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Aurora—A high-resolution powder diffractometer installed on the IEA-R1 research reactor at IPEN-CNEN/SP

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ABSTRACT

A high-resolution powder diffractometer (HRPD) was installed on the IEA-R1 research reactor at IPEN-CNEN/SP, São Paulo, SP, Brazil. It is an extensive upgrade of the old IPEN-CNEN/SP multipurpose neutron diffractometer which used a single boron trifluoride (BF_3) detector and a flat mosaic single crystal copper monochromator. The main modifications introduced were the installation of a position sensitive detector (PSD) and a focusing silicon monochromator. Placed at a distance of 1600 mm from sample, the PSD spans an angular range of 20° of a diffraction pattern. An extensive powder diffraction pattern can be obtained by collecting data in contiguous 20° segments. The double-focusing perfect single crystal silicon monochromator, installed in a take-off angle of 84°, can be positioned to produce 4 different wavelengths, namely 1.111, 1.399, 1.667 and 2.191 Å (nominal values). Diffraction patterns, obtained with the instrument, have shown a quite good resolution. Curves *full width at half maximum (FWHM) vs. scattering angle* (2θ) have been measured with powdered alumina using three vanadium sample holders 1/8, 1/4 and 3/8 in. in diameter. Such curves give the resolution that can be expected from the instrument in an interval 2θ from 5° to 130°. In comparison to the former instrument, the new diffractometer has much better resolution and is, per intensity point, ca. 600 times faster in data acquisition.

1. Introduction

A position sensitive detector (PSD) neutron powder diffractometer was constructed and installed at the IEA-R1 research reactor. The reactor operates at 3.5 MW thermal with the possible maximum power of 5 MW. The new instrument, Aurora, is an extensive upgrade of the old IPEN-CNEN/SP multipurpose neutron diffractometer. The old diffractometer constructed in the middle of the sixties, was also called Aurora. It was a single-detector instrument with a boron trifluoride (BF_3) detector and a flat mosaic single crystal copper monochromator. All collimators were Soller-type. Such a configuration coupled to the low power of the reactor led to a monochromatic beam of low intensity. Furthermore, due to the single detector, the 2θ scans were carried out in a point-to-point basis. Consequently, it could require several hundred hours of reactor time to measure a diffraction pattern. In the new diffractometer, installation of a PSD (at a distance of 1600 mm from sample) allows the measurement of intensity points in a 2θ interval of 20° all at once. Then, an extended pattern is obtained by measuring several 20° segments. Depending on the extension of the pattern (2θ range: 5–130°) and time required to measure each segment, a complete pattern with good statistics can be obtained in a matter of few tens of hours. The focusing monochromator and open collimators (without plates) produces a high-intensity monochromatic beam on the sample—another reason for a quick data acquisition.

The PSD neutron diffractometer Aurora has several new components. All those components belonging to the detector system, including its electronics, computer and basic software for control and data acquisition, were acquired from Instrumentation Associates (IA). IA has also furnished the focusing monochromator as well as a rotating-oscillating collimator (ROC), the latter an essential component of the new instrument. Other components were designed and constructed at IPEN, e.g. a main neutron shield, a PSD shield and a beam shutter. In what follows, a brief description of the essential elements of the new instrument is presented.

2. The position sensitive detector

The position sensitive detector (PSD) [1–3] consists of eleven linear position-sensitive detector elements, clamped together at each end to form a rigid plane. Each linear PSD element is a

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proportional counter manufactured by Reuter-Stokes Inc. The 25 mm diameter stainless steel detector elements have a wall thickness of 0.25 mm and an active length of 610 mm. The anode wire is nickel chrome with a diameter of 0.015 mm and specific resistance ca. 8000 Ω. The counter gas fill is 8 atm of ^{3}He, for neutron detection, and 4 atm of Ar, for stopping the reaction products (with 0.5% of CO_2 for quenching) [4]. Fig. 1 is a representation for the equivalent circuit of the detector-preamplifier assembly for a single linear position sensitive detector element. In operation, each end of a detector element is connected to a charge-sensitive preamplifier and the detector anode is maintained at a bias of 2000 V. A thermal neutron entering the detector element at an x-position from the A end of a detector element, if captured by a ^{3}He atom in the fill gas, produces energetic ^{1}H and ^{3}H reaction products that ionize the gas. Electrons created in the detector fill gas are drawn to the detector anode, injecting (with gas multiplication) a charge pulse on the detector anode. The positive ions are drawn to the detector wall. The signal in the anode propagates to each end of the detector element, is amplified by the preamplifiers and is passed to position encoding modules (PEMs). With this configuration, the linear detector element becomes a position sensitive detector.

