8 - TECHNICAL AND INSTRUMENTAL DEVELOPMENTS

One of the important goals of the LLB activities is the research and development of neutron scattering methods. This topic concerns very different aspects of neutron scattering technique, the increase of the luminosity of neutron spectrometers, realisation of large area position sensitive detectors and development of new approaches in the data acquisition and in the data treatment.

It also includes the development of new sample environment devices, which allow to perform neutron measurements in extreme conditions, for example at low temperatures (down to 80 mK) in high magnetic fields (8 T), and high pressures (4 GPa) applied simultaneously.

A special attention during the last year was paid to elaborate a new instrumentation program, called CAP2010, which summarizes the "roadmap" of future technical and instrumental developments at the LLB for the following 8 years.

Development of spectrometers

Polarised neutron option on the upgraded thermal three-axis spectrometer 2T :

After the enlargement of the beam tube size 2T in 1999, a polarized inelastic neutron option has been installed on the thermal beam triple axis spectrometer 2T. The setup is a standard triple-axis spectrometer with Heusler monochromator and analyzer, similar to that of IN20 and IN22 at ILL, with an incident energy up to about 100 meV. Heusler single crystals have been grown at the ILL. The horizontal and vertical sizes of the monochromator and analyzer, respectively 140x130 mm^2 and 120x100 mm^2, have been chosen to fully make use of the available beam size. Permanent NdFeB magnets, delivering a 1.2 T field, are used to saturate the Heusler magnetization. The magnetic field is applied horizontally on the monochromator, which has a vertical focusing, and vertically on the analyser, which has a horizontal focusing. This combination of curvatures has been chosen as the most effective and hence, the beam geometry for polarized neutron is basically similar to that of the unpolarized beam. The flipping ratios of ~12 so far obtained are encouraging and will be improved in the near future. A preliminary polarized experiment has been performed on high-T_c superconductors: an intensity ratio between the unpolarized and the polarized setups is about ~20. Some improvement is expected from a realignment of the Heusler analyzer, which was found to be non optimal. In any case, with the enlargement of the beam size on the 2T spectrometer the intensity counted in the detector is now comparable to that of the polarized triple axis spectrometers IN20 and IN22 at the ILL.

Bidimensional neutron detectors for the 7C2 spectrometer on the hot source:

The LLB is currently developing micro-strip gas counters (MSGC) based on the charge division principle. These detectors, developed in the frame of the European TECHNI (n° HPRI-CT-1999-50005) contract, are suitable for small area 200×100mm^2 and good spatial resolution (~ 2mm). They rule out the possible mechanical problems related to wire grid detectors by replacing the wires with anode and cathode metal stripes deposited on a glass substrate.

A MSGC consist of a gap chamber filled with high-pressure ^3He gas (up to 10 bars) to capture the neutrons and create an electrical charge. The charge amplification is performed by a high electrical field (~1000 V/m) between anodes and cathodes (the amplification gain of about 10^4). This electrical charge is split in two by a resistive line. The position of the neutron is determined by measuring the charge ratio between the two ends of the resistive line. The charge division design has been chosen because of the very low cost of the associated electronics (the number of charge amplifiers is limited to 3.) The gamma discrimination is performed by measuring the total electrical charge collected on the cathodes. At the moment, the largest size available is 200x100mm^2 (see Fig.1). It is limited by the fabrication and the capacitance of the micro-strip glass plate itself. A bidimensional position-sensitive detection can be achieved by using two perpendicular sets of stripes (on the front and on the back of the glass substrates).

Ultra High Vacuum techniques have been used for the design of the detectors casings in order to maintain the purity of the detection gas. This is essential to prevent any deterioration of the metallic anodes stripes and to avoid parasitic signals.

