





INSTRUMENTATION

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Introduction

In 2004 and 2005, funding for the LLB was limited. Though investments were reduced the CAP2010 instrumentation program was continued. Mid 2005, the signature of the new contract for the LLB between the CEA and the CNRS opened up a bright future. The CAP2010 program is now moving ahead at full speed and nearly half of the spectrometers of the laboratory take advantage of our efforts.

2005-2006 achievements

DIFFRACTION

Much effort has been made on the diffraction spectrometers. Most of the work has been devoted to the improving the detection efficiency of the spectrometers. This will enable us to reach two targets; to be able to perform experiments with smaller sample quantities; and to be able to perform experiments faster which will make it possible to measure more points of the temperature, magnetic field, pressures and/or concentration diagrams.



Figure 1. VIP final design with multitubes detectors on 5C1

• The new High Resolution Powder Diffractometer **3T2** [*CI*, F. Damay] built with the support of "**Région Aquitaine**" was installed at the end of 2005. Compared to the previous version, it has higher number of larger detectors, and the efficiency is multiplied by 5. New protections will be installed end of 2006 to obtain a decrease of the background.

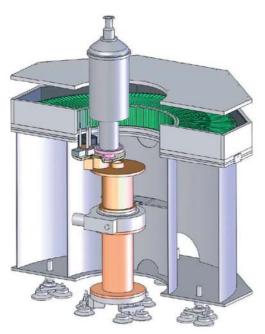


Figure 2. Modernized Very High Resolution Powder Diffractometer G4-2

- The single crystal diffractometer **6T2** [*H1*, **A. Gukassov**] is now equipped with a XY multidetector of a surface of 26x26cm² from INEL branch with the help of the "**Rennes métropole**". Development of software is now underway to enable more efficient use of a greatly increased collection of data.
- The design of the new detector of the **G6-1** powder diffractometer devoted to micro samples measurements is now completed [*H2*, **I. Goncharenko**]. It will use a set of 16 tube detectors. Tests with 8 tubes have confirmed an expected gain of intensity close to 30 compared to the current configuration. A new guide with supermirror coating was installed for this spectrometer in summer 2005.
- A new multitubes detector has been designed for the polarized neutron **5C1** crystal diffractometer (fig. 1). Its electronics, detectors and radial collimators have been ordered. It will take advantage as 6T2 did before of a large detection surface to improve the overall detection efficiency by a factor of more than 50. It will be installed in 2008.

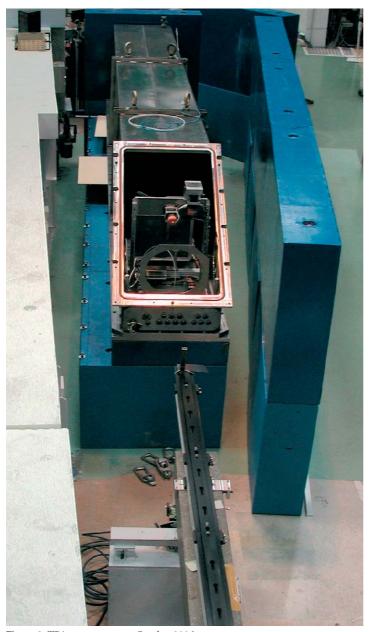


Figure 3. TPA spectrometer at October 2006

Two other important features must also be noticed. A new traction machine for small samples is now available on 6T1 and Diane. Installed on a three circles goniometer, it enables measurements on single grain in large grain alloys and will be mainly used for understanding energy and stress distribution in these alloys, which are more and more frequently used in industry. After a long shutdown due to technical problems, G4-2, the very high resolution powder diffractometer from Gatchina is under reconstruction. A new setup has been designed and will be installed at the beginning of 2007 (fig. 2).

LARGE SCALE STRUCTURES

In the field of the study of structure at large distances, the CAP2010 program comprises two main projects. The most important one is the construction of a new spectrometer for measurements at very small angles. Called TPA from the French "Très Petits Angles", it is a standard pinhole type small angle scattering spectrometer which has been designed to measure diffraction at Q values as low as 10-5Å-1 instead of 10-4Å-1 for our current small angle scattering spectrometer PAXY. It will enable the measurements of structures up to a 1 µm in size, and will fill the gap between neutron and light scattering. Basic concepts of this new spectrometer were tested in 2004. It is equipped with a double multilayer monochromator [C2, S. Desert] and a multibeam collimator [C3, S. Desert] built with the help of our participation in the Joint Research Activities (JRA) "Neutron Optics" of the Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy (NMI3) European program. The detailed design and fabrication of the main components started in 2005. Delivery and installation occured in 2006 (fig. 3). All parts are now ready to be tested and are awaiting the restarting of the reactor.

An innovative detector with a CCD camera was developed for specific structural measurements at an intermediate scale, and soon provided very promising results [C4, P. Baroni].

Our second important operation of CAP2010 in this area is the optimization of the Eros reflectometer for higher flux on the detector to measure thinner layers. This is a four-phase project. Phase 1, consisting of the change of the chopper, has already been done, and has provided a doubling in flux. Phase 2 consists of the shortening of the collimator. The design study has been done (fig. 4), and the collimator has been ordered. It should be delivered and installed in spring 2007 and should provide a flux gain of a factor of 5. Phase 3 consists of an optimization of the sample to detector space; and phase 4, in a transfer of the spectrometer to a better end guide position.

