

## 7 - TECHNICAL AND INSTRUMENTAL DEVELOPMENTS

In the last 2 years the investments on the development of new spectrometers has been limited. The 2 main developments are connected with Small Angle Neutron Scattering and 2 new spectrometers are being built. Other instrumental developments have consisted in incremental improvements of existing spectrometers. They either allow to obtain “soft gains” and to improve the flux of the spectrometer by factors of the order to 2-3 or consist in providing to the users improved sample environments.

### Spectrometers

#### SANS - Reflectivity

- PAPYRUS  
One of the existing SANS spectrometer (PAPOL) has been upgraded in order to make it suitable for the technique of GISANS (Grazing Incidence Small Angle Neutron Scattering). This spectrometer is optimized for the study of magnetic thin film nanostructures and can use a polarized neutron beam. Two persons are now fully dedicated to this spectrometer. More details can be found in the following highlight.
- TPA  
The study of new composite systems require Very Small Angle Neutron Scattering. Thus the spectrometer TPA (Très Petits Angles) is being development at the guide position G5bis. This spectrometer is optimized for the measurement of anisotropic large size nano-systems ( $2 \cdot 10^{-4} \text{ \AA}^{-1} < Q < 10^{-1} \text{ \AA}^{-1}$ ) (see following highlight).
- EROS  
The Time of Flight reflectometer EROS has benefited from incremental developments. A new multi-discs chopper system has been installed. It allows to work with a constant instrumental resolution over the entire Q-range. The collimation part of the spectrometer has been completely redesigned to make it shorter. The fabrication of the new collimator is under way. Large gains in flux are expected (up to 50 for a significant  $\lambda$  range).

#### Material science

- Textures  
The sample environment of the texture spectrometer 6T1 has been significantly improved. A furnace is now available which allows to perform in-situ experiments. An in-situ traction machine has also been designed in collaboration with the LPMTM Univ. Paris XIII (see following highlight).
- Strains  
The strain spectrometer DIANE has been upgraded so as to able to accommodate very heavy samples (~500kg) adapted to the study of “industrial” objects. An Euler cradle is now also available (which can handle pieces up to 20 kg).

#### Diffraction

- Powder diffraction

The high resolution powder diffractometer 3T2 has been redesigned in order to be able to accommodate a completely new detector system (detectors twice as high and twice the number of detectors). The gains in measuring time should be of the order of 5 (gains obtained in detection surface). The different pieces (collimators; detectors) of this new detector have been tested individually ; they should be setup during 2005.



The new collimators - detectors block for the spectrometer 3T2 (seen from the sample position)

This new system will allow :

- an improved flexibility (intensity/resolution)
- The possibility to study monoclinic structures with  $V_0 \leq 1200 \text{ \AA}^3$
- to study microstructures as a function of the temperature
- to study complex systems such as : molecular compounds, bio-materials, cement...

- Single Crystal diffraction

A new method of type "Quasi Laue single crystal diffraction" has been tested on 5C1 by using a large PSD system (see CAP 2010). The first tests have been satisfactory. The new principle of measurement will be evaluated further during the year 2005. Large gains in measuring time are expected (from 5 up to 10).

### Sample environments

#### High pressures

Various techniques have been developed to study neutron scattering under high pressures combined with applied magnetic field and/or uniaxial stress. New "hybride" pressure cells with diamond, sapphire and moissanite anvils are compatible with neutron instrumentation at the LLB and X-ray scattering facilities at the ESRF, allowing to carry out X-ray and neutron studies on the same sample. These pressure cells are used at the LLB on a number of spectrometers (powder diffraction G61, single crystal diffraction)

An example of the use of such cells is presented below :

#### New Medium Pressure (3 GPa) Cell for Single Crystal Diffraction in LLB

(B. Annighöfer, A. Gukasov)

A new hydrostatic pressure cell has been developed for single crystal diffraction experiments (see Fig. 1). The design of the cell allows a maximum pressure of 3 GPa, with the inner diameter of 4 mm and the maximum height of 8 mm. The cell is compatible with a superconducting magnet of 7.5 Tesla.

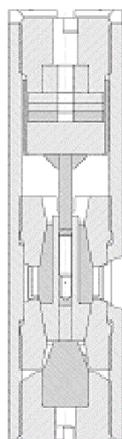


Figure 1: Pressure cell.

