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The new powder diffractometer D1B of the Institut Laue Langevin

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Abstract. D1B is a medium resolution high flux powder diffractometer located at the Institut Laue Langevin, ILL. D1B a suitable instrument for studying a large variety of polycrystalline materials. D1B runs since 1998 as a CRG (collaborating research group) instrument, being exploited by the CNRS (Centre National de la Recherche Scientifique, France) and CSIC (Consejo Superior de Investigaciones Científicas, Spain). In 2008 the Spanish CRG started an updating program which included a new detector and a radial oscillating collimator (ROC). The detector, which has a sensitive height of 100mm, covers an angular range of 128º. Its 1280 gold wires provide a neutron detection point every 0.1º. The ROC is made of 198 gadolinium-based absorbing collimation blades, regular placed every 0.67º. Here the present characteristics of D1B are reviewed and the different experimental performances will be presented.

1. Introduction

The neutron diffractometer D1B at the Institut Laue Langevin (ILL), was built in the 70´s. It was designed mainly to study magnetic structures; however its characteristics make possible to investigate a vast variety of scientific problems, such as phase transitions by thermo-diffraction experiments, time-resolved or *"in situ"* studies like crystallization experiments, or to perform texture analysis. Since 1998 D1B is a CRG (collaborating research group) instrument, handled by the CNRS (Centre National de la Recherche Scientifique, France) and CSIC (Consejo Superior de Investigaciones Científicas, Spain). In the last years the Spanish CRG has undergone some technological updating of the instrument, mainly a new position sensitive detector (PSD) and a radial oscillating collimator (ROC) have been built, commissioned and now are at the service of all users of D1B

2. Neutron delivery

The thermal curved guide H22 delivers neutrons to three instruments D1B, SALSA and VIVALDI, being D1B the first of them up stream (see Figure 1). The monochomator´s casemate of D1B accommodates two monochromators mounted on a set of motors. Thus, it is possible to change from one to the other in few minutes. Neutrons are delivered to the sample with a take off angle of 44.22º. On one hand, three highly oriented pyrolytic graphite (HOPG) crystals supply a neutron beam of 2.52Å focalized on the sample, with a flux of $5.6x10^6 n/cm^2 s$; on the other a germanium single crystal

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provides a neutron beam of 1.28 Å, however the neutron flux at the sample position is ten times lower. Downstream of the monochromators there is a set of movable graphite filters used after the HOPG monochromator which reduce the second harmonics up to 8/10000.

The already monochromated neutrons pass through an evacuated tube where there are 5 diaphragms which collimate the beam. A set of adjustable horizontal-vertical slits, permit to accurately define the size of the beam on the sample.

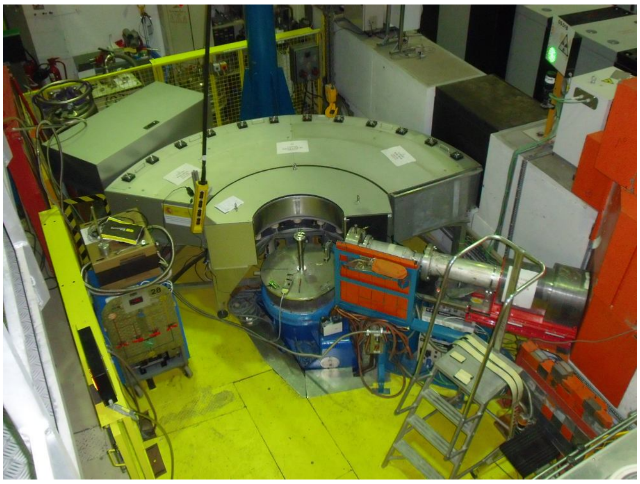

Figure 1.Top view of the D1B neutron diffractometer at ILL.

3. Neutron detection

Since June 2011, it is in operation a new detector at D1B. After more than thirty years working, the old detector of D1B was in a big risk of failure. It was in 2008 when the Spanish part of the CRG D1B decided to start a project for building a new detector for D1B. Thanks to funding from the Ministerio de Ciencia e Innovación (present Ministerio de Economía y Competitividad) in less than three years it has been possible to replace the detector of D1B. The new large PSD for D1B was designed and constructed in a fruitful collaboration between the Spanish CRG and the ILL.

The current detector of D1B covers 0.15 steradians. 1280 W/Re gold plated wires placed every 0.1º inside a sealed gas chamber are the responsible of detecting the neutrons scattered by the sample. The gas filling of 5bars of ^3He and 1bars of CF_4 provides a detection efficiency of 80% at 2.52 Å and 60% at 1.28 Å. The PSD is placed onto a vibration isolated table at 1500mm of the sample position.