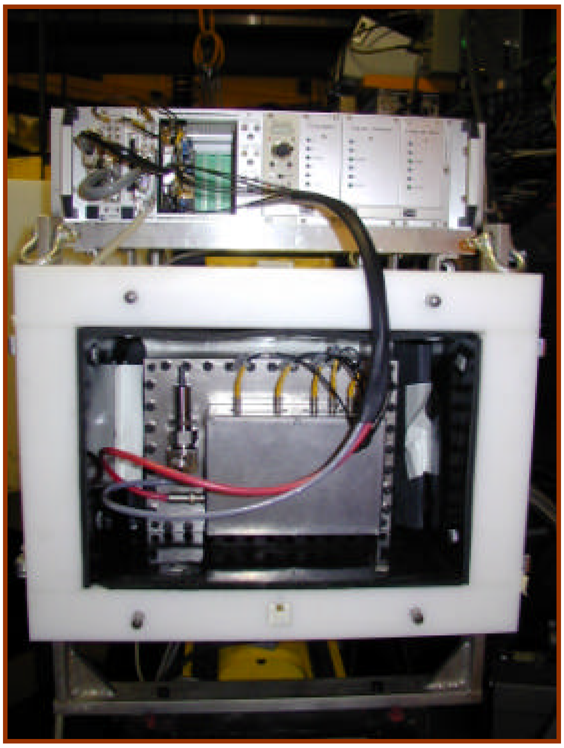

Figure 1. View of 200x100 mm² MSGC detector mounted on 7C2 diffractometer

The electronics is composed of a compact electronic chain including the charge amplification and the charge division calculation, a conversion board with two 12 bits ADC converters, an interface board to communicate with the standard LLB EuroPSD and EuroScaler counting boards.
The speed of the electronics counting rate is limited to 5MHz, but the actual limitation is given by the detector itself (due to the resistance of the charge division line.)
These MSGC can achieve a 1.5-2 mm spatial resolution (see Figure 2). The background noise is 30 counts/min on a 100x200mm^2 detector.

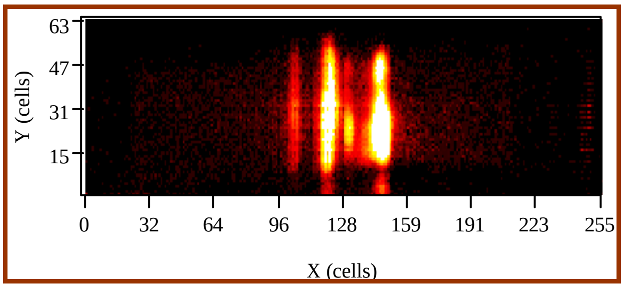

Figure 2. Picture of the reflectivity measured on a grating (right, direct beam; left reflected beams).

The diffractometer 7C2, dedicated to the study of liquids and amorphous systems, is being renewed. The present « banana-type » detector of 7C2 consists of 640 cells covering a scattering angle of 128° and a BF_3 detection gas under atmospheric pressure. It is very stable but has a quite low efficiency (17% at 0.07nm, which is the most commonly used wavelength). This detector will be replaced by a set of 14 2D-micro-strip detectors described above, filled with 15bars of ^{3}He (see figure3).

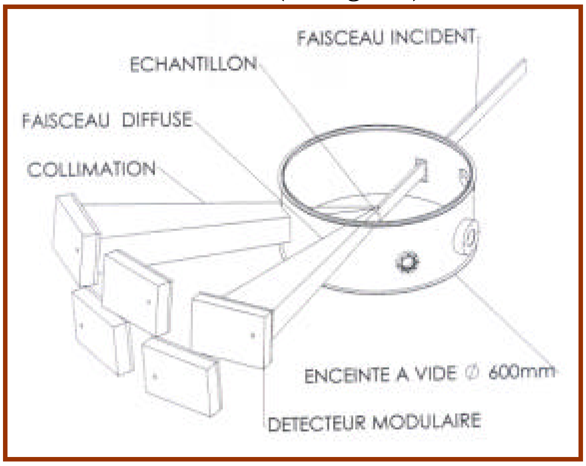

Figure 3. Layout of the 7C2 diffractometer equipped with MSGC detectors.

This high pressure gives a good detection efficiency (~92%) at short wavelengths (0.07nm). Due to the better resolution of such a detector, it can be placed closer to the sample (1m instead of 1.5m), which will result in a larger solid angle for detection. Each detector will have its own collimation system. The detectors will be arranged in such a way that the same angular acceptance as the actual one will be covered. In 2002 one of the micro-strip detector was regularly tested on 7C2, and the collimator for each detection module has been designed and tested. Monte Carlo simulations of the present and the new instruments (using the VITESS program, HMI-Berlin) show that the resolution will be close to the present one.

New spectrometer for nano-objects "TPA":

The very small angle spectrometer **TPA** ('Très Petits Angles') is currently developed on the guide G5bis. Its aim is to cover a q-range from 10^{-4} $Å^{-1}$ up to intermediate angles. It will allow neutron studies of larger objects (≈ 1000 Å), like giant micelles, membranes, biophysical gels, large-scale porosity and precipitation in alloys for metallurgical applications.