The charge injected at an x-position along a linear detector divides into two currents that flow to the left and right through the anode resistance and the input impedances of the two preamplifiers coupled to the ends of the linear detector. The PEMs contain two computer controlled pulse shaping amplifiers, a lower level discriminator and two high speed 16-bit ADCs. Signals that fall within the discriminator window cause the ADCs to simultaneously capture and digitize the pulses from each end of the detector element. The outputs of the ADCs are then used to calculate the neutron capture event position (x). The PEMs maintain the event position and amplitude histograms which are transmitted to the host computer by USB on demand [3].

Fig. 2 is a photograph of the array of eleven linear PSD elements that forms the PSD of the new instrument. Close to the PSD is a junction box that distributes the signals to the PEMs, and connects the preamplifier power supply, electronic alignment tail pulser and high voltage bias supply to the detector assembly [3]. One of the eleven PEMs of the system (one for each linear detector) is shown on the left side of the photograph. The PSD is installed inside the PSD Shield at a distance of 1600 mm from sample.

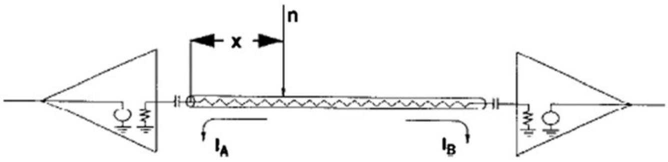

Fig. 1. Equivalent circuit representation of the detector-preamplifier assembly for a single linear position sensitive detector element.

Fig. 2. The neutron PSD array, the junction box and one of the linear PSD signal processing electronics elements (PEMs).

3. The focusing silicon monochromator

In order to increase the neutron beam flux at the sample position, a double focusing silicon monochromator was installed [5,6]. It is composed of 9 vertically stacked silicon blades each approximately 5 mm thick, 14 mm high, 190 mm long, mechanically bent in the horizontal plane to obtain focusing in Q-space and mounted on a polygonal approximation to a sphere to achieve focusing in the vertical plane. At a take-off angle of 84°, four different wavelengths can be obtained from reflections in the [0 1 1] zone: 533/1.111 Å, 511/1.399 Å, 331/1.667 Å and 311/2.191 Å. Switching between 533, 511 and 311 reflections is achieved by rotating the crystal around the vertical [0 1 –1] zone axis. Switching to the 331 reflection requires flipping the monochromator bottom up. The 511/1.399 Å reflection takes advantage of beam compression due to a Fankuchen cut of the crystal, giving rise to a more intense monochromatic beam on sample.

Fig. 3 is a photograph of the double focusing silicon monochromator supported by the goniometer used for orientation in the polychromatic neutron beam. The goniometer, used for many

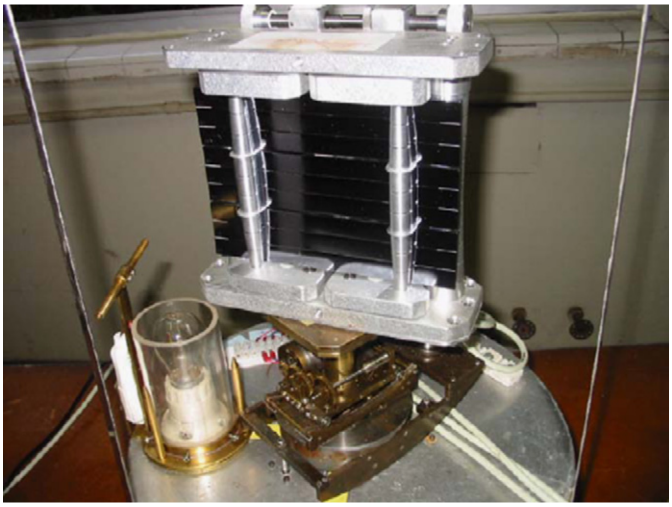

Fig. 3. Close-up of the focusing monochromator, goniometer and luminaire.

