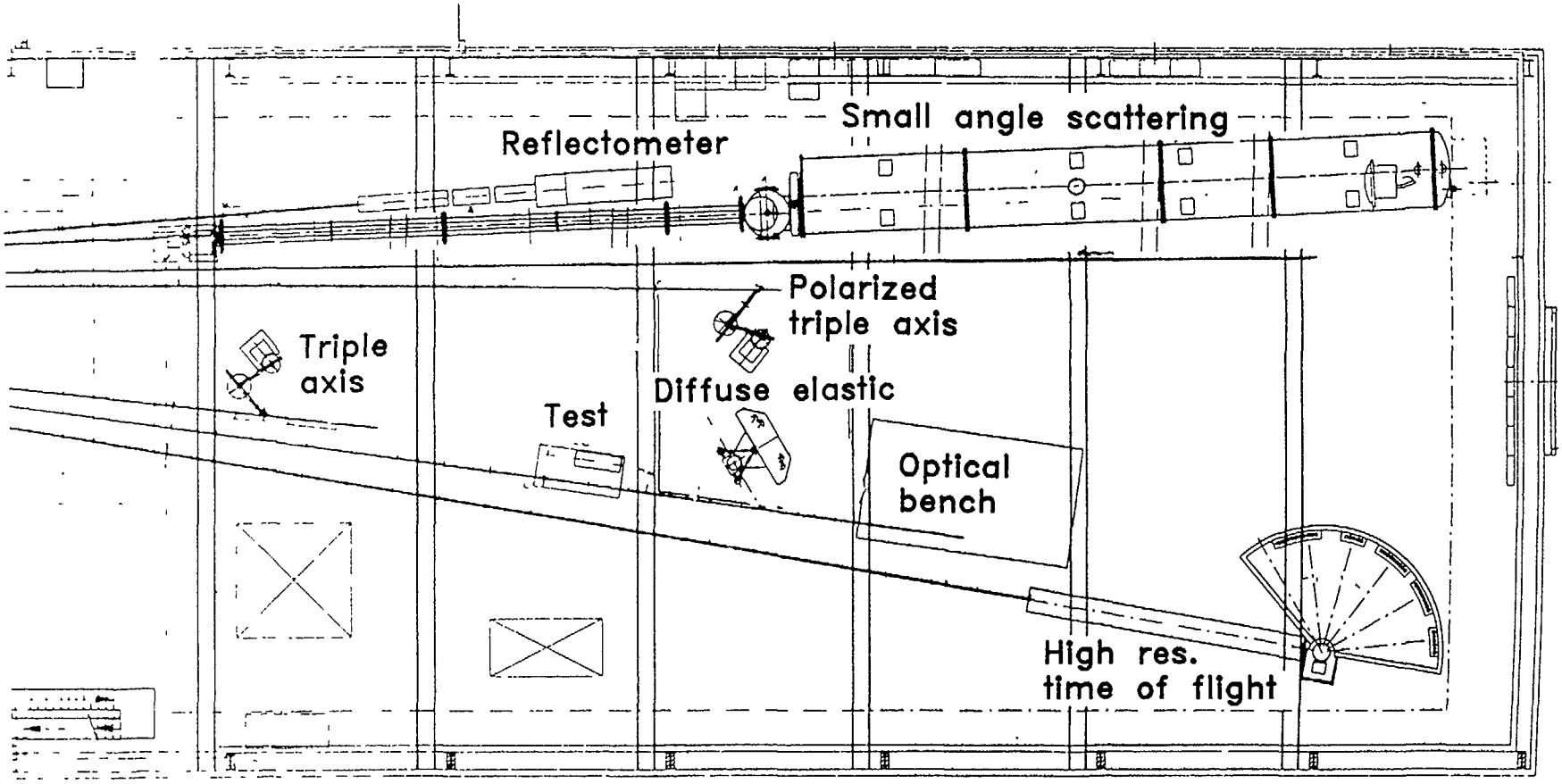

Figure 9: Floor plan of the neutron guide hall showing the arrangement of neutron guides and projected instrument locations.

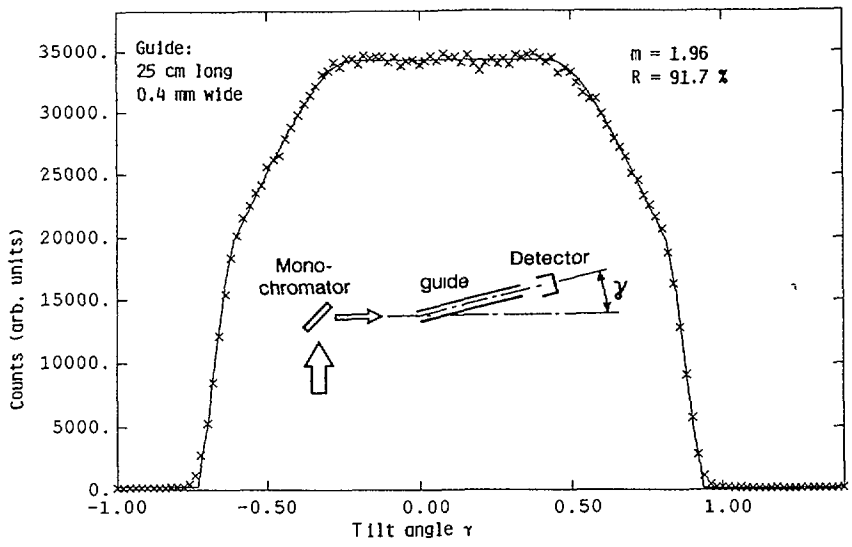

Figure 10: Transmission through a 25 cm long and 0.4 mm wide "microguide" coated with supermirrors designed for m = 2 as a function of tilt angle relative to a well collimated beam of 4 Å neutrons.

One of the six neutron guides viewing the cold D_2-moderator will be divided in its height early on and the two guides of reduced cross section (5 x 5 cm^2) will continue with different curvatures so that actually 7 guides will penetrate the shielding of the neutron guide cavern (Fig. 9). The properties of the guides are listed in Table 2.

While the two guides which will serve instruments with narrow ingoing collimation will be coated with carbon or nitrogen-doped nickel, we intend to coat the other 5 guides with supermirrors with twice the critical angle of natural nickel (m = 2). This will be done at PSI by our own staff. Tests have now routinely resulted in supermirrors with m close to 2 and reflectivities of 92% or more. An example of the transmission measured at the reactor SAPHIR with a narrow gap "mircroguide" as a function of tilt angle relative to the beam is shown in Fig. 10. Series production on polished glass plates will start in early April. Fig. 11 shows the calculated neutron wavelength spectrum for the D_2-moderator for a 6 m long beam tube of 120 x 35 mm^2 cross section together with those for the end of a 50 m long guide of the same cross section coated with natural nickel at 99.5% reflectivity and m = 2 supermirrior at 90% reflectivity. The gain from using the supermirrors is obvious; we expect to exceed the 90% reflectivity on our guides.

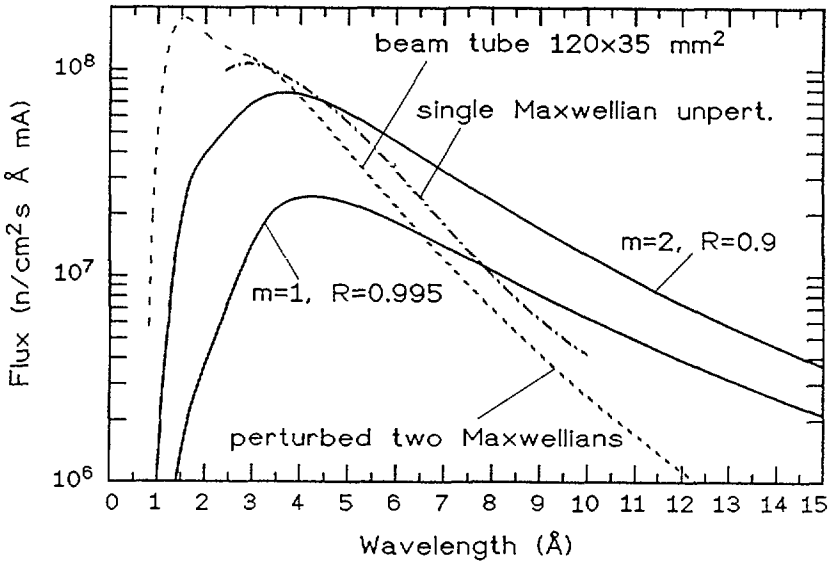

Figure 11: Calculated perturbed wavelength spectrum for the SINQ cold moderator for a beam cross section of 120 x 35 mm^2. The dash-dotted line is the single Maxwellian spectrum for the unperturbed case shown in ref. [1]. The length of the beam tube is 6 m. Also shown are the spectra for a 50 m long guide coated with natural nickel (m = 1, R = 99.5%) and with supermirrors (m = 2, R = 90%).

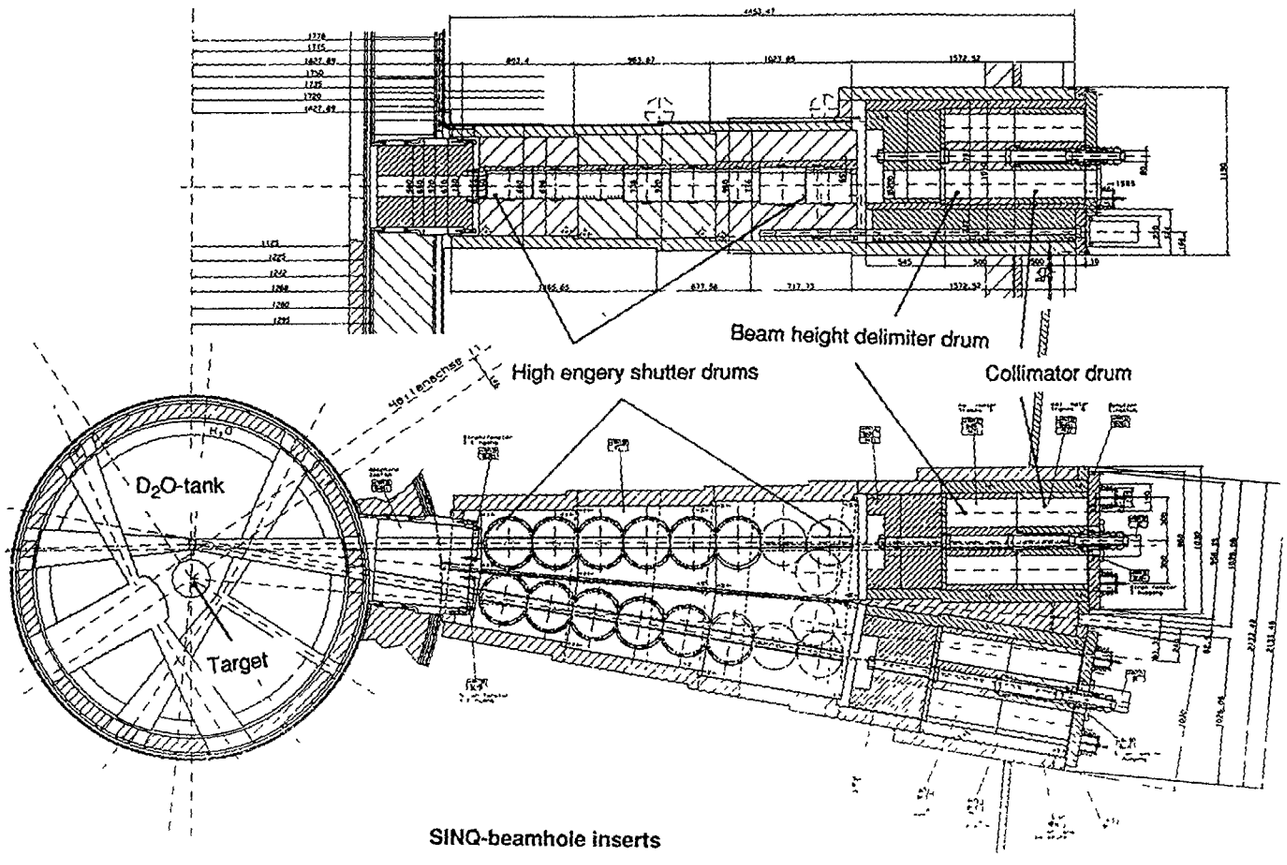

Figure 12: *Conceptual layout of the beam port inserts with a nine drum high energy shutter that can also limit the width of the beam. Two user defined drums with horizontal axes can be rotated indepently. In contrast to the situation shown, the penetrations in these drums will be arranged in such a way, that they only coincide on the beam axis and are azimuthally displaced from one another in the other positions. The first of the two drums is intended to limit the height of the beam and the second one to hold different collimators.*

The beam port inserts in the target block (Fig. 12) will comprise high energy shutters with nine synchronously driven drums with vertical axes and tapered slits that also allow to limit the lateral width of the beam. The beam height can be limited by another drum with horizontal axis that has three different open and one closed positions. A second, similar, drum in series with the first one will serve to hold collimators of different specifications. This provides a very high degree of flexibility in adjusting the beam on the monochromator to experimental needs and to avoid unneccessary activation of the monochromator environment by limiting the cross section of the beam early on. Detailed design work is in progress on these components.

All equipment needed to install and remove the components in the beam ports or cold moderator insertion penetrations remotely has largely been designed and is almost ready for tender.

8. Neutron scattering instruments

Fully equipped, SINQ could provide space for about 20 neutron scattering instruments even if, as presently envisaged, one of the thermal beam ports will be used to install a sample irradiation facility.