In the field of innovative instrumentation for reflectivity, we have performed the first tests of the TilTof concept [C5, F. Ott]. It make it possible to perform reflectivity experiments with white beams and opens the way to potential gains of intensity of an order of magnitude compared to usual monochromatic or time of flight reflectometers.

INELASTIC SCATTERING

Two main achievements have been realized in this field. The first one is a direct improvement of the flux available on the samples of the cold triple axis 4F1 and 4F2 [H3, B. Hennion]. Taking the opportunity of the preparation of the thimble change of 4F (fig. 5), we have installed new collimators equipped with m=3 supermirrors within the 4F beam plugs. The flux gain at the sample place has been estimated at up to 80% under certain conditions, at the price of a (small) 15% loss in resolution. On the same instruments, we continue our efforts to improve the efficiency of polarized neutron experiments with polarization analysis, and a new polarized guide has also been purchased from the PNPI Gatchina. Installed in front of the graphite analyzer, it will provide a better flux and better polarization than the Eusler currently used. The second achievement is the definition and purchase of a new 10T superconducting coil for triple axis measurements. It should be delivered at the end of 2007.

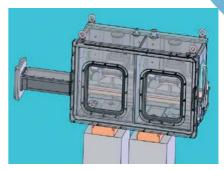


Figure 4. Eros short collimator

EUROPEAN NMI3 PROGRAM

In October 2006 we reached the mid-term of our participation in the NMI3 of the 6th framework European program. This program includes two different aspects. The main part is the Access program and is used to support the access to our spectrometers to new non French European users. The second part of the program is the technical networks called JRA. We participate in three of them:

"Neutron optics"

With the aim of setting up intense micro-beams, we have performed fabrication and tests of magnetic neutron waveguides [C6, S.V. Kozhevnikov]. We have already reported the development of the multibeam for TPA done within this JRA.

"Detectors"

We have succeeded in setting up a detection module with its electronics of 50x50 mm². We are now working on a project of tiling the space with many of these detection modules.

"Polarization Neutron Techniques"

We are working on setting up bent resonant coils for spin echo. This will allow the use of banana detectors to increase the counting efficiency of this type of spectrometers. With the collaborations obtained through this network, we have been able to install the longitudinal polarization analysis on our thermal single crystal diffraction spectrometer 6T2.



Figure 5. 4 steps of the works on beam tube 4. From left to right; 3T2, 4F1 and 4F2 in operation in June 2006, empty space beginning of July, thimble testing mid-July and 4F1-4F2 monochromator protection back in place in September.

Projects

MID TERM PROJECTS

Our modernization started with the CAP2010, projects will continue long after the end of this program. Our efforts will be put where neutrons will remain essential within the coming years: quasi-elastic scattering and small angle scattering. In quasi-elastic scattering two projects are already underway.

While preparing Fa#, our future quasielastic spectrometer, improvements of Mibemol continue. 100 detectors have been added in the small angle positions between 5° and 15°. A 64x64 cm² XY multidetector is ready to be installed near the direct beam to simultaneously provide structural and dynamical information in the 10 Å range. When Fa# becomes available, all the detectors will be transferred to the new instrument. Fa# will benefit of all the Mibemol improvements. A new guide will be completely optimized for it and will use the latest neutron optics development to provide this future instrument with the best possible optimization of the flux/resolution function.

For the study of even lower energy transfer, our resonant spin echo Muses will be improved. We are currently engaged in an NMI3 JRA network to design and test a banana-type coil that will enable measurements simultaneously at 10 different angles. This is expected to be in use in 2008.

Our small angles scattering spectrometers have provided good performances to numerous users for many years. Up to now, improvements have essentially been made in sample environments. We must now improve the instrument itself. Two actions have been undertaken. New velocity selectors are being purchased. They will provide a gain of intensity estimated at 30%, a more reliable operation, and the possibility of doing experiments with lower wavelength. Also long term studies have been started for the replacement of our current detectors by larger ones with better efficiency. Another 40% increase of intensity is expected in this direction. Studies are also being made in the evaluation of the ability of the time of flight technique in providing an easy way to do measurement on large Q range with a single measurement.

7TH EUROPEAN FRAMEWORK PROGRAM

Having in mind the 7th European framework program that will start in 2008, projects of collaboration are currently being set up with our partners of the NMI3 program. Efforts will be focused on collaborative programs that will end with a delivery that can be used immediately by our users. We have proposed to work in five directions:

- Software: Instrument simulation, data extraction, visualization and analysis
- Optic: Multi-beams and lenses focusing, neutron guide ageing
- Detector : Multi-tile detectors and integrated electronics
- Polarization: resonant spin echo with banana type detector, he versatile station for users
- Sample environment : pressure, shear, laser

LONG TERM PROJECTS

Other scientific areas will not be forgotten. A new innovative design for Diane, our strain scanner, is under preparation. The basic concept of the project is to provide an easy alignment of the sample by decoupling the sample stage from the systems of definition of the incident and scattered beams.

On longer time scale, we think that we are now at the maximum of flux delivery on the sample that can be performed on our triple axis with our source. Further improvement of our spectrometers to reduce the counting time or enable the measurements on smaller samples requires going towards multidetector systems. We are now undertaking the evaluation of **flat cone geometries** to find out if real improvements in efficiency can be provided by this technique. Conclusive arguments will be provided by simulation programs.