In Table 1 the main characteristics of the new and old detectors are detailed. Although the geometry remains the same, the improvements are evident, such as the present detector has incremented the 2θ aperture in 48º or the detection efficiency has increased. Summarizing, under the same conditions, the current detector of D1B allows obtaining a larger diffraction pattern in less time than what was with the old one. One can observe this in Figure 2, where are shown the diffraction patterns of a $Na_2Ca_3Al_2F_{14}$ standard sample mounted on a vanadium cylinder sample holder of 5mm in diameter, measured with both detectors at room temperature. There, it is also observable the neutron dead detection zone the old detector had after an electronic failure occurred in 2008.

Table 1. Main characteristic of the old and current detector of D1B.

	New	Old
angular range	128º	80º
height of the sensitive area	100mm	100mm
angular definition	0.1º	0.2º
readout	1280 cells 1280 anodes encoding	400cells 20 anodes and 20 cathodes encoding
radius of curvature	1500mm	1500mm
efficiency: 2.52 Å 1.28 Å	80% 60%	60% 37%

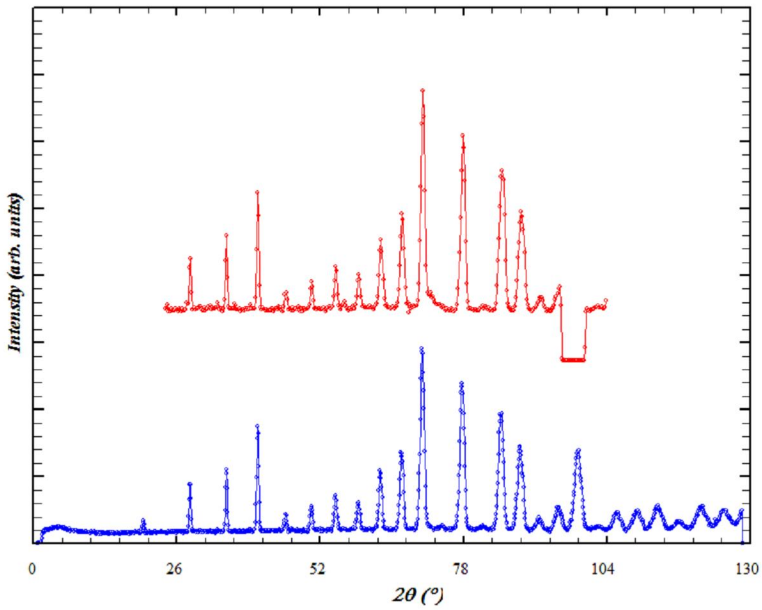

Figure 2. Diffraction pattern at 2.52Å of a ϕ=5mm $Na_2Ca_3Al_2F_{14}$.standard sample. Upper curve: 15minutes acquired with the old detector. Lower curve: 10 minutes acquired with the new detector.

4. Radial oscillating collimator

Many studies accomplished at D1B have the inconvenient that the diffraction peaks coming from the sample are merged with the diffraction arising from the environment. This is the case of cryomagnets, cryofurnaces, or pressure cells. D1B's radial oscillating collimator have been designed and constructed in order to suppress these non-desired scattered neutrons.

The ROC of D1B consist of 198 absorbing collimation blades placed radial in between the sample and the detector every 0.67º. Blades´ dimensions are 350x100x0.1 mm, made of PETP (polyethylene terephthalate) film coated with a neutron absorbing Gd_2O_3 paint. Each blade is under mechanical vertical tension for maintaining the radial structure (see Figure 3). In order to balance the shadows cast by the finite thickness of the absorbing blades, the ROC oscillates at a constant velocity. The oscillation half period is 30s with oscillation amplitude of 6 blade spacing. The dead time of acceleration/deceleration is around 0.5s. The solution adopted at D1B for having such a constant movement is a rotated heart-shape came which transmits the movements tangentially. The ROC has a full width focus of 18.9mm centered on the sample position, i.e. a given point on the detector can therefore see up to 9.4mm to the left and right of the sample's center. In this way neutrons scattered from materials placed beyond will be absorbed by the blades. Unfortunately, some neutrons diffracted from the sample are also blocked by the ROC. For a cylinder sample of 5mm in diameter, 86% of the neutrons scattered by it will arrive to the detector; for a thicker sample of 8mm 78% of neutrons will be transmitted.

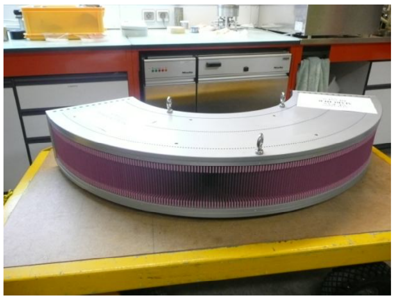

Figure 3. D1B´s ROC radial absorbing blades.