Apart from the fact that our available resources would not allow to provide instruments for all possible locations for day one, there are other reasons why we would not want to do this:

- experience has to be gained with the situation on a cw-spallation source, for which there is no real precedent world-wide;
- we feel that it is important to have a basic set of well designed and equipped standard instruments which are fully understood and can be used for science from the beginning, continuing the tradition that exists at our institute;
- we wish to reserve space for advanced developments which currently exist only as concepts or proposals, but which take more effort to implement than we can presently provide while the whole source is under construction.

According to current planning, there will be a minimum of five instruments ready when the source will be commissioned, with some hope that three more could become operational shortly afterwards with external support. In the following we give a brief description of the five day-one instruments, which are summarized in Table 3.

A high resolution powder diffractometer (HRPD) similar in design to the DMC presently in use at SAPHIR will be located at one of the thermal beam ports. The position sensitive detector covering an angular range of 160° can be rotated around the sample. A 20 cm high vertically focussing Ge-monochromator will illuminate a cross section of 2 x 5 cm^2 at the sample position. There will be two fixed monochromator angles at $2\,\theta_M = 90°$ and 120°.

HRPD will become a versatile, powerful tool to investigate both structure and magnetic ordering phenomena in a large class of materials. The applications range from solid state physics, chemistry, crystallography, materials science to biology and include both fundamental and applied research of interest also to industry (e.g. to refine crystal structures of low symmetry for unit cell volumes up to about 2000 $Å^3$, as a function of temperature in the range of 7 mK to about 1400 K with up to approximately 250 parmeters). Also zero matrix pressure cells, superconducting magnets etc. should become available for similar powder investigations

Table 3: First generation instruments planned for SINQ under PSI-responsibility

Instrument	High resolution powder diffractometer	Four circle diffractometer	High resolution triple axis	Polarized neutrons triple axis	Small angle scattering instruments	High resoluting Time-of-flight spectrometer
location	thermal neutron beam port	thermal neutron beam port	cold neutron guide (s.m.)	cold neutron guide (s.m.)	cold neutron guide	cold neutron guide
monochromator	Ge (hkl)	PG (002), Cu(220)	PG (022), Be (220)	Heusler (111) PG (002)	mech. velocity selector	chopper 3-12 Å
$2\,\theta_{mon}$	20 + 90 deg	15 - 90 deg	30-145 deg	30-145 deg	--	--
detectors	position sensitive detector 10 - 170°	2 dim, He-3 210 x 185 mm^2 (microstrip)	He-3	He-3	2 dim, 128 x 128 cells 960 x 960 mm^2	He-3 detector array
resolution/range	$\Delta d/d = 4 \cdot 10^{-4}$		≥ 7 μeV	≥ 7 μeV	$3 \cdot 10^{-3}$ $nm^{-1} \leq Q \leq 10$ nm^{-1}	0.005 + 0.5 meV
special features/remarks	vertically focussing monochromator; GdO mylar sollers in front of detectors	dilution cryostat; closed cycle cryostat			double crystal monochromator (option) polarization (option) specimen changer cryostat; furnace; electromagnet	counter rotating disc choppers 100 + 400 Hz; detector angular range 10 -135°; converging super mirror

in the future. Depending on the particular problem, the amount and quality of the sample available, resolution and intensity may be optimised.

The second beam on the same beam port will be occupied by a four circle diffractometer equipped with three 2-dimensional position sensitive detectors which are mounted on individual columns and can be moved around the sample on a common support plate. Since we intend to provide sample environment equipment for a rather wide range of conditions, including an ADP closed cycle helium refrigerator for 20 - 450 K and a dilution cryostat for ~200 mK, tilting options for the sample may sometimes be limited. For this reason it will be possible to move the detectors out of the horizontal plane individually. 20 x 25 cm^2 microstrip detectors with a resolution of 1 to 1.5 mm horizontally and 3 mm vertically will allow to obtain detailed information on the intensity distribution of individual reflections if desired. Variation of the sample-detector distance will be possible by manual adjustment. In addition to single crystal diffraction with the option of investigating the 3-dimensional intensity distribution of individual reflections by the 2-dimensional detector plus rotation of the sample, the instrument can of course also be used for texture studies with great benefits.

Fig. 13 shows the layout of the monochromator shielding and Fig. 14 gives a schematic representation of the four circle diffractometer.

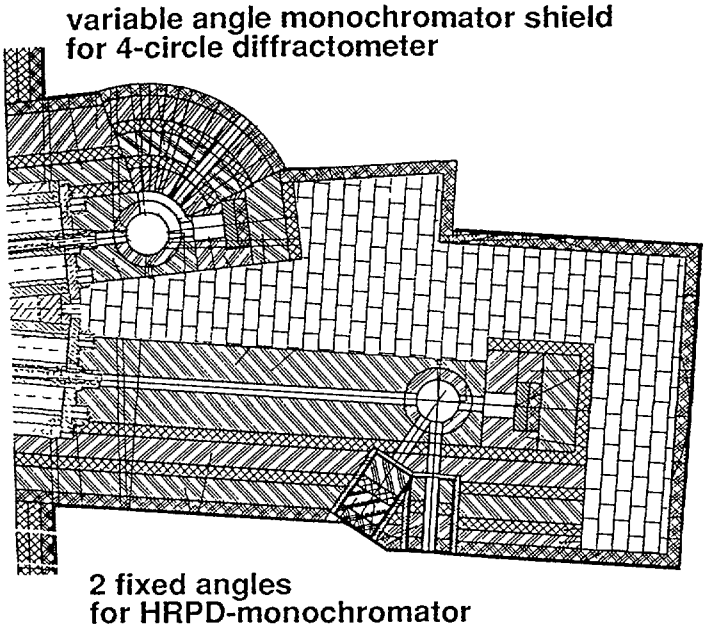

Figure 13: *Design of the monochromator shielding at the thermal beam port occupied by a high resolution powder diffratometer and a four circle diffractometer. To shield also against high energy neutrons, a sandwich-type shielding of heavy and moderating material was chosen.*

Two triple axis spectrometers of similar design are planned for installation on the neutron guides. One will be a conventional high resolution instrument mainly for small energy transfers and the other one will be equipped with a Heusler crystal for polarized neutrons.

A schematic representation of the design of the triple axis spectrometer is shown in Fig. 15 Great care is being taken to keep the background in the detectors as low as possible by opening up the shielding of the monochromator and analyser as the tables move only in such places where it is faced by other shielding parts.

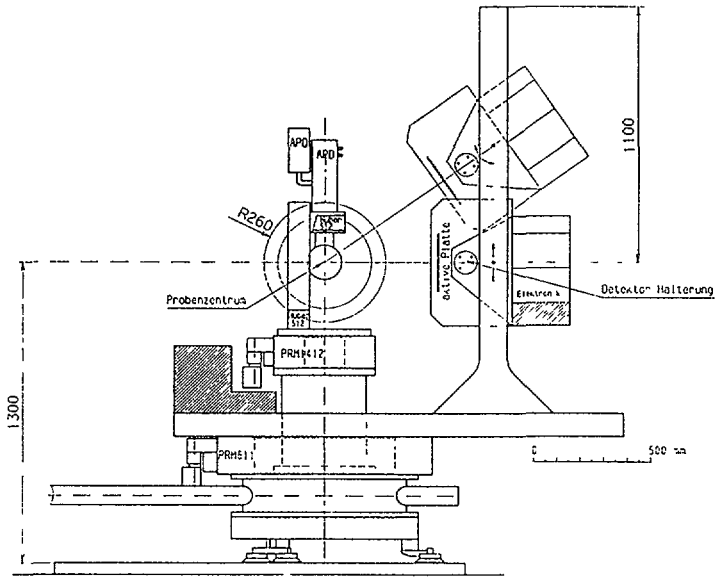

Figure 14: *Schematic elevation of the four circle diffractometer showing the detector in two differently inclined positions.*

The non-polarized triple axis spectrometer will be used mainly for the investigation of structural phase transitions and high resolution spectroscopy. The super mirror coating of the guide will alow to use incoming energies between approximately 2 to 3 meV and to reach a momentum transfer Q up to about 5 $Å^{-1}$.

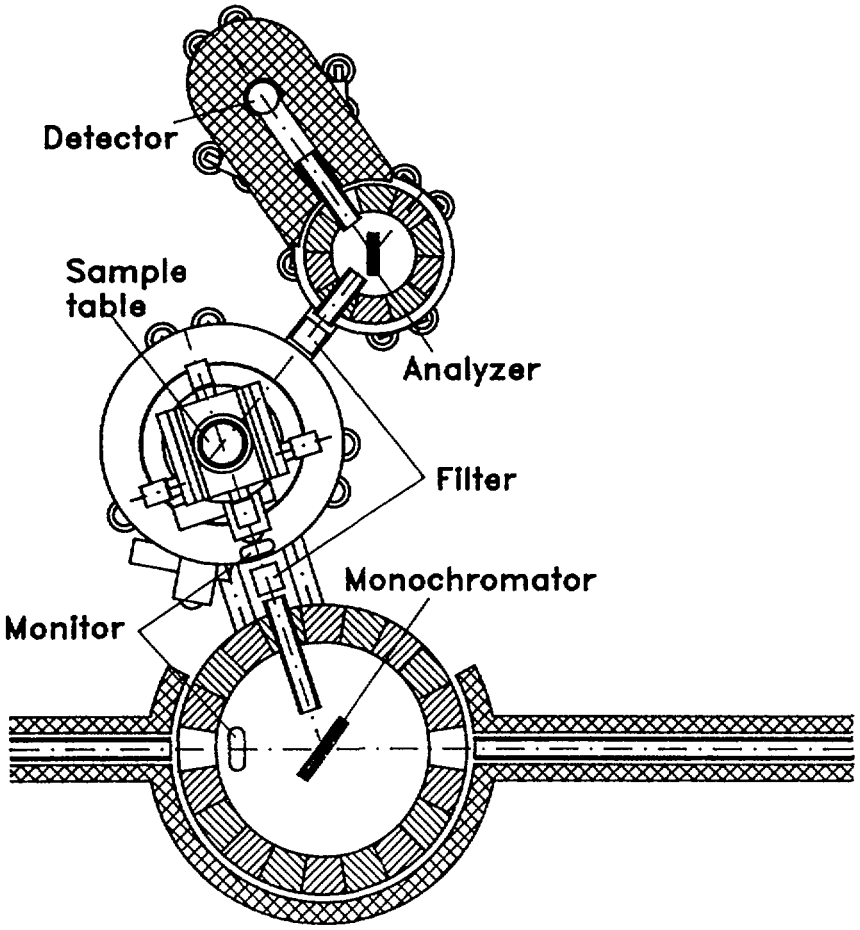

Figure 15: *Conceptual layout of the triple axis spectrometers at the SINQ neutron guides.*

While principally of similar design, spin flippers and guide fields along the neutron flight paths will make the TAS for polarized neutrons somewhat more complex. Apart from the obvious possibilities to use also this machine in a non-polarized fashion, curved Heusler crystals or polarizing super mirrors will be available as monochromators or analyzers, depending on the neutron energies used.

Standard components will be used to build the sample table for all four of the above instruments. They will be movable on air cushions and have compatible supports for standardized sample envrionment equipment. The clearance from the sample table to the beam centre line has been set to 400 mm. The spectrometer arms are designed for 0.01° positioning precision. The precision of angular positions of the crystals has been specified as 0.005°. For all four spectrometer the standard components such as air cushion tables and turn tables have been decided upon and are being purchased. The position sensitive detector for the HRPD has been ordered and individual parts for all spectrometers are being designed.

The fifth instrument planned for early operation is a small angle neutron scattering instrument with a maximum sample to detector distance of 20 m and a neutron guide-collimator

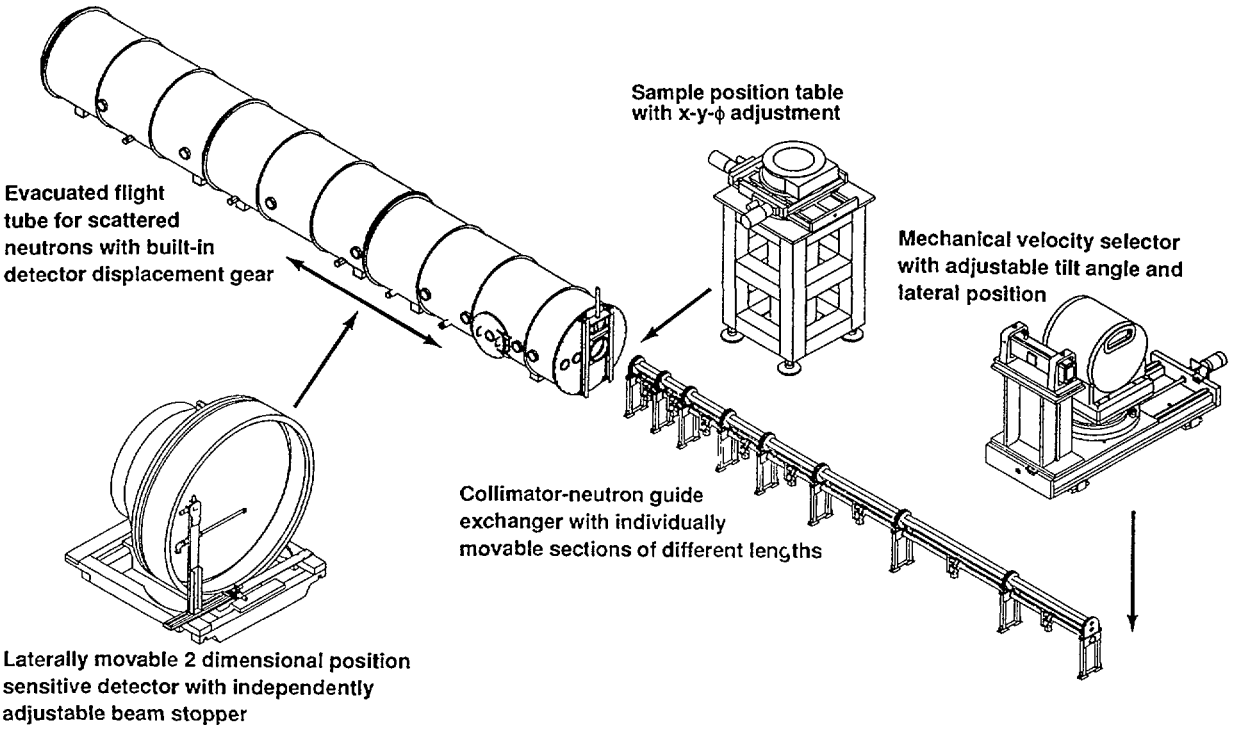

Figure 16: Main components of the SINQ-SANS instrument.

interchanger that allows to match the collimation in front of the sample to the resolution at the detector. It will be equipped with a mechanical velocity selector but could alternatively also use a double crystal monochromator by taking advantage of the fact that two vertically displaced primary beam paths are possible in the neutron guide-collimator exchanger. This might be of praticular interest, if polarized neutrons are desired. The 124 x 124 element 2-dimensional position sensitive detector can also be moved sideways inside the large vacuum vessel to increase the accessible Q-range. The beam stop in front of the detector can be adjusted independently.