We intend also to evaluate **new cold sources design**. New codes have developed in neutronics laboratories which make it possible to obtain a realistic simulation of the neutron distribution and thermalization close to the core. They are fast enough to perform parametric studies, and as a consequence

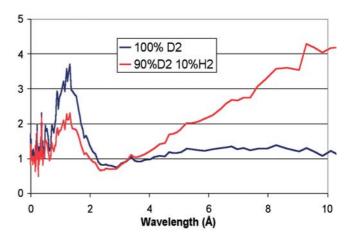
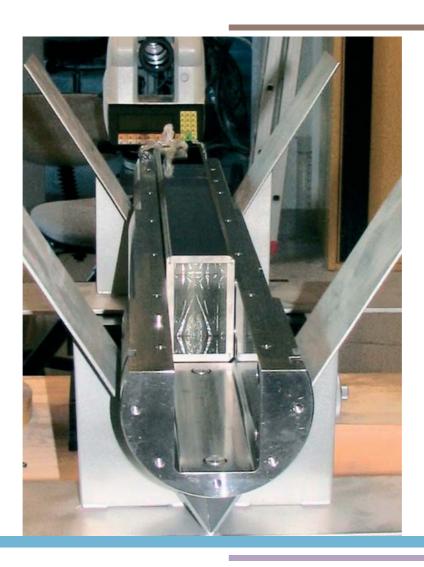


Figure 6. Evaluation of potential gain and losses in intensity compared to our current cold sources. Curves shown have been calculated with cold sources full of a mixture of liquid H2 and D2.

enable a better optimization of the shape and materials of the cold sources. In order to provide a good overall flux within a small size, our current cold sources suffer from under-moderation. The flux at large wavelength could certainly be increased by the use of new shape or new materials. These studies have to be started now, since if calculations show that an important gain can be reached, the requested modifications of the sources close to the core of the reactor will require long safety studies.

INSTRUMENTATION



- H1. Super-6T2, a new position sensitive detector polarized neutron diffractometer A. Gukasov, A. Goujon, J.-L. Meuriot, C. Person, G. Exil and G. Koskas
- **H2.**G6-1 "Micro", a powder diffractometer for micro samples measurements I. Goncharenko, X. Guillou
- H3. New Supermirror collimators for the in pile cold triple axis B. Hennion, P. Boutrouille
- [C1. F. Damay] Upgrade of the 3T2 High Resolution Powder Diffractometer
- [C2. S. Desert] A double super-mirror monochromator for the new Very Small Angle Neutron Scattering spectrometer (TPA, Très Petits Angles)
- [C3. S. Desert] Multi beam collimator prototype for the new Very Small Angle Neutron Scattering spectrometer (TPA, Très Petits Angles)
- [C4. P. Baroni] New developments for 2D High Resolution Neutron Scattering Experiments. Application case and experimental evidences from crystals to polymer science.
- [C5. F. Ott] TilTof a high intensity space-time reflectometer
- [C6. S.V. Kozhevnikov] Magnetic neutron waveguides

H1. SUPER-6T2, A NEW POSITION SENSITIVE DETECTOR POLARIZED NEUTRONDIFFRACTOMETER

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Polarised neutron diffraction (PND) is an important technique to investigate interatomic or intermolecular magnetic interactions. PND takes full advantage of the neutron magnetic moment and gives a direct access to the spin density distribution in the unit cell. In contrast to electron density, usually determined from high precision X-ray diffraction techniques, the spin density distribution is directly related to the unpaired electrons. Thus, by comparing spin and electron densities, one can get insights into magnetic interactions. The PND has been extensively used at LLB using the 5C1 diffractometer. Recently it has been successively applied to the studies of anomalous spin densities in ruthenates [1], of the origin of the field-induced ferro-metalic state in bilayer manganites [2], of staggered field effects in the one-dimensional antiferromagnet [3], of photoinduced molecular switching compound [4].

PND is also traditionally of particular interest for the community of chemists working in the field of molecular magnetism. Thus spin density studies in molecule-based magnetic compounds have permitted to obtain a very important information about the magnetic interactions in: ferromagnetically coupled copper(II) dimers [5]; ferromagnetic superexchange through the non-magnetic Ti(IV) ion in a Ti(IV)-(semiquinone)2 biradical [6] and the nature of the interaction in a Mn(II)-Ni(II) ferromagnetic chain compound [7]. Running a PND experiment is quite time consuming thus an improvement of PND diffractometers, which would boost the data acquisition rate, and as a consequence the precision of the information on spin densities is highly desirable.

Hence a new polarized neutron diffractometer has been commissioned at the ORPHEE reactor in Saclay. The instrument is installed on the thermal beam tube 6T2 at the ORPHEE reactor. Vertically focusing pyrolytic graphite is used to select the neutron of wavelength λ = 1.4 Å. The incident monochromatic beam is polarized with supermirror bender installed inside the monochromator protection. The bender has been designed and constructed in PNPI, Gatchina. It consists of 19 channels, each of 0.85 mm width, 50 mm height and 780 mm length. The supermirrors are deposited on 0.3 mm thickness glass and mounted with spacers of 0.85 mm thickness. An additional nonpolarizing supermirror focusing condensor can be inserted in the RF adiabatic flipper, when small samples are used. An adiabatic radiofrequency flipper [8] is installed between the polarizer and superconducting magnet of 7.5 Tesla, see Fig.1. The magnet is equipped by: ³He insert, CuBe pressure cell, very high pressure (sapphire) cell, kappa-geometry and photo-excitation inserts.

To reduce the scattering from the sample environment a radial collimator is installed in the detector protection (Eurocollimators Ltd) and covers 30° in vertical and horizontal directions. We found that the radial collimator reduces the scattering from the sample environment by a factor 10-20, which is of great importance for the performance of the diffractometer, working essentially in extreme sample conditions.