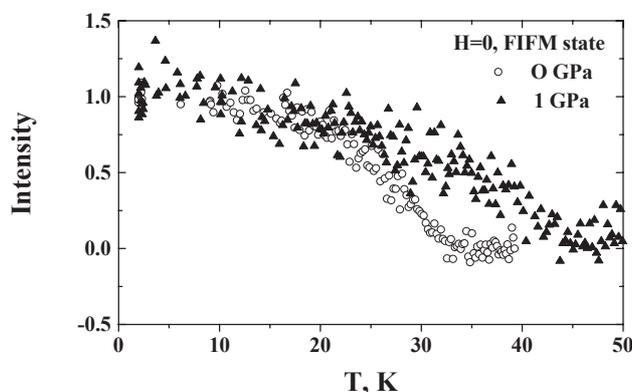


Figure 2. Temperature dependence of the (110) reflection in the FIM state under different pressures.

Test measurements have been carried out on a single crystal of bilayer manganite  $(La_{0.4}Pr_{0.6})_{1.2}Sr_{1.8}Mn_2O_7$ , which exhibit a field induced ferromagnetic-metallic (FIFM) phase transition. Without field, this material shows no spontaneous ferromagnetic ordering, and keeps its insulating state down to 2 K. A magnetic field of several teslas (at 2 K) triggers the onset of a long range ferromagnetism, simultaneously with the insulator-metal transition. This field-induced ferromagnetic (FIFM) state behaves like an ordinary ferromagnetic state and disappears at Curie temperature  $T_C=32$  K. Fig. 2 shows that the pressure of 1 GPa increases the Curie temperature of the FIFM state from 32 to 45 K.

### Pressure cells for biological studies

M.S. Appavou, G. Gibrat, B. Annighofer, M.-C. Bellissent-Funel (LLB)

This pressure cell has been especially developed for studying the protein folding and to comparing structural conformation and dynamics of native and pressure denatured states in solution. It has been optimised to allow both SANS and TOF experiments. With this cell, pressure experiments on NEAT spectrometer (HMI, Berlin) have been successively performed at a given pressure using SANS and TOF configurations of NEAT spectrometer.

This cell is made with a Cu-Be alloy (98%Cu-2%Be). The neutron scattering characteristics of this alloy are given in table 1. The cell has an internal diameter of 10 mm and an external diameter of 28 mm. It allows a maximum pressure of 600 MPa and can contain up to 7.4 ml of solution. A cylindrical insert is put inside the sample chamber to reduce the sample volume. The pressure transmitting media used is  $D_2O$ . (See photograph below)



New cells for the study of biological samples have been developed.

| Element | $\sigma_{coh}$ (barn) | $\sigma_{inc}$ (barn) | $\sigma_{abs}$ at $\lambda = 1.8 \text{ \AA}$ (barn) |
|---------|-----------------------|-----------------------|--|
| Cu      | 7.49                  | 0.55                  | 3.78   |
| Be      | 7.63                  | $1.80 \cdot 10^{-3}$  | $7.60 \cdot 10^{-3}$                                 |
| Alloy   | 7.49                  | 0.54                  | 3.7  |

Table 1 : Neutron scattering cross sections of the components of the cell.

### Non magnetic environment for 4F1

The spectrometer 4F1 has been modified to remove all the magnetic elements so as to be able to set-up a large cryomagnet.

Polarization analysis option is installed on the thermal beam triple axis spectrometer 2T. Unfortunately, the current setup is not fully operational as an insufficient flipping ratio is measured. This further depends on the incident wave-vector angle apparently due to a lack of neutron guide field between the monochromator and the sample. Additional tests are required to sort out that problem. They have been postponed due to reduced number of days of running reactor and the surcharge of the spectrometer.

### Guide systems

The guide G1, G2 and G6 in the plugs have been replaced by  $2\theta_c$  super-mirrors. Most guides have thus benefited from a measurable increase in flux (see report in the next pages). Some spectrometers do not show any gains because some of the main guides still use simple Ni coatings. During 2005, the guide G6 should be upgraded with  $2\theta_c$  super-mirrors. The main benefit will be for the spectrometer G61.

### Future developments

- A new PSD detector will be installed on 5C2 during the year 2005 to improve the collection rate of the spectrometer.
- The spin-echo MUSES will benefit from a multi-detector system increasing the collected solid angle.
- In the long term, a new time of flight spectrometer is being designed. The funding for this new spectrometer has however no been secured yet.