The main components of the SANS-instrument are shown in Fig. 16. With the exception of the electronics for the data acquisition system and instrument control, all of the main components have either been ordered or are out for tender at present.

In addition to these five instruments which are designed and looked after by PSI staff, we are preparing to accept at least three more spectrometers for which interest has been expressed by external groups. These include a neutron-optical bench with several sets of pairs of perfect crystals for neutron interferometry, very high resolution small angle scattering and related work, a back scattering spectrometer and a high resolution time-of-flight spectrometer. If these projects ma ture, SINQ can make available a full suite of high perfomance instruments to its customers and users, while more space is available for new developments.

9. Concluding Remarks

With the construction and operation of SINQ the Paul Scherrer Institut intends to further improve and enlarge its comprehensive set of tools for condensed matter research which also includes muon and pion beams and possibly even an intense positron beam facility in the near future, if an ongoing development turnes out successful. This is in accordance with the laboratory's shifting emphasis towards condensed matter and materials research. More long term plans aim at providing also a synchrotron light source.

During the planning, design and construction of our facilities we have received help and advice from many places and in many forms. We will be glad to pass our own konwledge and experience on to others as the need arises. PSI's facilities are open to outside users under various schemes of collaboration which can be agreed upon individually.

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NEUTRON SCATTERING RESEARCH WITH LOW TO MEDIUM FLUX REACTORS

J.J. RHYNE
Research Reactor Center,
University of Missouri-Columbia,
Columbia, Missouri,
United States of America

Abstract

This paper discusses neutron scattering instrumentation that is appropriate for research at low to medium flux reactors. A summary is given of the feasibility and potential for various categories of scattering instruments at reactors of different thermal flux levels. Included are suggestions for powder diffraction, small angle scattering, and triple axis spectrometers. A brief discussion is included on methods improving signal to noise ratios, particularly in light water moderated facilities.

1. INTRODUCTION

This paper presents suggestions and guidelines for types of instruments and associated research programs that are appropriate for reactors in the low to medium flux categories (i.e., 1 x 10^{13} < flux < 5 x 10^{14} n/sec/cm^2). The research and instrumentation examples given are admittedly highly subjective and certainly not meant to be all inclusive.

Much of the following discussion is based on experience gained by the staff at the University of Missouri Research Reactor Center (MURR) and from the author's own experience at this facility and at the NBS Reactor at the National Institute of Standards and Technology in Gaithersburg, Maryland.

The University of Missouri Research Reactor is a 10 MW research reactor that is the highest flux reactor at a university in the United States. It is a light water moderated and cooled, beryllium reflected, pool-type reactor with an annulus flux-trap pressure vessel. Eight MTR-type fuel elements form a 28 cm diameter x 61 cm high core and produce a flux of 1.2 x 10^{14} n/sec/cm^2 at the end of the six radial beam tubes used for scattering experiments. The peak flux in the flux trap is 6 x 10^{14} n/sec/cm^2. Three of the beam tubes are 15 cm in diameter and three are 10 cm diameter. Extensive use is made of liquid-nitrogen cooled and also room temperature silicon and sapphire filtering in the beams before the instrument monochromators. This is an essential requirement with light water reactors of greater than about 1 MW power level with radial beam tubes, in order to reduce the fast neutron background and to permit monochromator shielding of compact design.

The present neutron scattering instrument complement at MURR consists of a triple axis spectrometer, two double axis single crystal units, two powder diffractometers, and two neutron interferometers. A 15 m small angle scattering spectrometer is under development as are a new triple axis spectrometer, a reflectometer, and a dedicated residual stress facility.

A summary of MURR and other instruments used for powder diffraction, SANS, triple axis spectrometry, and neutron reflectometry will be given in the following sections.

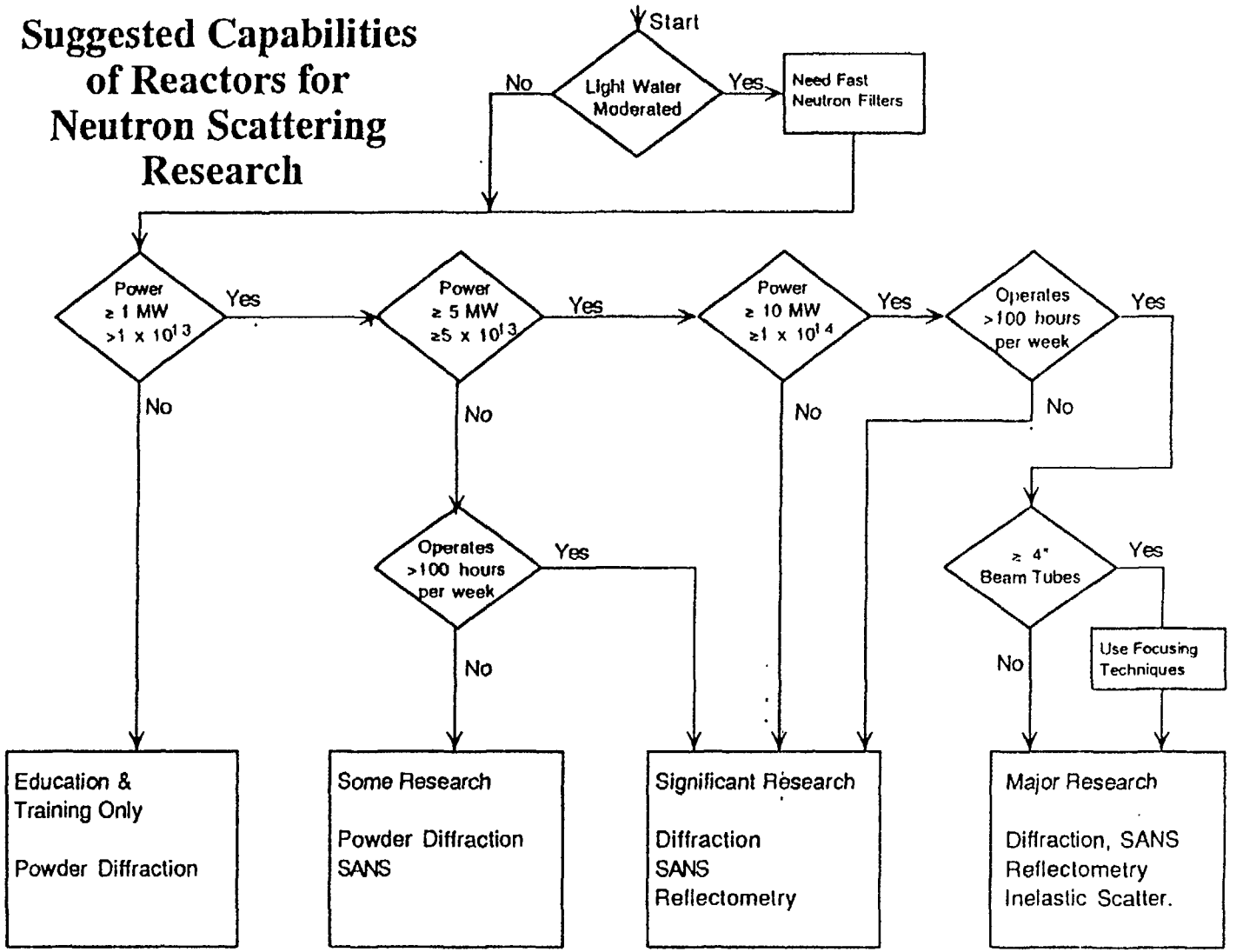


Figure 1. Diagram for suggested types of research and associated instrumentation for reactors with the fluxes given at the top. The flux numbers are considered more critical than the power figures presented. The flux/power relationship is scaled based on several H_2O and D_2O reactors in the U.S. and is characteristic of a compact core design.

2. GENERAL CONSIDERATIONS

In order to have truly competitive and useful research instrumentation at low and medium flux facilities, great care must be taken in the selection and design ofscattering instruments. The first advice is to carefully analyze the real and practical capabilities of the reactor source and use this analysis in the selection of appropriate research areas and the required instrument complement. The natural tendency is to build too sophisticated instruments or to try too complex types of experiments that are both beyond the capabilities of the reactor source. This approach is often wasteful and often doomed to failure.

Figure 1 gives a suggested (and highly subjective!!) guide to the selection of research and instrumentation, based on the available source flux for the beam tubes. The table can be summarized briefly as follows: (1) For reactors with fluxes less than 1 x 10^{13} n/sec/cm^2, particularly if they are light water moderated, the uses should likely be limited primarily to education and training rather than competitive research. Limited powder diffraction is certainly feasible with this flux level. (2) Reactors with fluxes above level (1) but less than about 5 x 10^{13} n/sec/cm^2 can produce research with powder diffraction and SANS instruments, and, if they operate more than 100 hours/week, could be considered to have significant research capabilities. A neutron reflectometer is also a suggested addition to the stable of instruments. (3) For fluxes higher than 1 x 10^{14} n/sec/cm^2, significant first-rate research is definitely feasible, and, if the source operates more than 100 hours/week, one can consider such a reactor a major research facility capable of performing diffraction plus inelastic scattering research. It is particularly important if beam tubes of greater than 10 cm diameter are available to use beam focusing monochromator techniques, especially on inelastic scattering spectrometers. *[It should be noted that, while the examples given in Figure 1 can be considered representative, there are many highly successful reactor research programs operating well outside the envelope of the above guidelines. Clever instrument optimization and good shielding concepts, plus patience with long counting times, can make up a lot for lower than optimal reactor fluxes.]*

It is often the case that lower flux reactor facilities must also contend with highly constrained budgets for instrument design and construction. This is most unfortunate and probably means that building a small number of really first-class machines is better than doing a mediocre job on a larger group of instruments. In general the advice could be that "if available funds are insufficient to build a state-of-the-art spectrometer -- wait and argue strongly for the additional money."

3. BEAM AND INSTRUMENT OPTIMIZATION

The necessity to optimize all instrument and beam parameters at every step to get the most out of the available reactor source flux is a critical guideline for instrument design at lower flux facilities. The important end result is the highest possible signal to noise ratio. Minimizing backgrounds often carries the highest payoff in this regard. The use of fast neutron filtering, appropriate high-density shielding materials, and adequate care to fully shield all neighboring instruments in the reactor hall is essential. Pressing of monochromator crystals to open up the mosaic to the range of 20 minutes and the use of focusing monochromators to take full advantage of the vertical divergence of the beams can increase intensity appreciably. As discussed below, for the case of powder diffraction, multiple discrete detector systems or position sensitive detectors can also raise data rates by significant factors.

Table 1*. FILTER MATERIALS TO REDUCE FAST NEUTRON COMPONENTS in THERMAL BEAMS

Note: $\sigma = \sigma_{fast}/(\sigma_{absorption} + \sigma_{incoherent})$ is the relative cross section for fast neutrons.

	Advantages	Problems
Si	Good crystals available	Needs cooling, poor γ reject.
SiO_2	Good crystals, modest Θ_D	Average σ
Bi	Excellent σ	Poor crystal quality, low Θ_D
Al_2O_3	High Θ_D	Oxygen window - 2.35 MeV
MgO	High Θ_D	Large crystals unavailable, oxygen window
MgF_2	Good σ	Poor crystal quality, low Θ_D
Bi-silicates $Bi_4Si_3O_{12}$ $Bi_{12}SiO_{20}$	Excellent σ, high dens.	Poor crystal quality, low Θ_D
Alexandrite $BeAl_2O_4$	Good crystal quality Modest σ	Small crystals only
Cubic Zirconia	Good crystals, high Θ_D high density	O-window, absorption (need Ca stabilization)

**Compiled by W. Yelon (MURR)*

Incident beam filtering with liquid nitrogen cooled silicon, sapphire (may be operated at room temperature), or alternate materials, preferably before the beam exits the biological shield, is very important as is a shielding design that minimizes self-generation of gamma rays. If only longer wavelengths are needed (e.g., > 3.96 Å) for SANS, a combination of Be and Bi is a very effective filter. Table 1 gives a list of candidate materials for fast neutron filtering prepared by W. Yelon[1]. The general criteria for a good filter are a material with (1) a relative cross section for fast neutrons [$\sigma = \sigma_{fast}/(\sigma_{absorption} + \sigma_{incoherent})$] of order 20-50, (2) good crystal perfection to reduce losses from Bragg scattering, (3) a high θ_{Debye} to reduce phonon scattering, (4) reasonably priced and available single crystals, and (5) a high density to provide good γ-ray rejection. As an alternative or in addition to filtering, beam offsets using a double monochromator design are also very effective at reducing background, but may extract a significant penalty also in primary beam intensity depending on the monochromator crystals.