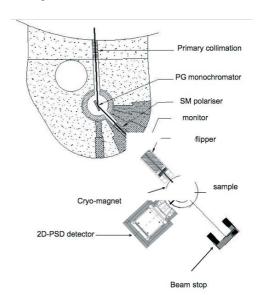


Figure 1. Schematic illustration of the Super-6T2 diffractometer.

The polarization of the incident beam was tested using CoFe single crystal and was found to be P₀=97% (Flipping Ratio ~70). Measurements on bench mark single crystals of CoFe and Ni using single counter, have shown the intensity gain of a factor of 15 compared to the intensity of existing polarized neutron diffractometer 5C1 at LLB. This gain factor will be increased even more with the use of the PSD. In this setup, the diffracted beam is collected by position sensitive multiwire gas detector BIDIM26 developed in ILL [9], fabricated by INEL and bought with the financial support of "Rennes Metropole". It is an individual readout square Multi-Wire Proportional Chamber (MWPC) with a single gas volume. The number of readout channels is (128X * 128Y), with resolution 2*2 mm. The vertical and horizontal acceptance angles of the PSD detector are 25°x25°, (0.19 steradian).

Spinning of sample and collecting of 2D images at only four consecutive detector positions will provide both integrated intensities and flipping ratios up to sin (J/l) ~ 0.6 Å-1 in the horizontal plane and sin (J/l) ~ 0.2 Å-1 in the vertical one. Then for the frames having low statistical accuracy flipping ratios will be re-measured with a longer exposition time to reach the required accuracy. Once the data collection is finished, the vertical axis of the crystal can be driven into the horizontal plane by using kappa-goniometer inserted in the magnet and the missing reflection intensities along the originally vertical crystal axis can be measured.

Data acquisition is realized by means of a EuroPsd microprocessor module designed at the LLB. The module assures connection between the driving computer and the ILL PSD interface via the IEEE 488 bus. It is able to sustain very high acquisition rates (up to 5 Mhz) and to run in different modes, like polarized neutrons, Time Of Flight (TOF) and time resolved modes. The module is equipped by an onboard (16 bit) memory adapted for detector sizes up to 512x512 channels. This makes the acquisition being totally independent from the performance and capacities of the driving computer.

Data are collected using a VISUAL BASIC (VB) software developed in LLB. We chose an industrial standard XML (Extended Markup Language) to store the data collected from the PSD. This standard describes in an easy and intuitive way how the information is stored. It has an advantage as well that it comes with ready made parsers and standard XML editors. The XML file created by the LLB acquisition software contains compressed data collected from the PSD and all current parameters of the diffractometer; date, time, angles, monitor, temperature etc. Visualization program, see Fig. 2, written in JAVA gives quasi-instantaneous visualization and access to any selected frame due to the XML standard, providing tabulation files. The same program assures: reading of frame content, binning and summation of frames and creation of dynamic mask of strongest pixels. Program for indexing, integration and flipping ratio calculations is written in Fortran 90 and MATFOR 4.

For the crystals having small lattice parameters the gain from using the PSD will be very small. In this case we consider a possibility to use several single crystal of different orientation simultaneously. In particular if crystals are mounted at different height, the Friedel pairs can be used to define the zero offsets in the horizontal plane and to separate reflections belonging to different crystals.

Quite large detector aperture (0.19 steradian) allows to measure polarized neutron diffraction from powder samples with a high efficiency. We note that its acceptance angle is nearly four times larger that that of the existing powder diffractometers G4.1 and G6.1 at LLB.

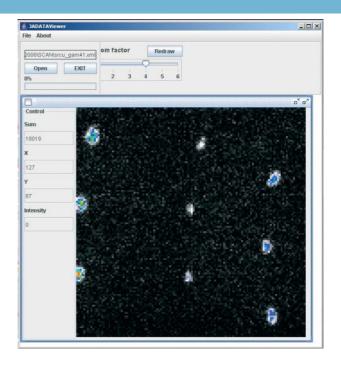


Figure 2. Visualization program window.

Using a long polarizing bender in combination with PSD on 6T2 improve the efficiency of the instrument by approximately a factor of 20-30 compared to the 5C1 diffractometer currently existing at the LLB. This will provide unmatched speed of data collection from very small samples with relatively large unit-cells.

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H2. G6-1 "MICRO", A POWDER DIFFRACTOMETER FOR MICRO SAMPLES MEASUREMENTS

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Pressures of about 30-50 GPa (about one tenths of pressure in the center of our planet) can be generated only in a small volume <0.1 mm³ using anvil pressure cells. Study of such small sample is a challenge for neutron techniques. For more than a decade, the G6-1 diffractometer in the LLB was actively used in high-pressure studies. The focusing system, installed in 1996-1998, increased flux at the sample place by order of magnitude, making the G6.1 one of the most powerful "cold" diffractometers in the world. Since 1998, the LLB holds world record in maximal pressure for neutron studies (50 GPa), and the capabilities of our neutron instrumentation and pressure techniques have been demonstrated in various studies of magnetically frustrated systems [1], or magnetic properties of high-pressure oxygen [2].



Figure 1. The "hybrid" cell; the latest generation of high pressure cell compatible with X-rays and neutrons.

At the present, the G6.1 undergoes a major reconstruction, which should make it fully optimized for high-pressure studies and keep it competitive with instrumentations installed on the next generation neutron sources. The main features of the upgraded version of the G6.1 ("MICRO") are:

- 1) supermirror guide before the monochromator;
- 2) monochromator allowing to vary wavelength from 2.3 to 5 Å;
- 3) focusing system between the monochromator and the sample place
- 4) multidetector covering the optimal solid angle ~ 1 steradian

NEUTRON GUIDE ENHANCEMENT.