The principles of the spectrometer are very close to traditional small-angle machines. In the first design, the collimation is a pinhole-collimation with smaller diaphragms, but more complex collimation devices (lenses, focalization, etc..) are also tested. The detection is 2D. A relatively high pixel definition is necessary for very small-angle scattering, because the maximum length of the spectrometer is limited due to guide hall constraints. The only commercially available detectors with a pixel size smaller than 1 mm are 'image plate' detectors. We have installed a MAR345, with a Fuji plate for neutron-photon conversion and a spatial resolution of 150 microns has been easily achieved. Its major drawback is the high γ-ray sensitivity. The complete instrument is therefore designed in order to minimize background: Monochromatisation is achieved by monochromating $3\theta_c$-mirrors (XENOCS, Grenoble) with 15%-bandwidth. This allows deviating the direct beam and eliminating the γ-ray background from the guide by an appropriate shielding. Moreover, the mirrors replace a mechanical velocity selector, also responsible for a high γ-ray background in the direct beam. The collimators are made of ^{6}Li diaphragms and heavy lead shielding behind the diaphragms, along the neighbouring guide G5 and around the detector is further decreasing the background. Preliminary test runs on the prototype show the feasibility of this spectrometer, and first parts of the final construction plans will be finished these months.

Development of new sample environment

Advanced sample environments have been developed, in particular for **high-pressure neutron diffraction.** In *soft matter and biology*, even modest pressures (<1 GPa) can induce considerable changes in interatomic distances and physical properties. Numerous studies of polymers or proteins (0.3 – 0.4 GPa) have been performed using specialised high-pressure cells (see chapter 4, 5 and 6).

In *solid-state physics*, much higher pressures are generally required to induce significant changes in electronic or structural properties (through *structural* or *valence transitions*, for example).

During the last years, original high-pressure and neutron techniques have been developed at the LLB in order to study magnetic and structural phenomena under very high pressures (>10 GPa) by neutron diffraction. The LLB holds a world record in high-pressure neutron studies: **50 GPa (500 kbar)**. The record measurements were performed on a specialized **high-pressure powder diffractometer "MICRO"** (G6.1) equipped with neutron focusing systems and high pressure cells with sapphire or diamond anvils. On this instrument, very high pressures can be combined with low temperatures (down to 1.4 K). The wide P-T range allowed discovering new physical phenomena, especially in studies of magnetic ordering and phase transitions under pressure. With a new multidetector for "MICRO" currently under tests and a project for replacing of the present G6 guide by a supermirror guide, LLB considers high-pressure studies as a high priority. Recently, ultra-compact pressure cells have been successfully used in combination with a superconducting magnet and a dilution refrigerator. At the single crystal diffractometer 6T2, pressures up to **7 GPa** can be combined with magnetic fields up to **7.5 T** and very low temperatures down to **100 mK** (see Fig.4 on the right).

Figure 4. High-pressure cells with sapphire anvils mounted on a ^{3}He – ^{4}He dilution refrigerator

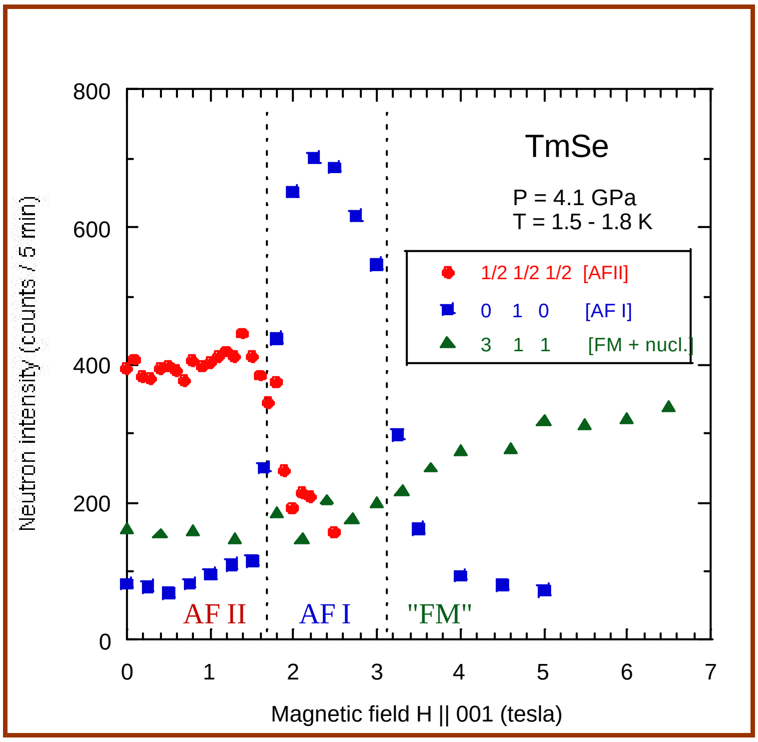

Figure 5

Several examples of neutron diffraction studies curried out under “multi-extreme” conditions can be mentioned, in particular experiments on $CeRhIn_5$ (0.15 K and 0.7 GPa) or a study of the H-P-T magnetic phase diagram of TmSe, shown in Fig.5 above.

Complemented by “medium-pressure” devices (He pressure cell, McWhan-type piston-cylinder cell), the sample environment techniques available at the LLB provide an unprecedented range of thermodynamical parameters for neutron studies.