Finally neutron guides are an optimal approach to better signal to noise ratios as they allow instruments to be located well away from the reactor face. If supermirror coatings are employed, guides will function extremely well even in the traditional thermal (not cold) neutron wavelength range.

4. DESIGN OF SPECIFIC NEUTRON SCATTERING INSTRUMENTS

The following are some suggested design considerations for neutron instruments targeted for use on lower flux reactors. These guidelines are very cursory in nature and details must be obtained from review articles or via communication with laboratories having completed similar instruments.

4.1 Powder Diffraction

The powder diffractometer frequently becomes the workhorse instrument at many reactors and often is the instrument (along with SANS) in greatest demand due to its applicability to a variety of problems in fields ranging from materials science, to biology, to complex chemistry. Powder diffraction research also necessitates the use of a modern Rietveld analysis computer code, generally capable of handling both multiple material phases and magnetic scattering, such as GSAS (Generalized Structure Analysis System) written by the Los Alamos National Laboratory scattering group.

For powder instruments, to optimize resolution, the monochromator take-off angle should be designed to be large (preferably $\geq 90°$) so that the resolution minimum falls at large scattering angles where the density of reflections is highest. A wavelength of approximately 1.5Å is often selected, and many instruments incorporate variable wavelength or multiple monochromators. For cutting edge research, Soller slit collimations should be of 10' or less throughout (before and after the monochromator and before the detector).

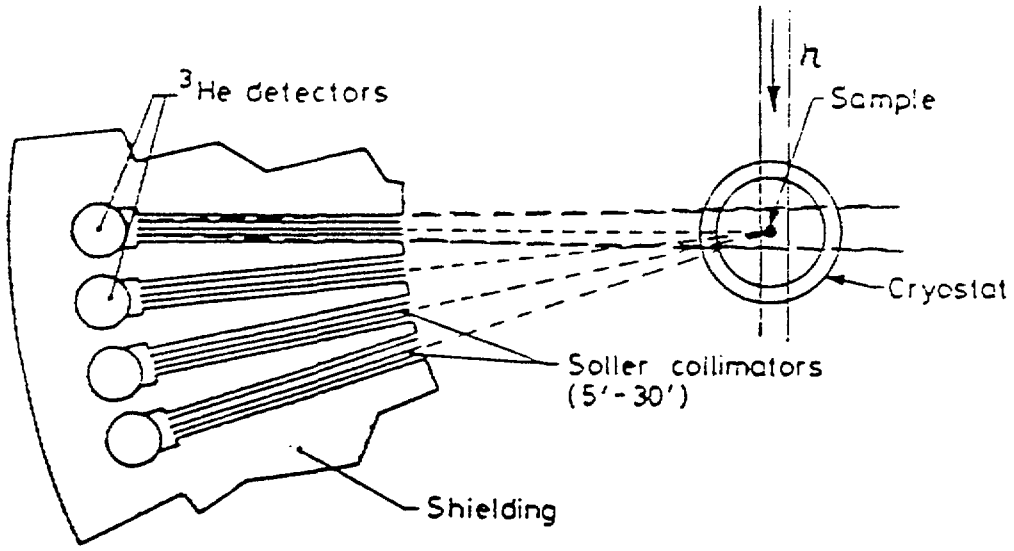
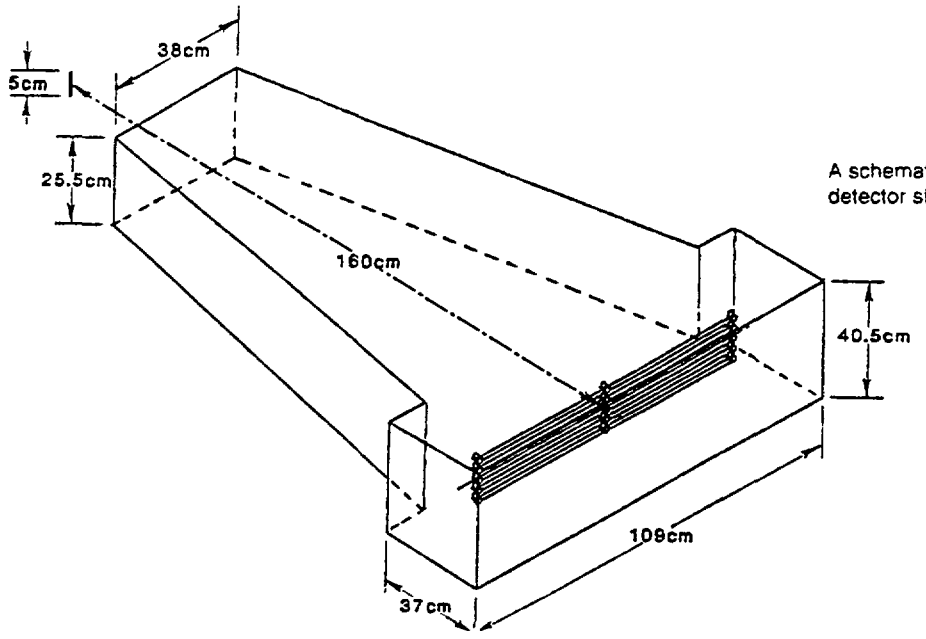


Figure 2. Schematic design for a powder diffractometer using multiple discrete detector tubes.

There are a variety of detector designs in use at various facilities. A single detector tube design is most basic, but today can really only be considered adequate for educational purposes. Multiple detector tube designs incorporating 5, 32, 64 or more discrete detectors, each with its own Soller collimator, allow simultaneous collection of data from multiple portions of reciprocal space. A diagram of such a



A schematic representation of the detector shield assembly

Figure 3. Schematic design for a powder diffractometer using a stacked array of linear position sensitive detectors. This machine is in operation at the University of Missouri Research Reactor Center.

multi-detector is shown in Figure 1. Rotating the entire assembly under computer control through an angle slightly greater than the detector angular separation provides a seamless data set for analysis. For arrays with a smaller number of detectors (e.g., 5 tubes placed 20° apart), larger angular movements may be necessary to adequately cover the full scattering range. The multiple detector instrument is a very powerful research tool if combined with a properly chosen monochromator, a high $2\theta_{mono}$ and very good angular Soller slit collimation (10' or tighter). A complication is in the calibration of the multiple detectors, which generally all have discrete zero corrections and detector counting efficiencies. Calibrations must be periodically checked to ensure that discriminator or electronic gain drifts are not a problem that can affect backgrounds.

Position-sensitive detector arrays have provided a major advance in powder diffraction especially in the speed of data acquisition. The most sophisticated is the "banana" detector in which the continuous detector element is curved at constant radius about the sample. This detection scheme can only be considered by facilities with very opulent instrumentation budgets. A much more modest approach (shown in Figure 2) is the use of commercial linear position-sensitive detector tubes, either singly or in a multiple vertically stacked array, to provide continuous data accumulation over a sector of the circle about the sample.

Multiple segments may be used or the array rotated to cover the full range of reciprocal space. The correction for the detector being a sector instead of a true arc can be made in the data acquisition software. Such an instrument, incorporating five stacked linear detectors and using electronics designed by R. Berliner of MURR, provides excellent resolution and stability, and has been in continuous use for several years. Increasing the number and height of the detector stack can further increase data collection rates, but a Debye Sherrer cone correction must be applied via software. A minor disadvantage to this type of detector is that resolution is dependent on sample size (and detector to sample distance) and an oscillating vane collimator is needed to suppress spurious incoherent backgrounds from cryostats, furnace shields, etc.

The conventional monochromator and Soller slit configuration can be replaced with a perfect crystal two-dimensionally focusing monochromator as developed by Popovici et al.[2] This remarkable and simple breakthrough uses a hemispherically bent perfect crystal (e.g., Si) to provide a significant gain in resolution over conventional mosaic monochromators. Peak width is typically reduced by a factor approaching three at high scattering angles with a simultaneous gain of three in peak intensity. The gains can be made largest at the high scattering angles where good angular resolution is most critical.

4.2 Small Angle Scattering Spectrometers

Sharing the limelight with powder diffraction, small angle neutron scattering (SANS) is a technique in great demand for research in a broad cross section of disciplines from materials science, to polymer chemistry, to biology, and magnetism. SANS bridges the real space dimensional gap between wide angle diffraction (interatomic spacings ≈ 5-10Å) and conventional microscopies in the micron size range. Modern SANS spectrometers typically cover a range or wave vector transfer $0.0005\ Å^{-1} < Q < 0.5\ Å^{-1}$ and find applications for examining defect properties, large molecule structures, local magnetic distributions, phase transitions, and critical and subcritical scattering in disordered systems among others. As shown in Figure 4, a conventional SANS machine includes a pre-filter of Bi-Be to screen out neutrons with wavelengths less than 3.96 Å and unwanted γ's. A wavelength appropriate for the resolution and other parameters desired is selected by a broad band ($\Delta\lambda/\lambda \approx$ 10-20%) variable wavelength monochromating device such as the helical velocity selector shown. Neutrons diffracted at small angles from the sample are detected on an area detector (continuous wire, discrete wire, or micro-strip) providing a two-dimensional representation of the scattered neutrons.

SANS instruments are among the more expensive machines to build, largely due to the size of the shielded evacuated flight paths and the cost of the area detector and velocity selector (or crystal monochromating devices.) Horizontal space from the reactor face to the outside wall of the confinement building is also a problem in many facilities, unless a penetration is available. Useful spectrometers must be at least 8-10 m in length.

An alternative is to construct a vertical flight path in which the beam from the reactor is deflected upward 90° by a crystal monochromator (e.g., pyrolytic graphite) and then the detector and flight path are located on a tower. The vertical design has some severe drawbacks, however, among them (1) the difficulty of achieving a sufficiently large $\Delta\lambda/\lambda$ from the monochromator, (2) lack of variable wavelength, and (3) the difficulties of accommodating cryostats, magnets, and

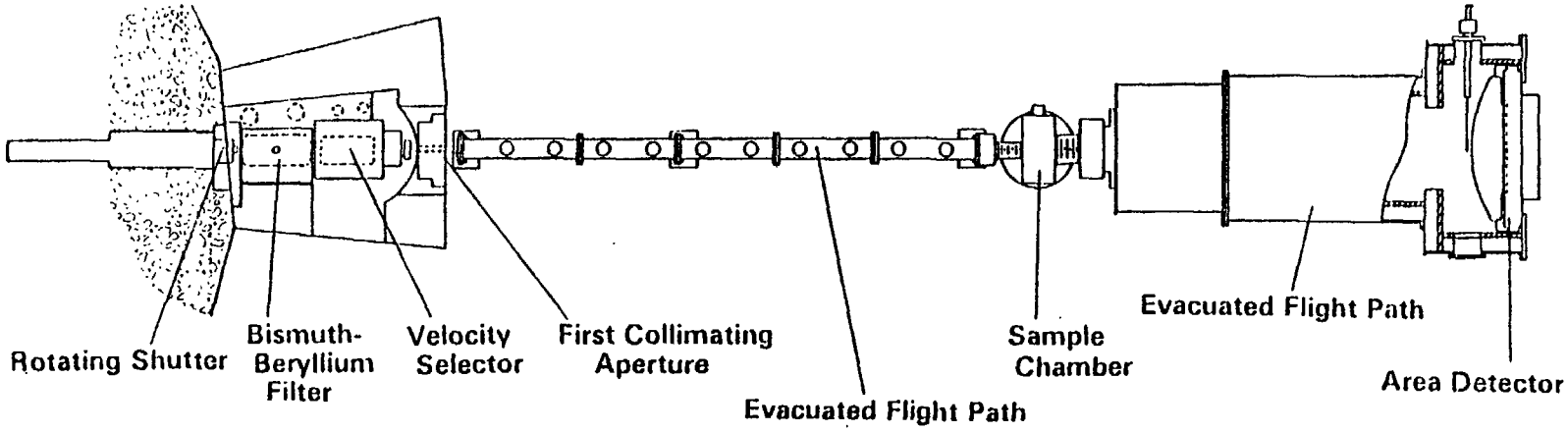


Figure 4. Drawing of a typical SANS machine using an helical velocity selector as beam monochromator and a 64x64 cm area detector.

other sample environmental equipment. A vertical SANS facility does offer great advantages in the study of liquids, however. For both horizontal and vertical configurations, it is necessary to provide an evacuated chamber for the area detector that allows a variable sample to detector distance to control (along with the variable wavelength) the q resolution (minimum q) and to vary the total range of q accessible on the detector face.