The G6-1 is located in the guide hall of the Orphée reactor. Far from the reactor the fast neutrons and gamma rays background is very low. Measurements at high pressures do not require high resolution. As a consequence, gain in intensity can be obtained using a lower resolution, that is to say a larger divergence of the incident beam. Neutron guide with m=2 supermirror coating provide a double divergence in the horizontal and vertical directions compared to the previous G6 natNi guide, and hence a nearly proportional increase of intensity. Monte Carlo simulations have been used to calculate the intensity distribution on the G6-1 monochromator (see fig. 2). They show that a gain of 2.9 in intensity on the monochromator can be achieved.

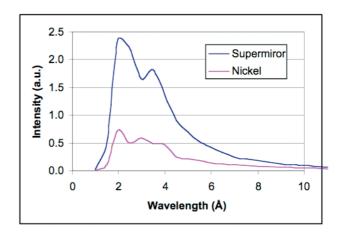


Figure 2. Intensity distribution calculated at the G6-1 monochromator for the two guide coatings: "at Ni (red) and m=2 supermirror (blue).

The replacement of the G6 guide by elements with m=2 supermirror coating has been done by the CILAS company in three steps, and is now completed.

2003: first 2.3 m within the beam plug, starting at 1.45 m from the cold source.

2001: elements between the beam plug and the reactor containment (a length of 9.8 m).

2005: last 23.7 m to the G6-1 monochromator.

All guide elements have been made in BORKRON glass except the last 21 m which have been made of borofloat glass. This provided additional 60% of intensity at the sample place (fig. 3). To take full advantage of the divergence given by the new guide, the G6-1 monochromator has now to be optimized.

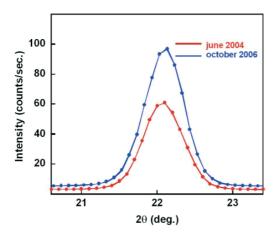


Figure 3. Bragg reflection from zeolite sample before and after the replacement of the guide

MONOCHROMATOR OPTIMIZATION

The current monochromator used on G6-1 is a made of 5 focusing blades of pyrolithic graphite of a mosaic of 40'. Each blade has a height of 3 cm. Made 25 years ago, the maintenance of the focusing system is now difficult. In addition, a 1.6 gain in intensity can be obtained by using 9 smaller blades with a higher mosaic of 60'. This new monochromator will also allow varying wavelength in the wider range $2.3 < \lambda < 5$ Å (compare to the present $4 < \lambda < 5$ Å). It had been fabricated in June 2006, will be tested by the end of 2006.

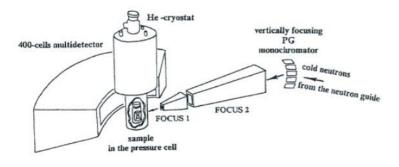


Figure 4. Full setup of the G6-1 instrument

IMPROVEMENT OF THE FOCUSING SYSTEM

Between the monochromator and the sample a double stage converging guide enables to obtain a maximum density of neutrons on the sample (see Fig. 4). Made with m=2 and m=3 supermiror coatings, it was optimized for the divergence delivered by the natNi coating of the guide. With a guide made with m=2 supermirror coatings, the mirrors of the converging guide have to be replaced by higher m coatings in order to made full use of the highest intensity available.

DETECTOR REPLACEMENT

The solid angle of the current multidetector of the G6.1



Figure 5. Test assembly of 8 linear-sensitive detectors.

(400 cell BF₃ "banana-type" counter) is only 0.1 steradian. Development of new multidetector, having solid angle by order of magnitude larger then the actual one, is the most crucial part of the "MICRO" project. After taking into account requirements for spatial resolution, efficiency and stability for the new detector, an assembly of 16 linear-sensitive detectors stacked horizontally had been chosen. The detectors (2.54x102.4 cm) had been fabricated by Reuter Stokes. In October 2005 a prototype assembly of 8 tubes (fig. 5) had been tested at the G6.1 in the real conditions of neutron. Result is presented on fig. 6.

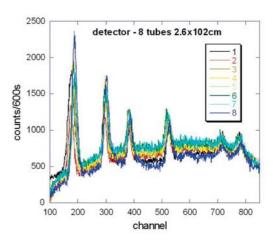


Figure 6. Neutron diffraction patterns collected by every tube of the test assembly at the G6.1.

Every single tube provided 70-90% of intensity of the banana detector, which results in gain in intensity of factor ~ 6 for the 8-tubes assembly, and expected factor of 12 for the final 16-tubes assembly. After the positive results of the test, the work is focused on design and construction of the new supporting table and protection. The detectors will be installed on a translation stage within their protection as reported on fig. 7.

Two different configurations will be available. At 40 cm of the sample, they will provide a high flux, low resolution configuration covering an horizontal angle of 100° and vertical angle of +/-25°. Efforts are currently done in setting up of the electronics (fabricated by Mesytec) and developments of appropriate software for collection and treatment of 2-dimensional spectra. First experiments with the final 16-tube detector are expected in 2007.

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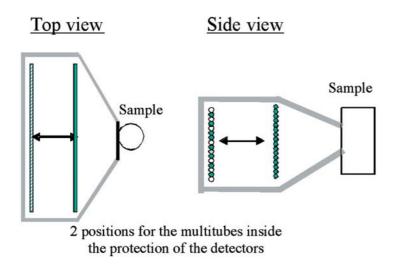


Figure 7. The new 16 tubes detector setup for G6-1.