The advantage of using such a device can be observable in Figures 4 y 5. The former illustrates the diffraction pattern of $Na_2Ca_3Al_2F_{14}$.standard sample inside a cryomagnet, where one can hardly recognize the characteristic peaks of this material. The latter shows what it is obtained when the ROC is in place, there all neutrons scattered by the environment have been absorbed. As mentioned above some neutrons diffracted by the sample are absorbed by the ROC, this in principle would increase the acquisition time, however this lost of neutrons arising from the sample are compensated, in most of the cases, with the reduction of background that the ROC causes.

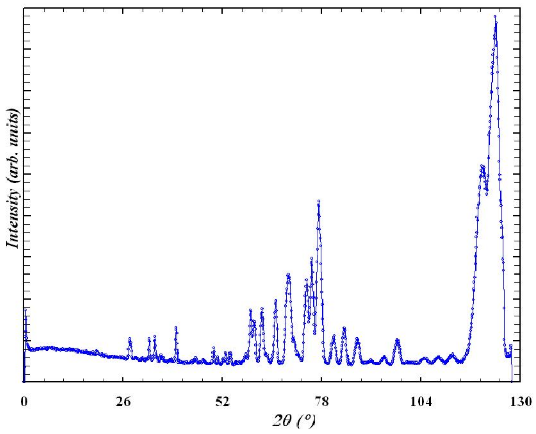

Figure 4 Diffraction pattern at 2.52Å of a ϕ=5mm $Na_2Ca_3Al_2F_{14}$.standard sample measure inside a cryomagnet.

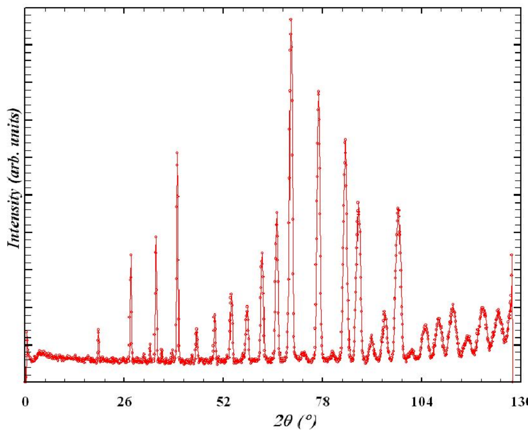

Figure 5. Diffraction pattern at 2.52Å of a ϕ=5mm $Na_2Ca_3Al_2F_{14}$.standard sample measure inside a cryomagnet with the ROC in place.

5. Sample environment

D1B has three dedicated sample environments, an Orange cryostat (2K<T<300K) with a vanadium tail of ϕ=24mm, a furnace (T<1000ºC) with a vanadium resistor ϕ=50mm and a 4-circle Eulerian cradle goniometer for studying textured samples at room temperature.

Moreover, it is also feasible to use the sample environments from the ILL- “pool” like furnaces (T<1600ªC), cryofurnaces (2K<T<550K), cryomagnet (up to 5T), gas and clamp pressure cells or the Paris-Edimburg pressure cell. Recently has been also tested the feasibility of using containerless sample environment under acoustic levitation, with very promising results.

All sample environments at D1B are mounted in a X-Y translation platform (ϕ=700mm) which is on a 360º ω-axis. This makes possible to place in the neutron beam several set-ups.

6. Data acquisition and instrument control

From a standard PC computer running in LINUX, sample conditions and detector acquisitions are controlled. Two programs are offered for instrument control, a command line based: MAD and NOMAD based in a graphical user interface. In addition, a WINDOWS and LINUX workstations are at disposal of the user for reducing and analyzing of D1B´s data, where most used programs like LAMP [1,2] or the FullProf suite [3] can be found.

7. Conclusions

The present performance of D1B, the new detector and the ROC, permits now to face scientific problems which before had to be resolved in other instruments. The increment of the angular definition and the efficiency in neutron detection of the new detector permit to lower the acquisition time of a diffraction pattern in comparison with the old one. These improvements implemented together with the use of the radial oscillating collimator open the possibility of using a vast set of sample environments (cryofurnaces, cryomagnets, etc.) which has a big impact in the feasibility of experiments at D1B.

The ideal situation of D1B in the guide hall of the ILL (high flux and low background) and the upgrades described in this work have pushed D1B to continue being a highly demanded powder diffractometer with a high rate of publications.

Acknowledgments

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[1] Richard, D.; Ferrand, M.; Kearley, G.J. J. Neutron Research 1996, 4, 33-39.
[2] LAMP, the Large Array Manipulation Program. http://www.ill.fr/data_treat/lamp/lamp.html.
[3] Rodríguez-Carvajal, J. *Physica B* **1993**, *55*, 192. The programs of the FullProf Suite and their corresponding documentation can be obtained from the Web at http://www.ill.eu/sites/fullprof/