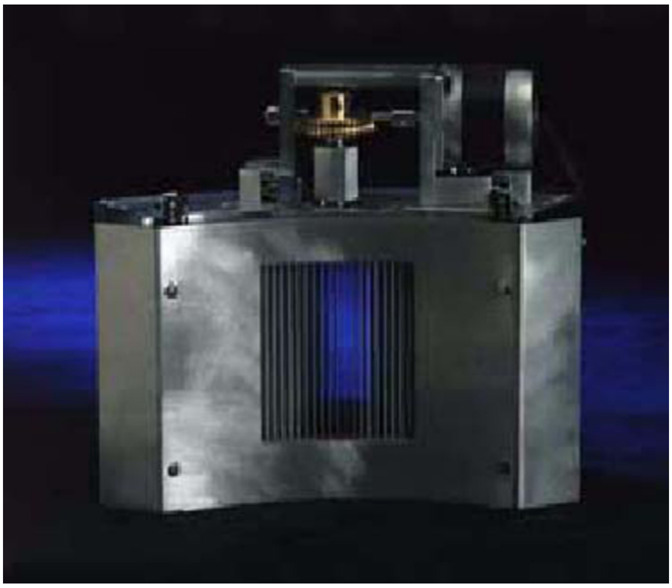

Fig. 4. The rotating-oscillating collimator (ROC).

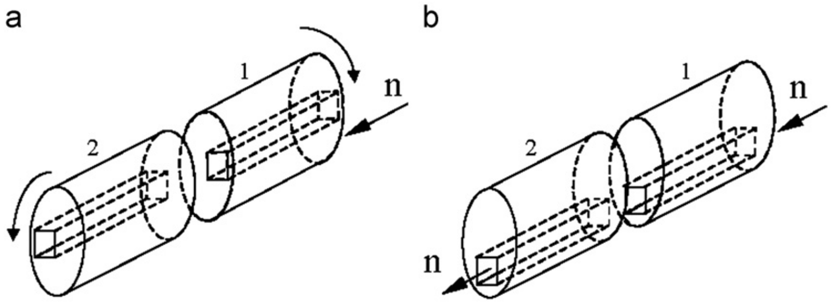

Fig. 5. Schematic drawing of the beam shutter in two different situations: on the left (a), channels are in opposite positions corresponding to the beam off condition; on the right (b), the channels are aligned corresponding to beam on condition.

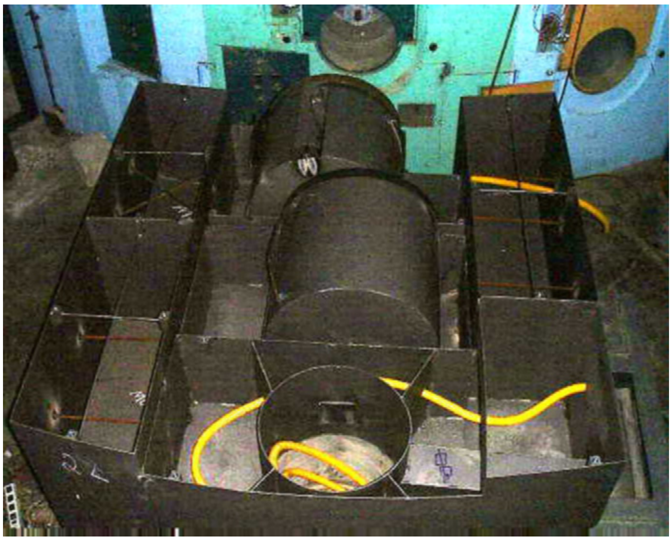

Fig. 6. Photograph of the beam shutter took during its installation in the second layer of the main neutron shield.

years in the old diffractometer, was slightly modified to support the new monochromator. A small luminaire is also shown in Fig. 3. It serves to illuminate the ensemble to check for malfunction. It can be removed/replaced through the opening where the monochromatic-beam collimator is inserted. An aluminum cylinder, filled with a mixture of paraffin+boric acid, supports the monochromator–goniometer ensemble providing additional shielding for the bottom of the cylindrical hole where it is installed (see Fig. 6). A similar cylinder, although several times longer than that shown in the photograph, closes the top of the hole completing the shielding.

The goniometer has three movements: *rocking* around a vertical axis, *tilting* around a horizontal axis, *translation* along an axis normal to the monochromator surface. All movements are remote-controlled via torque transmitters electrically coupled to torque receivers installed in the goniometer. When the old diffractometer was built (~1965) and stepper motors were not yet available, these devices – also called selsyns (self-synchronizing transmitters/receivers) – were commonly employed in instrumentation. The three movements are remote-controlled: DC motors drive the motions of the selsyn transmitters in both directions through suitable gear reductions. Together with the gears, a disk with a cam in its periphery allows a step by step movement by acting on a microswitch coupled to the DC motor. A continuous movement is also a mode of operation of the device. Mechanical counters, installed on the front of the panel of each module, indicate the relative position of the movements.