H3. NEW SUPERMIRROR COLLIMATORS FOR THE IN PILE COLD TRIPLE AXIS

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Neutron optics elements are now commonly used to improve the flux on the instruments. Taking advantage of a maintenance on the 4F thimble, during the summer 2006, m=3 supermirror have been installed in the collimators of the 4F1 and 4F2 beam ports which feed two cold neutron triple axis. An expected gain of intensity on the sample up to 80% for some experimental conditions is foreseen.

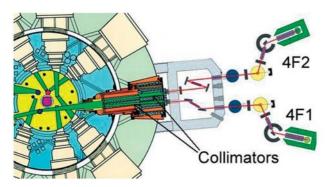


Figure 1. Setup of 4F1 4F2 cold triple axis spectrometers around the Orphée reactor.

Thimbles of beam ports are submitted to high radiation damages. They modify the metallurgical properties of the alloy which becomes more fragile. An important part of this effect is due to transmutation of aluminum atoms in silicon. As a consequence, thimbles have to be changed before the amount of Si in the Al alloy becomes larger than a known limit. This limit is close to be reached by the 4F thimble which has to be replaced. This is a long and delicate operation which requires the removal of 4F beam plug and the whole 4F1 and 4F2 spectrometers.

This is a unique opportunity to change the collimators inserted within this beam plug (see fig. 1) since the flux available on the 4F1 and 4F2 instruments is very dependant upon the geometry of these collimators. Such a modification has already been done in 1999 on the channel 2T [1] where an increase of the size of the beam plug collimators did provide a gain in intensity of 80%. However, an increase of

the mean gamma radiation level on the 2T area has been notice afterwards.

During these last years, tremendous progress have been done in supermirrors neutron optics elements [2]. They provide a way to transport more neutrons through the beam plug collimators without having to change their size, and hence without changing the radiation protection provided by the beam plug. Supermirrors are especially efficient with the cold neutrons used on the 4F spectrometers. This is why we have chosen to insert m=3 supermirrors in the beam plug collimators without changing their size (see fig. 2).

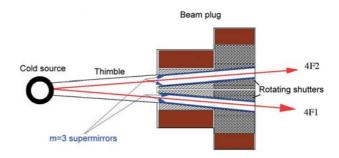


Figure 2. Schematic view of the cold source, thimble and beam plug assembly.

M=3 supermirrors enable the transmission of neutrons which hit the mirrors up to angles of $0.3*\lambda$ degrees, (λ being the wavelength of the incident neutron in angstroms). Compared to collimators without guides, they allow a transmission of a larger divergence, and provide a higher flux on the monochromator.

Simple ray tracing simulations have been performed in order to obtain an estimation of the gain provided by the supermirrors. A source of equal and homogeneous intensity has been assumed. These simulations enable to obtain an estimation of the loss of angular resolution and energy resolution due to the increase of divergence of the incoming beam on the sample.

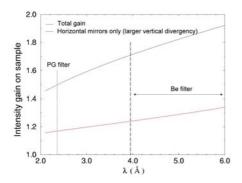


Figure 3. Calculated gain with supermirrors as a function of wavelength in two different configurations (see text).

Two different configurations have been taken into account :

- **a.** Install only horizontal mirrors at the top and bottom part of the collimators (increase only transmitted vertical divergence).
- **b.** Mirrors installed on the 4 sides of the collimators (increase transmitted vertical and horizontal divergence)

Results of the expected flux gain compare to the current collimators without mirrors are presented on Fig. 3.

It shows that horizontal mirrors (configuration **a**) bring only a gain of 20% in intensity (red curve). This comes from the fact that the current height of the beam (8 cm) provides already the transmission of a divergence of 5°. M=3 supermirrors just add an additional 0.9° at 3 Å. However, this gain is obtained with a negligible loss in resolution.

Configuration **b** with horizontal and vertical supermirrors in the beam plug is more interesting since it provides a potential gain varying from 40% at 2 Å to 90% at 6 Å. The corresponding energy resolution degradation has been calculated to be 15% at maximum. For most of the inelastic experiments performed on these spectrometers this is an acceptable degradation.

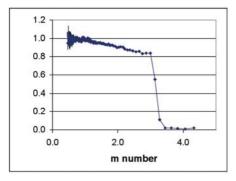


Figure 4. Neutron reflectivity measurement of one of the supermirrors inserted in the beam plug.

Our rotating shutters offered 3 different positions. In order to be able to perform high flux or high resolution experiments, we have chosen to install the 3 following configurations:

- High flux: Horizontal and vertical supermirrors m=3
- Intermediate : 40' collimators without mirrors
- High resolution: 15' collimators without mirrors

Supermirrors have been purchased from the Mirrotron company, and collimators from the GMI-STEEM company. Supermirrors have been deposited on float glass substrate which is known to sustain high irradiation damages without degradation. The reflection quality of each mirror has been checked before being inserted in the beam plug. A typical measurement is represented on fig. 4. They show up a reflectivity coefficient above 80% at m=3.