4.3 Triple Axis Inelastic Scattering Spectrometers

Triple axis spectrometers are among the most demanding of precise attention to detail and careful design of any of the instruments discussed here. As stated above, they also require fluxes in or above the 1×10^{14} range to be scientifically competitive. Elaborate care must be exercised in the design of the shielding to reduce the intrinsic background and also the thermal neutron and γ background generated in the shielding itself from fast neutrons. Well-designed instruments can achieve background levels (beam open without sample) of a fraction of 1 count per minute. Building both monochromator and analyzer assemblies for variable energy transfer also significantly increases the cost of the instrument, but this feature is highly desirable for appropriate flexibility in selecting resolution conditions. In addition to filtering the white beam against fast neutron contamination, filters (usually again of costly pyrolytic graphite) are needed in the fixed energy (monochromator or analyzer) scattered beam to remove $\lambda/2$ and higher order contaminations. In general, the decision to build a triple axis spectrometer at a low to medium flux reactor should be made only after careful thought and planning and examination of such instruments at comparable reactor sources.

4.4 Neutron Reflectometers

Neutron reflectometers are among the newest instruments at most reactor facilities and have recently enjoyed wide success for problems in magnetism, polymeric films, and biology. Essentially, the reflectometer is a two-axis diffractometer in which the monochromatic beam is narrowed by slits to typically much less than 1 mm. The outgoing beam, scattered at grazing incidence, is then detected by a well-shielded conventional detector. Because of the small scattering angles involved, elaborate steps must be taken to minimize backgrounds and to remove higher order wavelength contamination. In a typical experiment, the data at the lowest Q represent neutrons that are totally externally reflected which defines unity reflectivity. Above the critical angle the reflected neutron curve falls sharply in intensity. It is in the analysis of the Q-dependence of the reflected data above Θ_{crit} that the physical information on the details of the interface (magnetic moment or density gradient, etc.) are obtained. The field of neutron reflectometry is currently attracting a lot of attention and is an instrument that should be seriously considered by a reactor facility if sufficient flux is available and low backgrounds can be achieved.

5. SUMMARY

In this paper we have tried to present some rudimentary details and considerations for developing neutron scattering instruments at low and medium flux reactors. Many of the conclusions and recommendations are admittedly subjective and far from comprehensive. Because of their ubiquitous nature, the only instruments considered for this discussion were powder diffraction, SANS,

triple axis, and reflectometer machines. There are other types of scattering instruments, generally more specialized, which have been built and very successfully used at many intermediate flux sources throughout the world. Polarized beam scattering instruments were also not considered here because they are more demanding of source flux (with the exception of depolarization experiments).

As a first line of information for reactor facilities contemplating new or upgrading scattering instruments, the many articles in *Neutron News*[3] can be very helpful and point the way to more extensive references concerning details of specific instruments.

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THE ORPHEE VERSATILE LOW-COST MULTIPROCESSOR SYSTEM FOR DATA ACQUISITION AND CONTROL OF NEUTRON SPECTROMETERS

G. KOSKAS
Laboratoire Léon Brillouin,
CEN Saclay,
Gif-sur-Yvette, France

Abstract

This paper describes the new data acquistion and control system of the neutron scattering instruments at the ORPHEE research reactor. The existing system has undergone a complete change: the original CAMAC system and minicomputer controlling each experiment have given way to commercial CPU boards and microcomputers like the IBM PC. The communication links between these two components are the IEEE 488 or RS232 standards. Emphasis is placed on the flexibility and modular nature of such a system which makes a maximum use of commercial products thus guaranteeing reliability and ease of use. A study of the requirements and evolutions, technical as well as philosophical, is detailed to demonstrate the motivation of the choice of the system architecture. A survey of the various hardware and software achievements and finally an overview of future improvements is given.

INTRODUCTION

There are 23 neutron spectrometers installed around the ORPHEE research reactor at the CEN-Saclay (France). As the instruments were constructed, their hardware and software became extremely varied and uncoordinated. Most of the experiments used the CAMAC system and a multitasking minicomputer shared by several users, while others used specialized calculators (DIDAC, MULTI 20) or a centralized microprocessor system. The CAMAC included a number of different modules dedicated to each kind of command or measurement. The physicists were not able to write, on the driving computer, the software required by each module, so their handling was reserved for programmers only. In addition, 4 or 5 layers of serial electronics were introduced between the computer controlling the experiment and the physical measurement or control. This complexity made tests and repairs extremely difficult. With such a diversity in material and design, problems arose concerning maintenance and implementation of new instrumentations. A new acquisition and control system was therefore conceived, bearing in mind that most of the existing spectrometers had to be reused and that the new system should be easy to maintain, expand and control. These requirements are similar to any neutron and X-ray spectrometer of medium rate data acquisition. The system architecture described below is a solution in updating any reactor or synchroton instrumentation and in meeting new requirements.

ELECTRONIC REQUIREMENTS OF ORPHEE SPECTROMETERS

The requirements are similar to those of process control. Unlike neutron spectrometers for pulsed sources, the time of flight instrumentation is a small part of the entire facility (1/4 at ORPHEE)

and acquisition rates and time resolutions are 20 times less. Our spectrometers (like triple-axis or two axis) are machines where control features are as essential as acquisition features because the experiments are not static: the experimental environment must change automatically (wave length, temperature, pressure, magnetic field, sample etc...).

Even though our 23 spectrometers vary considerably they share a certain number of functions:

PULSES COUNTING AND TIME COUNTING

The duration of an experiment varies from a few minutes to several days. Two to ten counters are used with preset scaler or timer operation. The time resolution is 1/100 sec. wide and the maximum content of the scalers is more than 10,000,000 counts. There are 2 modes of operation: continuous mode and "flipping" mode where neutrons are alternatively submitted to a polarizing field.

POSITION SENSITIVE DETECTOR (P.S.D.) ACQUISITION AND TIME OF FLIGHT

A P.S.D. can be assimilated with a mosaic made of counters or "cells" (30 to 16000 at ORPHEE). Discriminator modules and position digitizer modules are already integrated in the P.S.D. electronics whose role is to send the number of the cell hit by the neutrons. The simple non time of flight mode consists in counting the number of times each cell is touched. The time of flight operation is considerably simplified by the performances required in acquisition rate (highest rate: 50 KHZ), time resolution (a few μsec) and maximum time channel number (250). Nevertheless, in order to reduce the amount of data, it is necessary to group cells into sets of various sizes and shapes.

MOTOR MONITORING

Some spectrometers have more than 15 motors (D.C. or stepping). Their power ranges from 1 to 100 Watt typically. The positionning precision (1/100°) requires a closed loop feedback control.

SIMPLE TTL I/O

TTL I/O are used to monitor functions like position readings, D.C. currents, temperature control, relay switching etc... These equipments exist in commercial systems and are conceived generally to be monitored by about 50 TTL I/O bit (e.g. position encoding, D.C. supplies).

ABANDON OF CAMAC SYSTEM

CAMAC was not suited to our instruments because CAMAC modules were either overdimensionned, inadequate or inexisting.

Pulse counting in the "flipping" mode needed 5 commercial and 1 customized CAMAC modules. Commercial CAMAC modules are excellent for high data rates in time-of-flight acquisition but overdimensionned for our needs. Such an application generally requires 3 or 4 kinds of CAMAC modules: histogramming module, TDC (time to digital converter) module, memory module (for data compression), FIFO module. There are no commercial CAMAC units to solve the problems of closed loop feedback

control of D.C motors with different kinds of position encoders (resolver, absolute or incremental optical, potentiometer). They have to be designed, are therefore expensive, and complicated to use because logical commands are separated from power commands. In conclusion, CAMAC was not advantageous in the control of our spectrometers because:

- CAMAC standard is not commonly used in industrial process control whereas our systems can profit from industrial applications (robotics, digitally controlled machine-tools, regulation, automatization etc...)
- Communication with CAMAC units is bulky because it is encoded
- CAMAC is expensive because of its poor distribution in industrial firms
- Computer choice is limited since few computer makers offer interface and CAMAC firmware on their products.

NEW SYSTEM DESCRIPTION

The aim of the system is to create a structure able to reuse the existing equipment more efficiently, accept commercial devices immediately and permit transformation into or conception as "intelligent spectrometry peripherals" of systems.

The basic concepts of the new system are:

- Division of the spectrometer into intelligent parallel completely independant electronic functions or "peripherals"
- Undertaking by the peripheral instead of by the computer of all the necessary firmware for driving each function
- Abandon of the interconnecting bus between electronic boards
- Simplicity of the interactive dialogue between the peripheral and the computer or the manipulator
- Unicity of the CPU board used in the stand alone mode to drive or fulfill each function
- Realization of the function by software rather than hardware
- Reduction of the number of interfaces between the functions and the computer
- Driving of the spectrometer by any kind of computer
- General use of widely marketed products
- Adoption of the IEEE 488 and/or RS232 standard to connect each peripheral (or CPU board) to the computer

To understand the system structure, the recently installed Spin Echo spectrometer will serve as an example (fig N° 1). It is composed of two levels:

1/ the commanding level and its 2 micro-computers - one commands the instrument while the other does a first order data processing in order to optimize the experiment

2/ the level that groups the different functions or modules of the instrument, each equipped with the same CPU board but with different customized firmware. The particular nature of the instrument requires the following 6 modules:

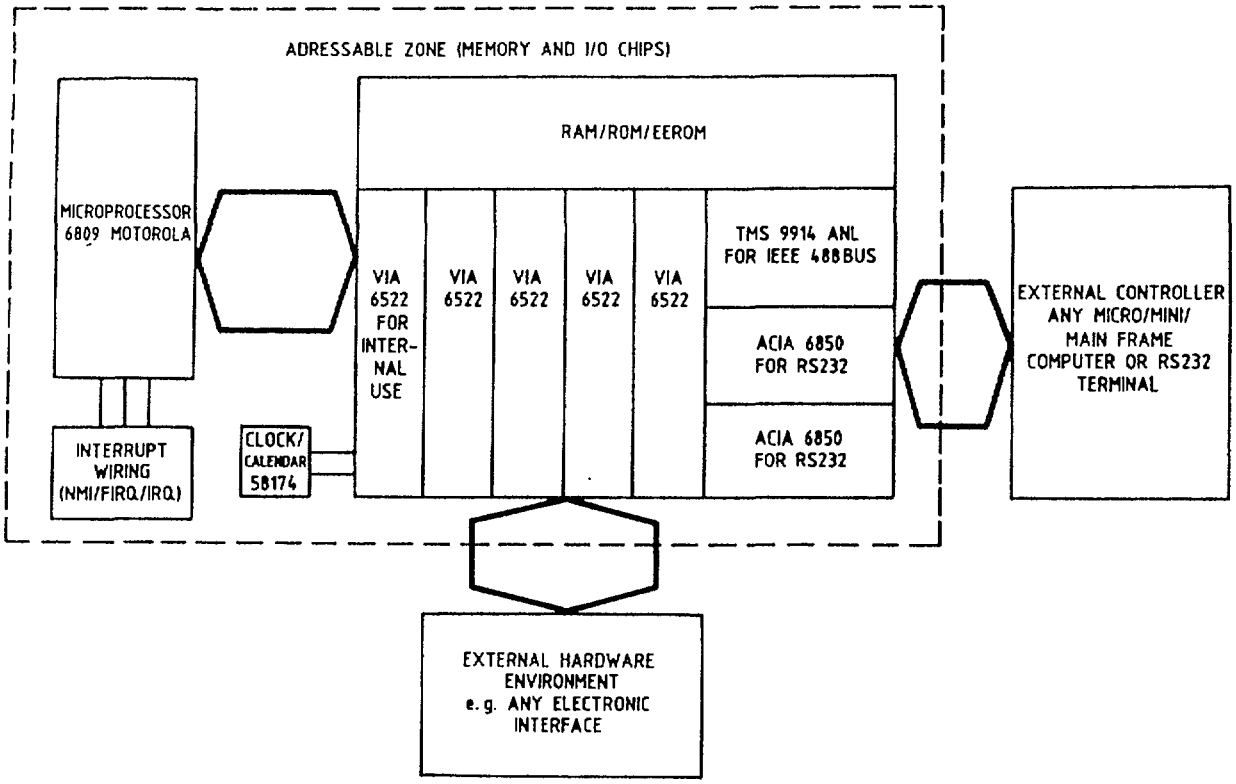

Figure 1

a) the module monitoring the stepping motors (one CPU card can control 12 or more motors)
b) angular position measurement module
c) the module monitoring the D.C. regulated power supplies
d) time monitoring and pulse counting module (5 counters)
e) fast time of flight with one counter module
f) data acquisition module with XY 32X32 P.S.D. and time of flight

Besides these peripherals, the spectrometer is equipped with other IEEE 488 commercial instruments (high performance voltmeter, high power D.C. supplies ...)

Each module is an IEEE 488 peripheral of the controlling computer and can be directed separately by just one RS232 line for the whole spectrometer. It goes without saying that, whenever a new function has to be added to the spectrometer, it is enough to connect a new IEEE 488 module on the BUS.

NEW SYSTEM ADVANTAGES

The structure is simple and regular and places the commercial and customized peripherals on the same level. Being autonomous, the peripherals off-load much of the monitoring computer's tasks and several instructions can be given simultaneously (eg counting and moving) due to the parallel structure. In addition, separating the spectrometer into intelligent and independant functions follows the physical features of the instrument. The users (physicists, biologist, chemist) can easily understand the system design and technicians can repair it

quickly. They can, for example, communicate with each part of the spectrometer by using a simple mnemonic language without passing through the controlling computer or disconnect momentarily a function (e.g. sample changer) without disrupting the experiment. The modular nature thus reduces further developments and brings uniformity, for though each spectrometer is specific, it is composed of a subset of common functions.