4. The PSD shield

To reduce the ambient background, a shield for the PSD was constructed at IPEN. It has the form of a truncated pyramid coupled with a box in its basis. The PSD is installed inside the box. The shield is made of double-walled high-density polyethylene (HDP) panels. Neutron absorption is provided by a mixture of paraffin+boric acid (approximately equal parts in volume) filling the panels. Additional shielding panels (25 mm plates of HDP with 5% boron) cover the outside of the assembly. The inner surface of the shield is painted with a neutron absorbent paint prepared by dissolving a very fine powder of gadolinium oxide in varnish (ca. 60% Gd_2O_5+40% varnish, volumetric). Three coatings of this paint became necessary to obtain a uniform layer of gadolinium oxide. The PSD shield installed in the diffractometer can be seen at the right side of Fig. 9(a).

5. The rotating-oscillating collimator

A rotating-oscillating collimator (ROC), placed at the entrance to the PSD shield, eliminates parasitic scattering from furnace or cryorefrigerator heat shields in the vicinity of the sample, while reducing the scattered intensity by ca. 10%. The ROC also makes the PSD less sensitive to ambient background leaking in through the shielding entrance [7].

Fig. 4 is a photograph of the ROC. In the upper part of the ROC, a stepper motor is coupled to a worm gear that makes the central region of the collimator assembly oscillate over a small angular range. The stepper motor is operated by a driver, designed and constructed at IPEN. Reversion is provoked by two micro-switches placed at both ends of the movement. Frequency of oscillation can be changed adequately. In general, the frequency is set to one complete oscillation in 2 min. Oscillation is required to average over small mechanical differences in the angular aperture of the collimator blades and eliminate the neutron shadow of the blades on the detector plane.

6. Other collimators

Two additional collimators were constructed for the instrument: the in-pile and the monochromatic-beam collimators [8]. Both are open collimators, i.e. they have no plates. A brief description of them is given below.

The in-pile collimator is used to guide polychromatic neutrons towards the monochromator. It is separated into three parts. A longer tube is its body which is inserted into the beam tube (BH-6) of the reactor. The body allows the beam tube to be flooded with water to avoid the “radiation ring” normally formed around the collimator exit end. A shorter tube with a “cage” in the middle is the collimator itself. It is inserted into the body for normal operation of the instrument. In the in-pile collimator a central $70\times70\,mm^2$

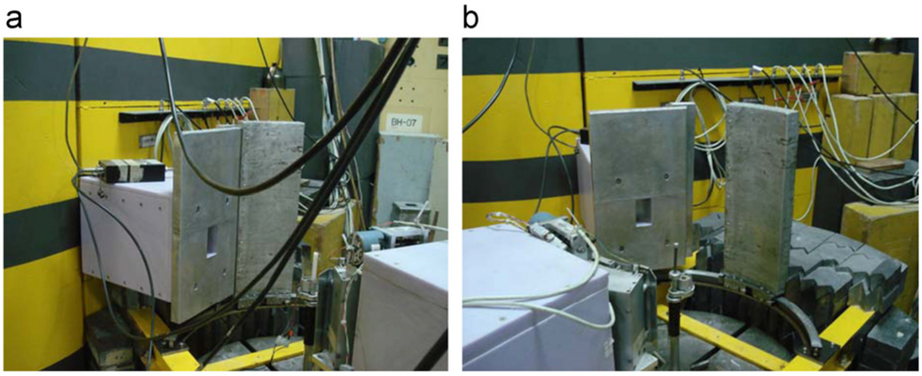

Fig. 7. The extra shield for the PSD in two positions: in a low (a) and a high 2θ (b).

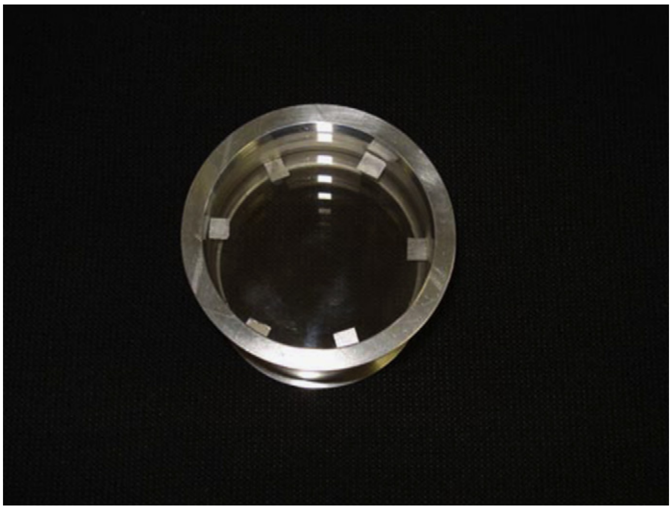

Fig. 8. The three sapphire windows inserted into the centralizing support.