Mirrors have then been carefully aligned, and inserted in the

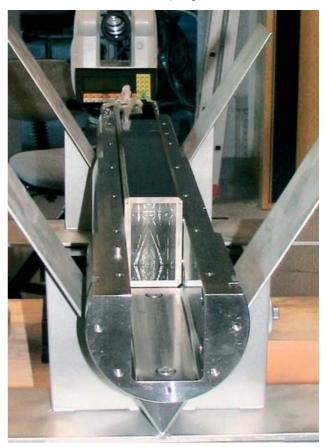


Figure 5. Alignment of the mirrors in the collimator

beam plug in july 2006 (fig. 5). At the restart of the reactor, test measurements will be performed. They will have to show that, no significant increase of the gamma radioactive background is present, and validate the calculated gain of flux on the sample. If deviation to the predictions will be detected, we will take the opportunity of the second part of the maintenance of 4F thimbles that will occur in summer 2007 to apply corrective actions.

LLB Scientific report, 1999-2000 p.108-113
 O. Elsenhans, P. Boni, H.P. Friedli, et al., Thin Solid Films, 246 (1994) 110-119.

[C1. F. Damay] Upgrade of the 3T2 High Resolution Powder Diffractometer

Research on novel materials of technological interest is an ever expanding area which requires fine structures determination to fully understand and tailor materials properties. Examples in ionic conductors, solid electrolytes, catalysers, high temperature superconductors or magnetic semiconductors can easily be found. Neutron powder diffraction plays a key role in these fields, because it provides an easy way to locate light atoms and to determine an existing magnetic order.

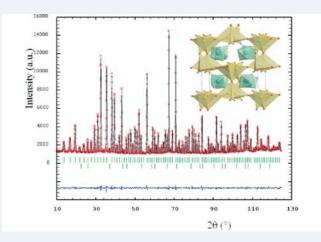
LLB proceeded mid-2005 to an important upgrade of the detection module of the 3T2 high resolution powder diffractometer. Electronics has been replaced by news LLB elements and the spectrometer is now equipped with an array of 50 new ³He large detectors and 50 new collimators made by the EuroCollimators Ltd company, which provide a resolution of 10'. Tests show that a gain of 2.5 in the overall detection efficiency has been achieved compared to the previous 3T2.

The next objective now is to reduce the background noise in order to get high quality data with smaller quantities of sample. To this purpose, additional neutron shielding has been designed and will be installed at the end of 2006. This project has been financed partly by the "Région Aquitaine".

[Contact: F.Damay, F.Bourée, B.Rieu, LLB]



View of the new bank of 50 detectors



Na₂Ca₃Al₂F₁₄ measured with the new 3T2

[C2. S. Desert] A double super-mirror monochromator for the new Very Small Angle Neutron Scattering spectrometer (TPA, Très Petits Angles)

On TPA, the very small scattering vector 2.10^4Å^{-1} is accessible by using a very high resolution (pixels of 150 µm) image plate detector located only 6 m far from the sample. Due to the high sensitivity to γ radiation of such detector, a conventional velocity selector cannot be used as monochromator because its neutron absorber, Gd, is a strong γ emitter. Instead, a new monochromator (figure 1) composed of two super mirror monochromators (critical angle m=3, Δ m/m=0.15) is now installed on TPA. The wavelength selection is achieved with the mirrors angle θ , according to $\lambda = \theta/(m\theta_c)$, where θ_c is the critical angle for ordinary Ni. Both mirrors are mounted on rotations, the second one being also mounted on a translation stage in order to keep the outgoing monochromatic beam at a fixed position when changing the wavelength. This monochromator has a good transmission (~70%) while avoiding the strong γ emission and a direct view of the guide. Figure 2 shows Time of Flight measurements of various wavelengths obtained with this new kind of monochromator. The resolution is constant: $\Delta\lambda/\lambda$ =0.11.

[Collaboration: V. Thévenot, A. Gabriel, P. Permingeat, S. Désert, A. Brûlet, LLB; J. Oberdisse, LCVN, Montpellier]

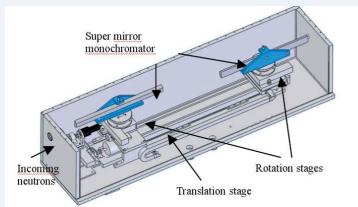


Figure 1. Drawing of the monochromator

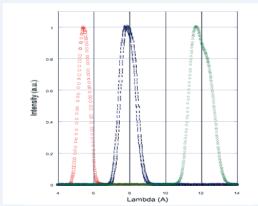


Figure 2. Raw TOF measurements of monochromated beams

[C3. S. Desert] Multi beam collimator prototype for the new Very Small Angle Neutron Scattering spectrometer (TPA, Très Petits Angles)

The very small scattering vector expected for TPA, q_{min} =2.10⁴Å⁻¹, will be achieved by using a tiny collimation. Indeed, the collimation for TPA requires 1.8 mm and 1mm diameter pinholes at the collimator entrance and exit. The drawback of the latter is the huge loss of neutrons. In order to enhance the effective neutron beam used, a multi beam prototype collimator has been built and successfully tested. It features 7 individual masks (figure 1), made from ⁶Li in an epoxy matrix, with 51 pinholes per mask. These masks are defining 51 beams converging on the detector while absorbing the unwanted neutrons (i.e. not focusing on the detector). Measurements comparing a simple collimation and the multi beam prototype with 16 pinholes show a gain in flux of 12. The advantage of such a setup is its flexibility regarding the wavelengths. Indeed, the fall of neutron due to gravity is not negligible compared to the pinhole diameter of 1 mm at the collimator exit (20 Å neutrons fall 2 mm after 4m path). The masks are mounted on translation stages to take into account for the gravity and thus only one setup is required for all the wavelengths. A multi slit prototype (used for isotropic scattering samples) is currently being manufactured and should improve the gain by a factor of 60 compared to the pinhole multi beam prototype. Deconvolution should then be achieved to get the true scattering curve.