Due to the integration of components and electronic functions, all the features needed to solve each problem (CPU, memory, TTL I/O ports, serial I/O ports, A.D. and D.A. conversion, communications etc...) can now be set on a single board. From now on, the interconnecting bus can be abandonned and immediate advantages are obtained:

- no central controlling of the bus (which blocks the whole system in case of break down)
- Total independence of the CPU boards; their programming therefore becomes simple
- simplicity in setting up due to the independence of each function
- gain of speed and reliability due the decrease in interfacing devices
- no physical restraints of connecting the board on the same crate or linking differents crates
- possibility of integrating the CPU board into existing devices to convert them into intelligent computer peripherals, just like those in industry.

The reasons for choosing software rather than hardware were determined by several observations:

- The microprocessors available on the commercial market enable, in many cases, a microcoded electronics to compete with a previously hardwired one in speed of execution, and does more.
- Software design outclass hardware design in flexibility and ease-of-operation
- The software conception of a function is cheaper because it entails only the conception cost, not the manufacturing.

The IEEE 488 bus (GPIB) corresponds exactly to our requirements because it is the "IEEE STANDARD DIGITAL FOR PROGRAMMABLE INSTRUMENTATION" and is widely used in industry. This communication bus also provides the "opening" of the structure: for example, if performances so required, the system could integrate a CAMAC or VME crate equipped with a GPIB/CAMAC or VME interface. Vice versa, our peripherals can be connected to CAMAC or VME crates equipped with CAMAC or VME/GPIB interface.

COMPARISON WITH THE FORMER CAMAC SYSTEM

INITIAL COST

The setting up of the "SPIN ECHO" spectrometer with the CAMAC system would have necessitated 11 modules of 8 different kinds:

one stepping motor controller ($1500)	for function a)
one TTL I/O port ($1000)	for function b)
one TTL I/O port	for function c)

one clock unit ($1000) and
one scale unit $1500) for function d)
one histogramming unit ($2000) and
one TDC unit ($3000) for function e)
one histogramming unit,
one TDC unit and
one memory unit ($2000) for function f)
one crate controller type A2 ($2000)

plus a CAMAC crate and a CAMAC interface board inside the driving computer which amounts to a total of $27000.

In the new system, only 6 identical CPU modules are needed with a crate and a PC GPIB ($600) controller board, the whole totalling to $10000.

IMPLEMENTATION

To handle the CAMAC system, a library of subroutines was needed for each kind of module. The implementing of such a software is comparable to the specific firmware for each application integrated in the CPU board of our new system. On the other hand, when a commercial IEEE 488 device is available for a spectrometer (temperature regulation, DC supplies etc..), its programming is condensed to a few simple instructions - typically "write or read " character strings - instead of a serie of C(i)N(j)A(k)F(l) for a CAMAC module, if it exists.

EFFICIENCY

Several CAMAC modules (3 for "flipping" counting, 3 for P.S.D. time-of-flight etc...) can be replaced by a CPU board which provides the automatic monitoring of all the microcommands of a function (e.g. switching of multiplexers for position reading, air pressure command for motor driving etc...). It is able to send and receive data in a format adapted to a computer or a manipulator (e.g. formating of data, mnemonic language etc...). These characteristics have improved 10 to 50 times the speed of execution of several existing devices (position reading, transfer of PSD data etc..).

SYSTEM LIFECOST

The immediate financial advantages are further enhanced in our system by the use of the same kind of board (the CPU board) which considerably reduces maintenance duration, so efforts can be concentrated on the programming of a unique board. Besides, in the CAMAC system the driving computer, being the intelligence of the system, is often deeply involved in the BUS management; that is why the handling of the modules is often "computer dependant" (specially with interrupt management system) making the adaptation of the software on another type of computer difficult. In our system, the peripherals are intelligent enough to be monitored by a simple non-computer dependant program and improve the availability of the computer. Due to the universality of IEEE 488 BUS, our peripherals can be used in other laboratories.

COMPOSITION OF THE NEW SYSTEM

The different components of our system were selected in relation to the performances required. Higher performances can be obtained with more powerful material without modifying the described structure.

CONTROLLING MICROCOMPUTER

A widely commercialized product, the IBM PC XT or AT, controls each spectrometer (this type of computer has practically become the standard personal computer). It is relatively inexpensive ($4000-5000) and all improvements in PC's are made compatible with the IBM. Each spectrometer can, therefore, be equipped with one or many microcomputers, thus increasing their reliability and availability and minimizing the expenditure.

The microcomputer runs on MS DOS and has 512K RAM memory, one 20 Mbytes disk drive, two 640K floppy diskettes, one graphics printer. It also has a high resolution 600 X 400 pixels graphics monitor and a color screen can be attached.

The controlling program itself is written in high level language. All the parameters defining the experiment are set up on one or many files by means of interactive multiple windows software. To carry out an experiment, the monitoring program executes these run files. During the acquisition period, the peripherals are autonomous, so the user can quit the controlling program to execute other programs (calculations, editing, image processing etc...).

The P.S.D. data image processing display depends on their size and shape. The XY data can be displayed in 3D under different angles and scales of color. The graphic software allows image processing like display of several views, magnification of a window, cursor manipulation (eg. location of a given data). The data contained between two amplitude marks can be extracted and displayed in another color scale. Data are transmitted to the data processing computer by a network like ETHERNET.

THE MICROPROCESSOR BOARD

A commercial medium range CPU monoboard 8/16 bit, equipped with a 6809 Motorola microprocessor was selected: the "DAFFODIL" board, manufactured in 1983 by a Belgian firm.

It should be kept in mind that, within the structure of the data acquisition and control system, this CPU board fulfills the role of interface between "pure" electronics and the computer. Due to the presence of RS232 and IEEE 488 components, it can be connected to any kind of computer. Five Versatile Interface Adapter (VIA) IC's provide the CPU board the following programmable capabilities:

- high performance digital I/O (80 Bidirectionnal I/O TTL lines) with multiple "Handshake" and strobed I/O modes
- non standard serial interface
- external pulse counting provided by nine 16-bit counter-timer
- calibrated pulse generation
- programmable frequency square waves generation

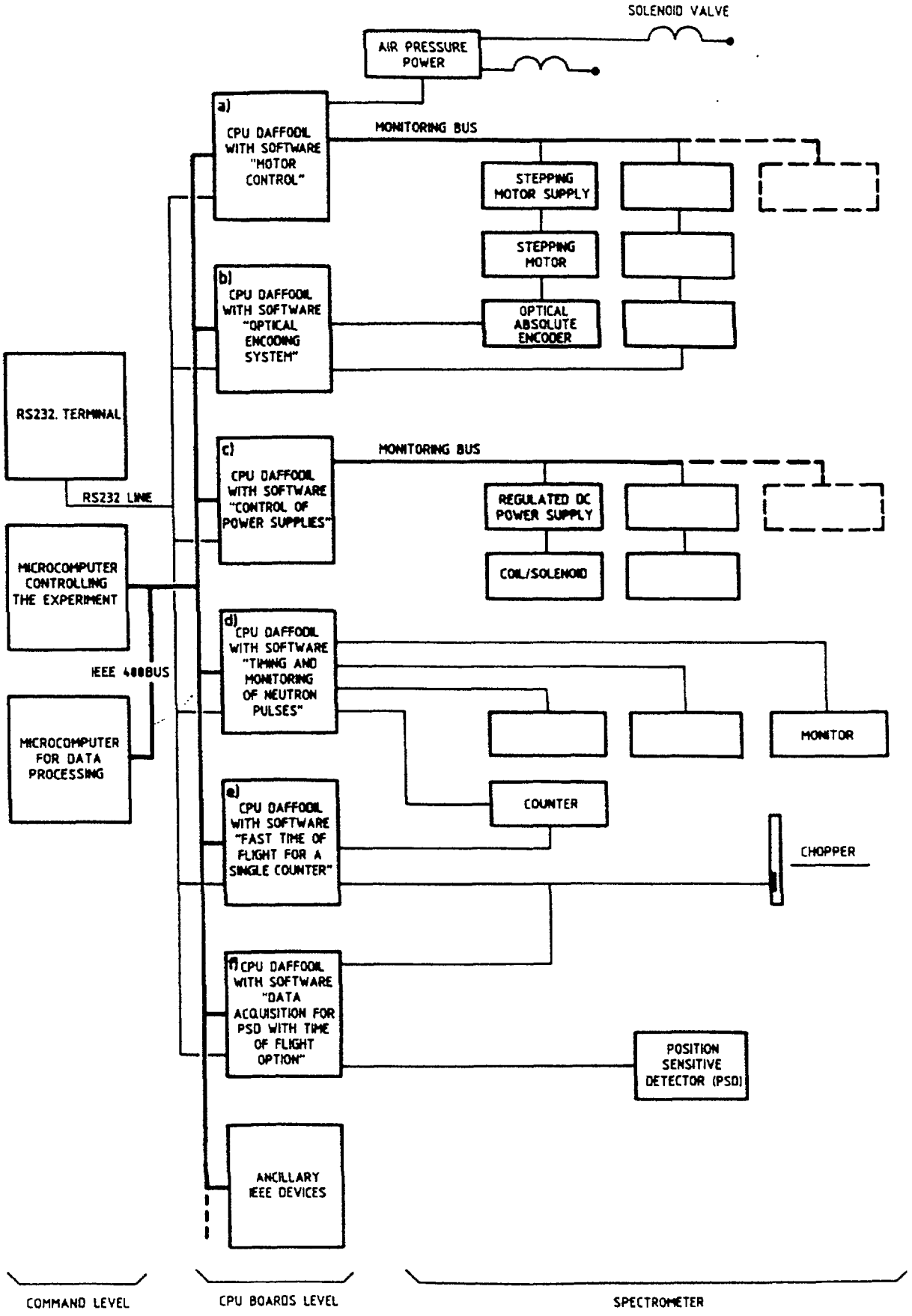
SOLENOID VALVE
AIR PRESSURE POWER
a)
CPU DAFFODIL WITH SOFTWARE "MOTOR CONTROL"
MONITORING BUS
STEPPING MOTOR SUPPLY
STEPPING MOTOR
b)
CPU DAFFODIL WITH SOFTWARE "OPTICAL ENCODING SYSTEM"
OPTICAL ABSOLUTE ENCODER
RS232. TERMINAL
RS232 LINE
c)
CPU DAFFODIL WITH SOFTWARE "CONTROL OF POWER SUPPLIES"
MONITORING BUS
REGULATED DC POWER SUPPLY
COIL/SOLENOID
MICROCOMPUTER CONTROLLING THE EXPERIMENT
IEEE 488BUS
d)
CPU DAFFODIL WITH SOFTWARE "TIMING AND MONITORING OF NEUTRON PULSES"
MONITOR
MICROCOMPUTER FOR DATA PROCESSING
e)
CPU DAFFODIL WITH SOFTWARE "FAST TIME OF FLIGHT FOR A SINGLE COUNTER"
COUNTER
CHOPPER
f)
CPU DAFFODIL WITH SOFTWARE "DATA ACQUISITION FOR PSD WITH TIME OF FLIGHT OPTION"
POSITION SENSITIVE DETECTOR (PSD)
ANCILLARY IEEE DEVICES
COMMAND LEVEL
CPU BOARDS LEVEL
SPECTROMETER

Figure 2

The components of the DAFFODIL board enable it to undertake the functions expected from it, namely, easy connection to already existing equipments (fig n° 2) and conception of new functions.

CPU BOARD FIRMWARE ORGANIZATION

The firmware consists of an on-board specific command interpreter for each module (pulse counting, time of flight etc...) is written in machine langage and uses a library of strict structured routines which allow for quick and simple modifications and improvements. The firmware is mainly divided into 2 distinct categories: one covers the user language (computer or manipulator) through IEEE 488 and RS232 links and the other connects external electronics, or makes the function itself. Including on-site interactive testing of all the subdivisions of the function (components, wires etc...), the firmware of each module, generally, does not exceed 5 Kbytes half of which are identical to all modules.

The programming of the CPU board is simple for two main reasons:

- each board is the interface of a unique function or fulfills a unique function, and then runs a few tasks (maximum 3)
- the programming of a new application does not interfere at all with the existing ones.

The tasks run on different priority levels:

1) low-level: command interpreter, orders execution and checking external electronics to detect defects.
2) medium-level: RS232 or IEEE 488 communication. This interrupt is masked during the command completion.
3) upper-level: interrupt management of VIAs request (start/stop, counting overflow, "data present" etc...).

The in-board interpreter: its aim is to make the use of the module easy to understand by non-specialists. The dialogue with the controller (computer or manipulator) consists in a character string made up of a mnemonic, followed by a variable number of parameters: eg "T1000" means start counting for 1000 1/100 sec.;"N100000" means start counting for 100000 neutron pulses; "I5,-10.123,7,3.25" means put -10.123 Amp. on power supply 5 and 3.25 Amp. on power supply 7.

To generate this language a few simple instructions are enough on the driving computer:

with a COMMODORE microcomputer in BASIC :

```
SET$="I5,-10.123,7,3.25" : PRINT#SUPPLY,SET$
```

Various command formats can be fed into the syntax analyser, but the output format is adapted to the high-level languages on the controlling computer. The language syntax on all modules is identical and a beginner can, with a general "help" command, use each module without entering into the details of its functionning. A number of commands are reserved for the maintenance technicians to question the board on its status and environment.

