

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

I. GONCHARENKO, X. GUILLOU

Leon Brillouin Laboratory,CEA-CNRS, CE Saclay, 91191 Gif sur Yvette, France.

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 $<0.1 \text{ mm}^3$ 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 ^{60}Ni 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.

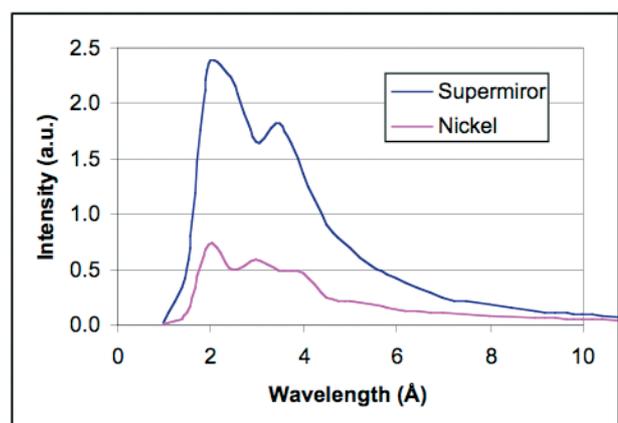


Figure 2. Intensity distribution calculated at the G6-1 monochromator for the two guide coatings : ^{60}Ni (red) and $m=2$ supermirror (blue).

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.

INSTRUMENTATION

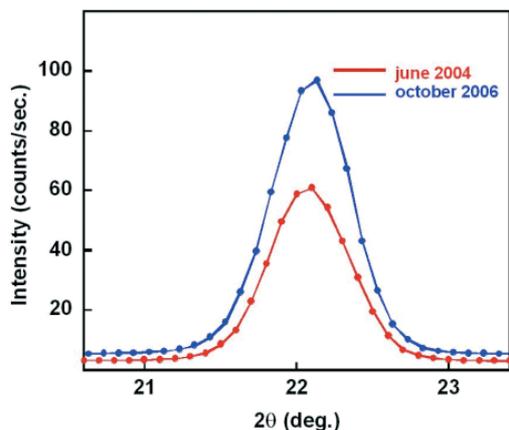


Figure 3. Bragg reflection from zeolite sample before and after the replacement of the guide

MONOCHROMATOR OPTIMIZATION

The current monochromator used on G6-1 is made of 5 focusing blades of pyrolytic 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 $2.3 < \lambda < 5 \text{ \AA}$ (compare to the present $4 < \lambda < 5 \text{ \AA}$). It had been fabricated in June 2006, will be tested by the end of 2006.

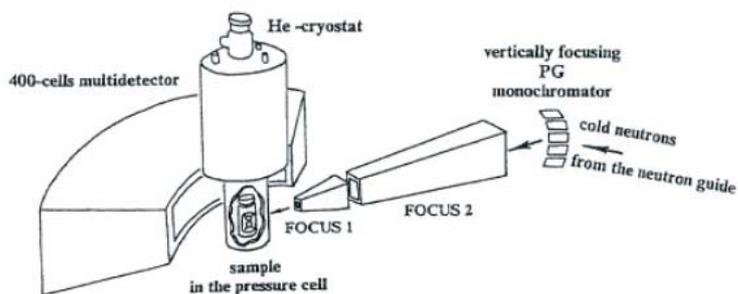


Figure 4. Full setup of the G6-1 instrument

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 $m=3$ supermirror coatings, it was optimized for the divergence delivered by the ^{60}Ni 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 make 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 BF_3 "banana-type" counter) is only 0.1 steradian. Development of new multidetector, having solid angle by order of magnitude larger than 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 ($2.54 \times 102.4 \text{ cm}$) 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.

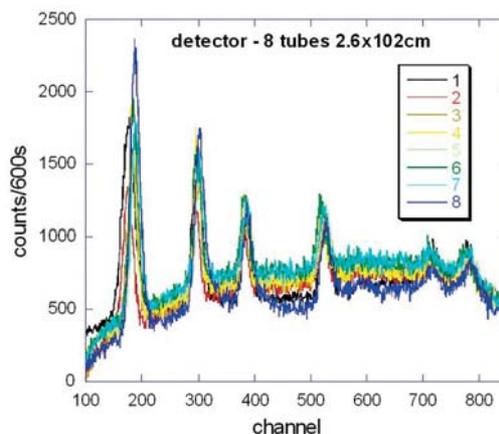
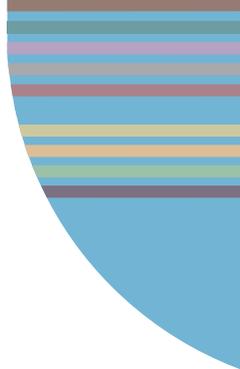


Figure 6. Neutron diffraction patterns collected by every tube of the test assembly at the G6.1.



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 $\pm 25^\circ$. 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.

[1] I. Mirebeau, I. Goncharenko, P. Cadavez-Perez, S.T. Bramwell, M. Gingras, J.S. Garner, *Nature* 420 (2002) 54.

[2] I. Goncharenko, O.L. Makarova, L. Ulivi, *Phys. Rev. Lett.* 93 (2004) 5, 2004 and I. Goncharenko, *Phys. Rev. Lett.* 94 (2005) 20

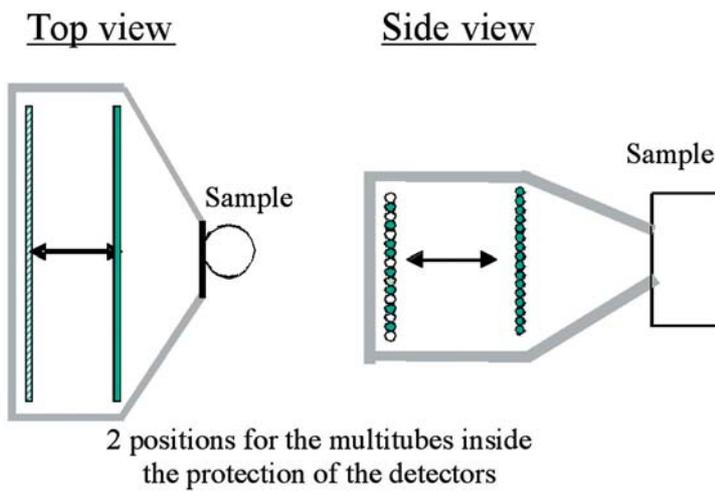


Figure 7. The new 16 tubes detector setup for G6-1.