square tube collimates the neutrons. The square tube is divided into two parts separated by the “cage”. Around these two parts the collimator is filled with barite cement for neutron shielding. The “cage” accommodates a sapphire filter to reduce fast neutron contamination from the polychromatic beam (the sapphire filter will be described later on). Another shorter tube filled with barite cement serves as a plug to block radiation during maintenance of the diffractometer. It is inserted into the body instead of the collimator.

The monochromatic-beam collimator is formed by an inner duct with a pyramidal form. Such duct has appropriate dimensions to allow focusing of the monochromatic beam on the sample. The duct is placed inside a body in a central position. Barite cement fills the body. The monochromatic-beam collimator is placed after the monochromator at a take-off angle of 84° from the polychromatic beam. It is inserted into the main neutron shield (described somewhere below). The exit end of the collimator has been surrounded by a neutron shielding made of 25 mm plates of HDP with 5% boron. In order to obtain a more effective neutron shielding, two plates were joined together to form a thicker wall. A low-efficiency fission chamber used as a neutron monitor is placed inside the shielding, close to the beam exit. The additional shielding can be seen in Fig. 7(a).

7. The beam shutter

The polychromatic neutron beam can be shut out by action of a beam shutter. Its function is to protect operators during sample manipulation or installation of any device in the sample position. It has been designed and constructed at IPEN and installed in the main neutron shield. Fig. 5 is a schematic drawing of the beam shutter showing it in both *beam off* and *beam on* conditions (a) and (b), respectively.

The beam shutter is formed by two 500 mm diameter × 500 mm length contrarotating drums, both with 92 × 92 mm^2 peripheral square channels. The drums are filled with barite cement and coupled each other by a pair of identical gears in order to make the drums rotate towards opposite directions. Both drums are supported by pairs of ball bearings. When the channels are aligned they are in the right position to allow the neutron beam, coming from the reactor, to pass and reach the neutron monochromator. Owing to the geared coupling of the drums, moving one of them towards a certain direction the other moves to the opposite direction. Consequently, the channels go to opposite positions shutting the passage of neutrons off. The shutter is driven by an 180 VDC/0.11 A electric motor provided with a 1100:1 reduction gearbox. A metallic gear attached to the drive shaft of the motor is coupled to a toothed rubber strap, fixed on the cylindrical surface of one of the drums. They form a sort of rack and pinion coupling. Movement and positioning of the shutter is controlled by an electronic control module [9], also designed and constructed at IPEN. The front panel of the electronic module has three colored push buttons (*red*, *black* and *green*) that command movement and positioning of the shutter. Its position is indicated by three lamps on a pedestal. Lamps are colored *red*, *yellow* and *green* for, respectively, *beam on*, *intermediary position* and *beam off*. The pedestal is placed on the top of the main neutron shield, for maximum visibility. When the shutter is moving, two lamps in the pedestal remain lighted till the movement is completed. For example, if the shutter is in the *beam on* and the green button is pressed to get the *beam off* condition, both *red* and *yellow* lamps remain lighted during the movement till it stops. When the end is reached, both *red* and *yellow* lamps are turned off and the *green* one is turned on. The *black* button, if pressed, interrupts the movement till the *green* or *red* is pressed to resume or change it.

Fig. 6 is a photograph of the beam shutter during its installation in the second layer of the main neutron shield. In Fig. 6, the boxes forming the layer are still empty. The yellow tubes are conduits that remain immersed in the barite cement allowing passage of the electric wires for control of the monochromator (front) and shutter (back). After the shutter installation, both drums and boxes were filled with barite cement for neutron shielding. The cylindrical hole in the frontal part of the shield is the place where the monochromator–goniometer ensemble has been installed after conclusion of the shield construction.