[V. Thévenot, S. Désert, A. Brûlet, LLB]

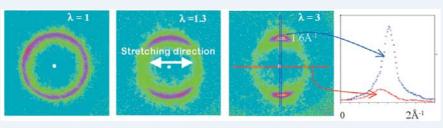
Figure 1. Picture of the prototype multi beam collimator with 7 masks

[C4. P. Baroni] New developments for 2D High Resolution Neutron Scattering Experiments. Application case and experimental evidences from crystals to polymer science.

We present here a new type of 2-dimensional neutron detector (CNRS CEA patent **WO2006095013** – publication date: 2006-09-14), allowing a high resolution investigation of the scattering space including both large to small angles.

This new system being almost insensitive to most radiations (γ , X radiation and visible light) except neutron radiation, is particularly adapted to the detection of neutron scattering or neutron diffraction, without exhibiting memory effects. Combining the advantages of the high resolution and of a 2 dimensional integrating readout, this new instrumental development appears as a reliable, efficient and evolutionary setup which could extend the possibilities of time-resolved experiments in this research field. An example of illustration of structural investigations carried out using this new system is displayed below. It is an original study at two-dimensions of the effect of an uniaxial mechanical deformation (elongational stress λ = L/L0 where L0 is the initial sample length) on the structure of a bulky sample of polytetrafluoroethylene (PTFE). The experiment points out different effects with respect to the stress, which can be interpreted by the uncorrelated contributions of the crystalline and of the amorphous parts respectively. The experimental conditions are: wavelength= 2.85Å, sample-detector distance: 53.5mm, exposure time: 5 min, beam diameter: 3mm, 16 bits data.

[P. Baroni and L. Noirez, LLB]

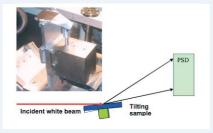


2D-Diffraction patterns displayed at 3 elongation rates (λ =1, 1.3 and 3) by the IV phase of the PTFE, observed from 0.05 to 2.2Å⁻¹. The fourth figure shows the vertical (blue points) and the horizontal (red points) profiles of the anisotropic scattering corresponding to the elongation rate (λ =3)

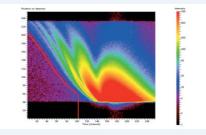
[C5. F. Ott] TilTof a high intensity space-time reflectometer

Being able to measure reflectivities down to 10° is the challenge of neutron reflectometers for the 21st century. The use of a periodic tilt of the sample, coupled with a position sensitive detector enables to perform specular reflectivity measurements on continuous neutron sources without any monochromator or chopper and thus allows intensity gains up to 10 compared to conventional reflectometers. The implementation consists in modulating the incidence angle of the beam by a periodic **Tilt** of the sample of a few degrees at a frequency of the order of 20 Hz. At each time, the sample reflects the neutron white beam at a different angle. The reflected beam arrives at a different position on the detector. The time of flight (**Tof**) from the sample allows wavelengths separation, and as a result, the full reflectivity curve is measured in each detector cell. The duty cycle of such a reflectometer is only limited by the velocity of the sample movements and may exceed 90%. We have performed the first measurements on a simplified setup (40% duty cycle) installed on the EROS reflectometer. Results show that the expected intensity gain has been obtained. However, mechanical instabilities conduct to poor resolution. In addition, off specular scattering from the sample gives a high unexpected background. A new version of Tiltof with less vibrations and equipped with a synchronized slit after the sample to cut background is under development to solve the two problems encountered.

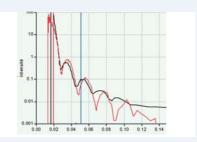
[F.Ott, A.Menelle, ICNS 2005]



Principle of TilTof and implementation on Eros



TilTof measurement of a Cu layer on silicon. X axis is time channels, Y axis is position on the detector.

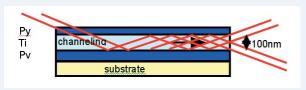


Reflectivity obtained on Eros with Tiltof (black) or with the usual chopper configuration (red).

[C6. S.V. Kozhevnikov] Magnetic neutron waveguides

In order to produce submicron neutron beams, we are developing neutron waveguides (NWG). The large magnetic neutron cross section allows to fabricate guides in which the optical index can be dynamically modulated. We produced NWG with the tri-layer structure: Py(10-20nm)/Ti(10-70nm)/Py(10-50nm)//glass. The top Py layer acts as the coupling layer with the incident beam, the Ti layer acts as the guiding layer and the bottom layer acts as the reflecting layer (see Fig. 1). We have characterized our systems by polarized neutron reflectometry (specular and off-specular) in order to probe the effect of the different imperfections (interface roughness, magnetic non-collinearity, dispersion of the layers thickness) on the reflectivity. We show that it is possible to guide up to 30% of the incident neutrons. This corresponds to a flux density of 10^8 n/cm²/s at the waveguide exit.

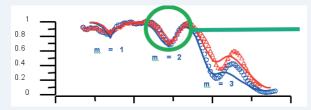
[Collaboration: S.V. Kozhevnikov, aFrank Laboratory of Neutron Physics, Dubna, F. Ott, LLB, E. Kentzinge, , Forschungszentrum Jülich]



Diffuse off-specular scattering on a waveguide structure.

The resonance modes in grey. (measured on HADAS)

Localization of the wavefunction in the NWG.



Specular reflectivity up to 30% of the incident neutrons are trapped in the guide