From the functionnal point of view, each character of the command string provokes a medium-level interrupt request until the validation terminator. This interrupt has, of course, no effect on the tasks running on upper-levels. The string is then entrusted to the syntax analyser which activates the corresponding processing subroutine.

Connection with external electronics is provided by the judicious use of VIAs and their programming. Depending upon the problem to be solved, the corresponding operating mode is selected by writing into VIA registers. Here again a library of subroutines provides the manipulation of the more commonly used external I/O electronic systems, although the difference between the various sytems begins to appear. For example, in a DC supplies command board application, a certain protocol respecting an established timing has been provided. In fact it amounts to driving a specialized bus, defined by the conceptor of these DC supplies, and its addressing mode, encoding functions and synchronization modes. The section of the program which undertakes the supplies interfacing allows the DAFFODIL board to be the controller of this specific bus. Below is an example, in assembly language, of VIA programming to send data to an external device:

Writing of 10 digits with automatic "data ready" strobe

```
LDA#OUTB          ;all 8 bits of B port as output
STA VIA-DDRB      ;into Data Direction Register B
LDA#PSM           ;Pulse Mode
STA VIA-PR        ;into VIA Peripheral Register

LDX#DATA          ;"X" CPU register points on data
LDB#10            ;10 data
LOOP
     LDA ,X+      ;reading data
     ANDA#$FO     ;low order data
     STA VIA-PB   ;each Port B output provokes "data ready"
                  ;signal
     DECB
UNTIL EQ
```

Conception of one timer, four scalers and one preset scaler : each CPU board offers 9 counters in its 5 VIAs. Four generate clock, the other 5 are presetable can record 65536 pulses, each at a rate up to 600 KHZ. An upper level interrupt is sent to the CPU when they overflow. The 24 bits amplitude is obtained by counting by software the number of overflows. One clock is programmed to produce an interrupt every 1/100 sec. The CPU counts these interrupts and detects either the right number of overflows of one scaler (preset scaler) or the preset number of clock interrupts (timer operation). In the "flipping" mode with polarized neutrons, another dissymetric programmable clock determines the moments when magnetic field is to be set. At each transition of the clock, counters are fixed and their contents memorized during the stabilization of the field (1/100 sec). The counters are then preset with the content they reached at the end of the preceding alternation and the cycle starts again. The end of the counting is calculated with the preset value or the duration value when there is an even number of rotations.

Conception of time-of-flight acquisition with P.S.D.: The P.S.D. acquisition (formerly hardwired, conceived with specialized computers) is now totally fulfilled by the DAFFODIL board by software. All the acquisition parameters (eg. time channel number and its width) are software selectable. Though the performances required are medium: 50 KHZ counting rate;16 or 24 bits amplitude per cell;256 time channels of 5 µsec depth minimum, the essential difficulty lies in acquisition and counting speed. The fact that the surface elements managed are not necessarily single cells is another difficulty; they can be a set of different shaped cells grouped together.

The acquisition takes place in the following manner: the DAFFODIL board awaits the start analysis signal; once received through a sensitive VIA bit, it triggers a clock the period of which is defined by a value written into a timer of another VIA. This period is the time channel width. The CPU then awaits the "DATA PRESENT" signal from the P.S.D. hardware through another sensitive bit of a VIA that triggers the latching of the data. This latching gives a buffer one data deep. Once received, the CPU simply reads the data, this reading sends automatically the "DATA TAKEN" signal. The cell number is combined with the running time channel number. Then the CPU finds out, through an indirection map, to which set the cell hit belongs and, depending on the time channel number, adds one in the definite address. The indirection map's task consists in memorizing for each cell the address of the first time channel of its set. This map is programmable cell by cell and/or in rectangular areas by means of the following command:

"R7,1,8,16,10" means: the cells contained by the rectangle at intersection of column 1 and row 8 in bottom left corner, 16 cells wide and 10 cells high, belong to area "7".

To avoid losing time in saving and recalling CPU registers, the corresponding programming sequence shown below does not run on an interrupt level. Data transfer to the computer stops the acquisition, but only during its memory refresh - 50 milliseconds every 5 seconds for a 32X32 cells P.S.D. which is quite negligible -.

Main acquisition sequence

```
WAIT LDA SIGNAL      ;Waiting for the start acquisition signal
     BNE WAIT
     LDX PERIOD
     STX TIMER       ;Triggering time channel clock
LOOP SYNC            ;Waiting for the "DATA PRESENT SIGNAL"
     LDB CHANNEL     ;Reading of the latched time channel
     LDX (CELL)      ;Reading of the latched hit cell
                     ;and finding its group set
     ABX
     ABX             ;combining time and group set
     INC ,X
     LDA ENDCHANNEL  ; Last channel ?
     BNE LOOP        ; No
     BRA WAIT        ; Yes
```

The loop sequence runs in less than 20 microsec., and the VIA buffer is freed 10 microsec after its reading.

CONCLUSION AND FUTURE DEVELOPMENTS

The implementation of the data acquisition and control system of the ORPHEE spectrometers, i.e. the basic counting modules, position encoding, motor controlling and P.S.D. data acquisition took about 6 months.

The delay of new instruments installation has been reduced from one month to one week due to the autonomous parallel modular structure, the integrated on-site testing programs, and the system's coherence. Despite the diversity of instruments, only 10 different modules firmware are used in the entire facility and each spectrometer uses, on the average 4-5 modules. The system is two or three times cheaper than the former CAMAC system, better adapted and easier to operate.

The costly motor driving and encoding systems on the experiments are gradually being replaced by a peripheral, designed by the Laboratory. This system, which integrates the DAFFODIL board, is very modular since the different types of position encoders: absolute optical, incremental, synchro/resolver (1/1000° accuracy) and potentiometer can be used with both D.C. and stepping motors.

Acknowledgements

The author owes a great debt to P. FROMENT regarding both his major contribution to the design of the control system and his valuable help during its completion.

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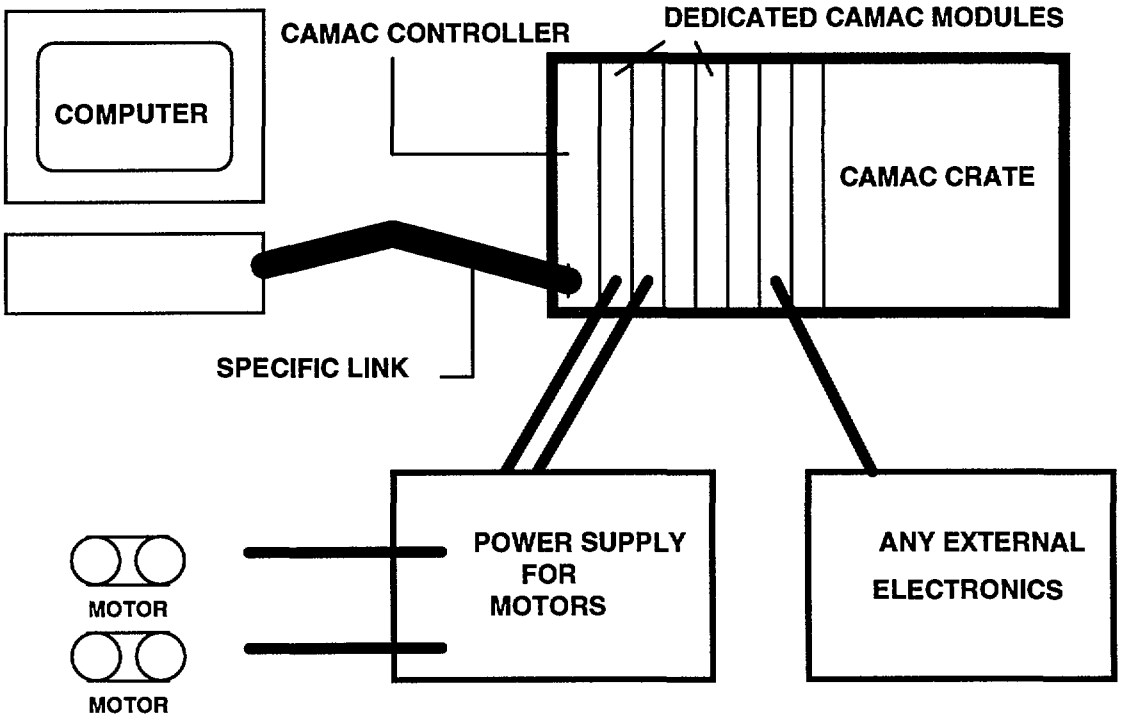
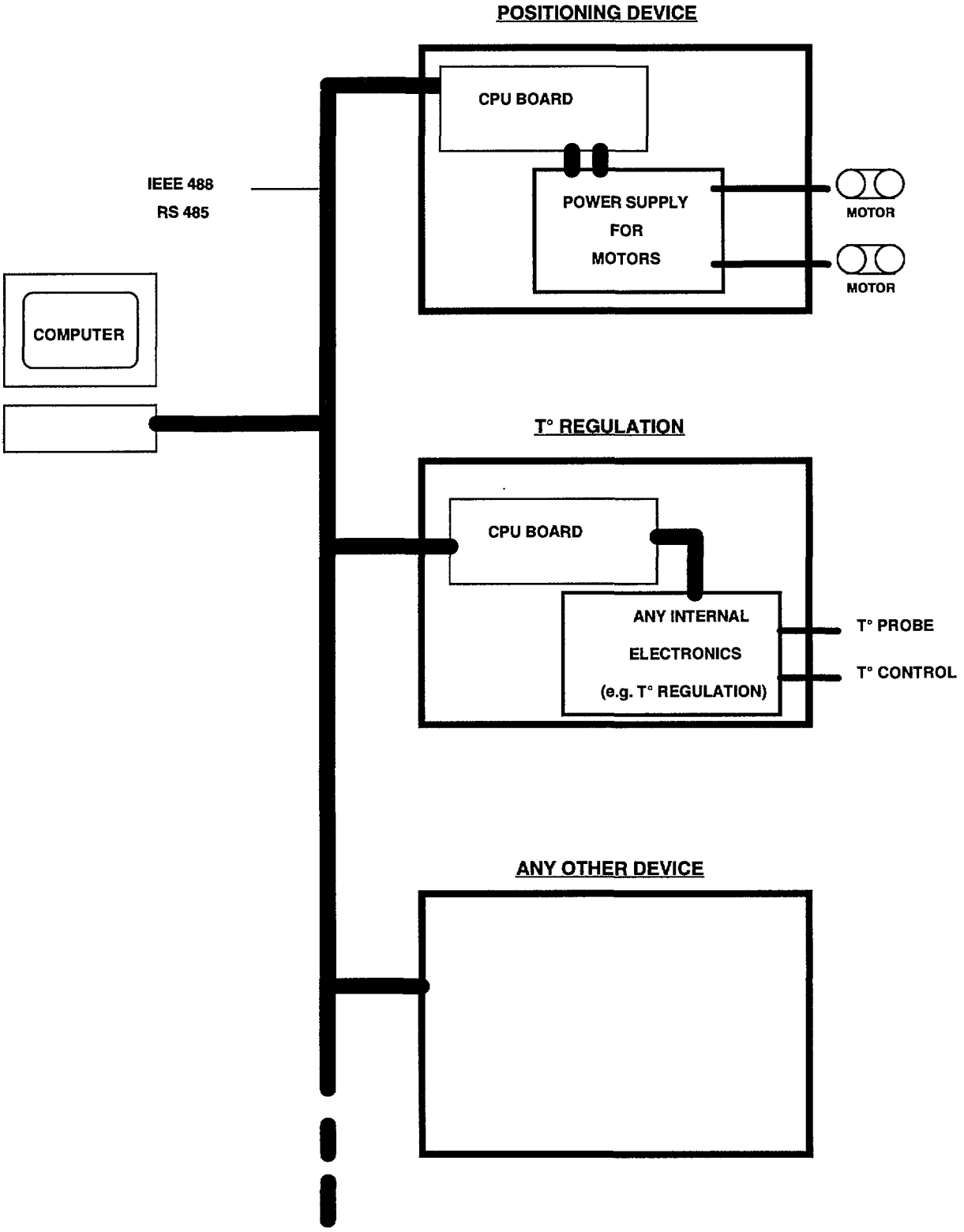
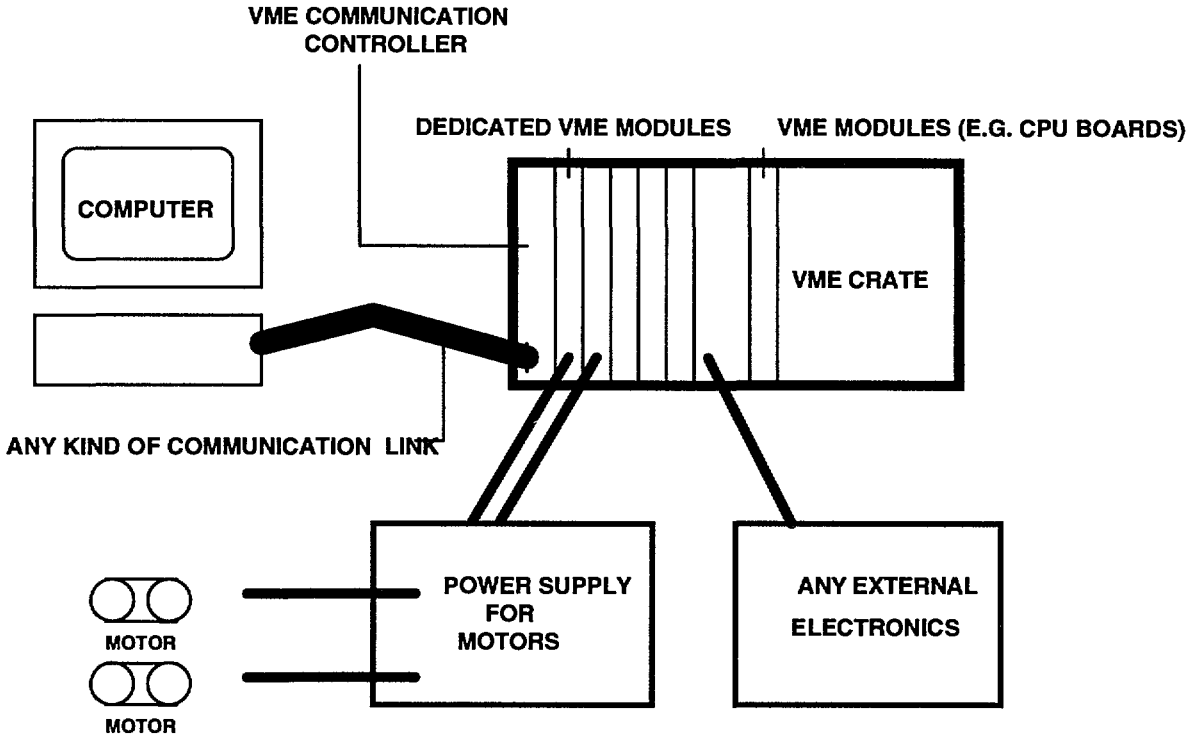
He is also much indebted to them for a critical reading of the manuscript as well as for helping him to cast this paper in its final form.



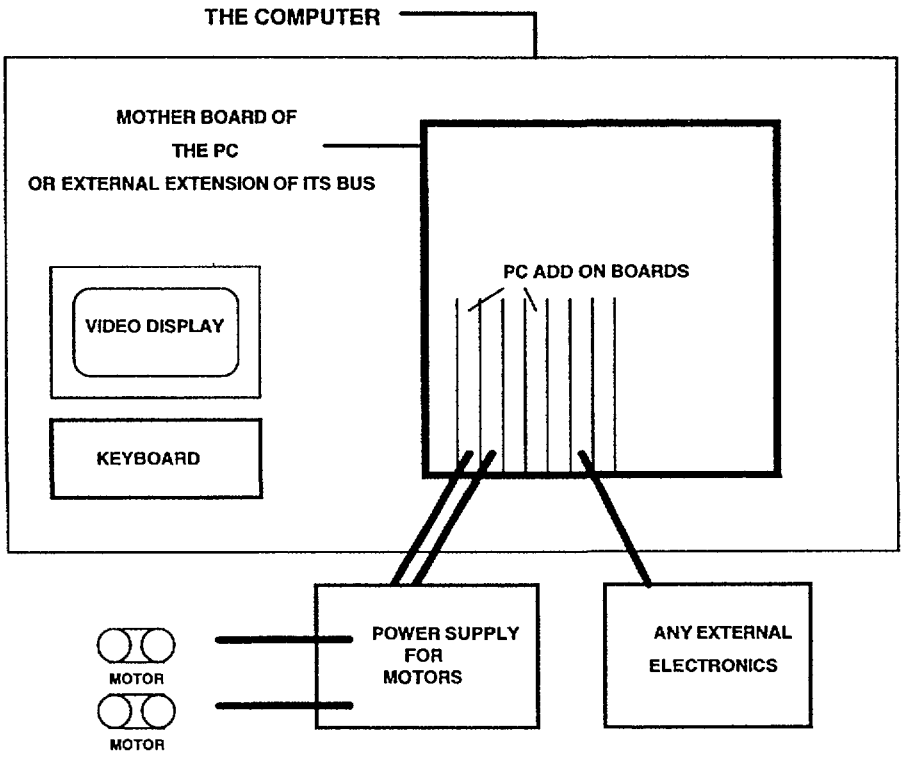
CAMAC style structure



Instrument approach style structure



VME style structure



PC with add on boards approach

TECHNICAL PROBLEMS IN ACQUISITION AND CONTROL SYSTEMS OF NEUTRONS SPECTROMETERS

Introduction

It is difficult to find a general solution for every case because the situation of each research center is different. The hardware of the spectrometer is often very old, sometimes it is too modern and there is a problem of local maintenance because of a lack of trained staff. Very often there are no financial resources to update the systems. Most of the time there is lack of organisation.

Because of this last point, the team (if there is one) in charge of the experiment does not have a clear view on the system architecture as a whole, and perhaps, does not even feel the need to have one. Yet, the adoption of a general architecture and standards is a key point. It will bring uniformity between the hardware and the software components and as a consequence a better efficiency of the technical staff.

If a system is to be chosen what should be its qualities ?

The key factor is: simple enough to be mastered and to be improved by the local task force: It should be

. inexpensive
. based on easily available components
. easy to install and to customize
. very easy to test and to repair.
. etc...

There should be a good compromise between high technology and simplicity. Therefore it must

. be modular
. rely on widely accepted standards
. accept the connection of equipement from commercial vendors
. be able to reuse parts of a previous system
. etc...

OVERVIEW OF SOME COMMON ACQUISITION AND CONTROL SYSTEMS

This little presentation is of course not exhaustive. It is intentionaly kept simplified . Therefore it is not objective and not corresponding with much more complex situations.

A) The CAMAC standard

Description:

The computer that controls the experiment is connected to a CAMAC crate. In this crate there are CAMAC modules that fulfill a specific task: for example, stepping motor control, position encoding and so on. (Sometimes the CAMAC controller is the computer itself, and sometimes inside the crate a CAMAC controller is connected to the computer via a specific link)

Advantages

- The CAMAC is a widely used standard in Nuclear Research. Many different kinds of CAMAC modules for various needs can be found, either in the commercial market or developped by other research laboratories.

- CAMAC is a modular standard. Typically, if a new function is to be added, it is enough to insert a new module in the crate.

- CAMAC is modular at the level of the communication between the CAMAC controller and the modules. Each module responds to a certain number of functions very well classified and defined for writing, reading and control.

Inconveniences

- CAMAC is rather expensive because of its poor distribution in industrial firms

- It is rather difficult to make a CAMAC module for unskilled personnel

- Computer choice is limited since few computer makers offer CAMAC interface and firmware on their products.

- CAMAC standard is not commonly used in industrial process control whereas systems can profit from industrial applications (regulation, digitally controlled machine-tools etc...)

B) VME or MULTIBUS type standard

Description

The computer is connected to a VME or MULTIBUS crate through standard communications links like ethernet, IEEE 488, RS232 etc... In the crate multiple processor boards are connected to specific modules dedicated for each feature of the experiment. The processor boards can work together to fulfill a complicated task. They run under a real time operating system like OS 9. In fact the VME or MULTIBUS crate acts more like a specialised computer than as a hardware extension of the monitoring computer whose role is mainly to process data.

Advantages

- VME standard tends to replace CAMAC: it is more and more often used in nuclear research

- there are now many kinds of VME modules available

- Very high acquisition rates can be reached by such a system

- Very complex and automated tasks can be performed by the system without control of the main computer

Inconveniences

- VME or MULTIBUS modules are still expensive

- VME modules are often overdimensioned for a simple use

- the knowledge necessary to master the BUS of the system is important

- the knowledge necessary to do the programming of each processor board is important. It is at the level of a UNIX like operating system.

- The boards inside the crate are not independent. In case of breakdown it may be difficult to debug the problem.

C) "Instrument" approach

Description

The computer is connected to several instruments or devices using a standard communication link like IEEE 488 RS232 or RS485. Each of these devices fulfills a complete function like counting, positionning, PSD acquisition etc....The devices are completely independent from each other. They share only the link to the computer. From the computer point of view the programming is fairly simple: it consists of sending messages to the devices and of receiving data using a more or less comprehensible syntax.

Advantages

- The system is conceptualy satisfying: for each function there is an independent and intelligent device

- Control and test of the devices are simple because they are independent, and physically separated

- IEEE 488 and RS232 are widely accepted standards and can be implemented easily in any kind of computer

- Many IEEE 488 and RS232 devices are available, connection of a new device is basically simple and direct

- No physical limitations of connecting the electronic boards on the same crate or of linking different crates

Inconveniences

- IEEE488 and RS232 devices are still expensive

- The design of a stand-alone device requires a good knowledge of how to write the firmware of a microprocessor board.

D) PC with add-on boards approach

Description

Add-on electronic boards are placed directly inside the PC. It is conceptualy like the CAMAC system except that the PC BUS is used instead of the CAMAC BUS.

Advantages

- The PC BUS is maybe the most widely distributed standard

- Many kinds of boards for various needs can be found, often at a cheap price

- ADD-ON board come frequently with very good software. Often they can be programmed using simple BASIC instructions.

Inconveniences

- No computer choice: the electronic system is completely computer dependent

- Physical restraint to connect all the wires to the core of the computer

- Difficulty to design a PC board because it requires a very good knowledge of PC hardware system

A single system should be selected among these standards. And if it is not possible, not more than two.

Controlling computer

Due to its wide distribution and its increasing power the use of a PC is a good choice. It is inexpensive and all improvements in PC's are made compatible. It is a general purpose computer, and it can be used also as a development system for microprocessor boards. The development tools are also inexpensive. User-friendly aspects of the program that controls the spectrometer can be met easily, using windowing software.

Communication with external devices

It is necessary to select a standard of communication between the computer and the external devices. RS232 and IEEE 488 can be opted for. They offer the possibility to implement easily new features to the experiment by simply connecting other devices. IEEE488 BUS (as well as RS485) has the advantage over RS232 to allow multiple connections on the same cable. Many inexpensive IEEE 488 interface boards are available for a PC.

System structure

It seems that the "instrument Approach" is a good solution. Its main advantage is to be easy to understand because it is coherent with the organisation of the experiment into separate and functional parts. e.g. for a neutron spectrometer:

-counting
-positioning
-PSD acquisition
-control of sample environnement
-regulation
- etc...

Because each of these functionnal parts are completely separated they can be designed carefully. They can also be tested easily which is very important in case of breakdown.

The major drawback to this approach is the difficulty to build such a stand-alone device.
There are mainly 3 problems:

1) the device, if it is intelligent, requires a CPU board running with a specialized firmware
2) the CPU board must be connected to a special hardware to fulfill the expected function
3) the device must provide communication with the controlling computer.

However, one should keep in mind that there are several suppliers of stand-alone microprocessor boards providing REAL TIME BASIC like environnements. Consequently, there is no need to write drivers to support the communication with the computer since it is ready made. In addition, there is often no need for additionnal hardware, the CPU board being directly able to fulfill the basic electronic functions like counting or TTL I/O.

In cases where the acquisition rates are not critical, which is normally the case for reactors with low neutron fluxes, the many TTL I/O facilities used in combination with the on board components and with a high level language such as BASIC is a key point:

-it allows to reuse already existing electronic parts using, for example, a simple BCD connection.
-it allows to connect directly a customized electronic board for special needs, opening the way to local development.

Conclusion

A structure for the acquisition and control system of spectrometers must be chosen. Once the choice has been made, all the development effort must be done following the guidelines of the structure. If the aimed purpose is the optimum exploitation of local ressources, the first step should be to adopt the most simple system. That does not necessarily means low end technology, in fact it is sometimes just the opposite: It must be taken into account that due to the integration of the electronics, it is more and more feasible to design spectrometer instrumentation. But it needs consistent training and information for local manpower.

LIST OF PARTICIPANTS

AUSTRIA

Badurek, G.	Atominstitut, Schuettelstr. 115, A-1020 Vienna
Rauch, H. *(Observer)*	Atominstitut, Schuettelstr. 115, A-1020 Vienna

CHINA

Ye Chuntang	Thermal Neutron Scattering Laboratory, China Institute of Atomic Energy, P.O. Box 275-30, Beijing 102413

FRANCE

Koskas, G.	CEA-CNRS, Laboratoire Leon Brillouin - CEN Saclay F-91191 Gif-sur-Yvette, Cedex
Mason, S.A.	Diffraction Group, ILL, Institute Max von Laue - Paul Langevin, B.P. 156, F-38042 Grenoble

INDIA

Rao, K.R.	Solid State Physics Division, Bhabha Atomic Research Centre, Trombay, Bombay 400 086

PAKISTAN

Butt, N.M.	PINSTECH, P.O. Nilore, Islamabad

PORTUGAL

Carvalho, F.G.	Physics Department, LNETI/ICEN, Estrada Nacional 10, 2685 Sacavem
Margaça, F.A. *(Observer)*	Physics Department, LNETI/ICEN, Estrada Nacional 10, 2685 Sacavem

SLOVENIA

Dimic, V.	Institut Jozef Stefan, Jamova 39, 61111 Ljubljana

SWITZERLAND

Bauer, G.S. — Paul Scherrer Institute,
CH-5232 Villigen

UNITED STATES OF AMERICA

Rhyne, J. — Research Reactor Facility,
University of Missouri, Research Park,
Columbia, Missouri 65211

INTERNATIONAL ATOMIC ENERGY AGENCY

Lewkowicz, I. — Industrial Applications and Chemistry Section,
Division of Physical and Chemical Sciences,
International Atomic Energy Agency,
P.O. Box 100, Wagramerstrasse 5,
A-Vienna, Austria

Akhtar, K.M.
(Scientific Secretary) — Physics Section,
Division of Physical and Chemical Sciences,
International Atomic Energy Agency,
P.O. Box 100, Wagramerstrasse 5,
A-Vienna, Austria

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