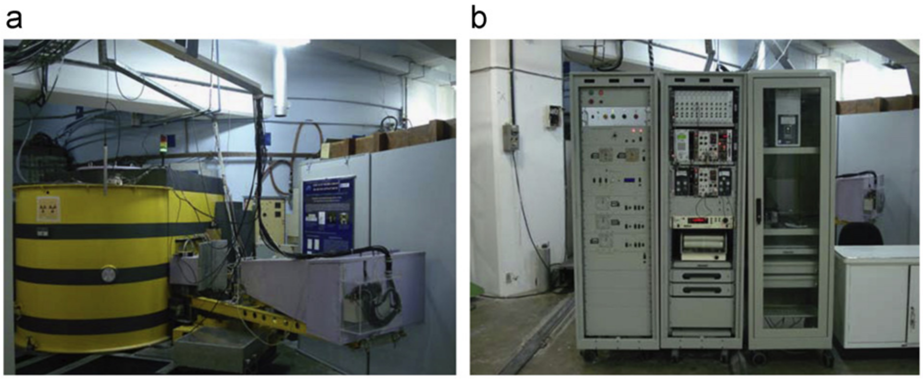

Fig. 9. The Aurora HRPD (a) and the three racks with the associated electronics and control modules used in control and data acquisition (b).

8. An extra shielding for the PSD

During a pattern measurement, in each 2θ position of the supporting arms, the PSD also detects those neutrons that are "seen" by the PSD together with the neutrons scattered by the sample itself. Such neutrons emerge from those regions that are directly opposite to the sample and PSD. In general, they are originated from failures in the neutron shields existing in the regions. Neutron scattering in the walls or other parts belonging to such regions is another origin for the neutrons. According to our experience with the new instrument, in spite of the presence of the ROC, the resulting background level due to these neutrons is high and, worst, irregular. In fact, it assumes the more different shapes for each measured 20° segment with a poor agreement between extremities of consecutive segments. In order to diminish the deleterious effects of this kind of background on the quality of the patterns, a neutron shield has been designed and constructed to provide an extra shielding for the PSD. It is placed in an opposite position to the ROC having in the center the sample. It is allowed to dislocate on a semicircular rail fixed on the supporting arms and centered on the sample. Below approximately $2\theta=33^{\circ}$, the shield collides with the massive exit end of the monochromatic-beam collimator. This was purposely done in order to avoid shutting the monochromatic beam off for a 2θ lower than ca. 21°. In its movement towards a lower 2θ, the shield remains in the central position of the rail till it encounters the front plate of the collimator. After this point the shield is allowed to dislocate on the rail to the right remaining in contact with the front plate. Two pairs of rare-earth strong magnets (small cylinders 10 mm diameter × 10 mm height), installed in both front plate and movable shield, maintain a firm contact between them no matter the direction is assumed for the supporting arms. Clearly the two magnets of a pair are installed in such a way that opposite magnetic poles face each other. When returning to a higher 2θ the shield remains in contact with the front plate till it reaches the central position. In such occasion a stopper attached to the rail forces it to separate from the front plate. Another (single) magnet of same kind maintains the shield firmly anchored to the stopper avoiding any accidental displacement to the right that would remove it out from the central position.

Fig. 7 is composed by two photographs of the extra shield in two different positions related to the monochromatic-beam collimator. In photograph (a) the extra shield is in contact with the front plate of the collimator. Note that the shield is dislocated to the right a certain distance from the center of the rail and from the stopper (barely seen in the photograph). The angular position is $2\theta\sim30^{\circ}$. In photograph (b) the extra shield is in its normal central position, anchored to the stopper. The angular position in this case is $2\theta\sim40^{\circ}$.

9. A fast neutron filter

The beam tube where the PSD diffractometer is installed is directed towards the reactor core. This implies in a high level of gamma and fast neutron radiation accompanying the thermal neutrons of interest in neutron diffractometry. In general, gamma rays are not a problem but fast neutrons create a large environmental background that inevitably increases the background level of the measured patterns (unless a very high efficient shield for the PSD has been installed). It should be understood fast neutrons also contaminate the monochromatic beam. In order to cut such neutrons from the ambient as well as from the monochromatic beam, we have installed a sapphire filter approximately in the middle of the in-pile collimator, in the "cage" constructed for this purpose.

According to the literature, many different materials can be used as filters for neutrons with the more different purposes [10–12]. To cut fast neutrons, a sapphire crystal is one of the best choices since it does not need to be refrigerated to have a quite high transmission of thermal neutrons [13]. More recently, Stamatelatos and Messoloras [14] have published a paper concerned with the optimization of a sapphire filter to be used in neutron diffraction measurements. According to these authors, a 75 mm thick sapphire crystal is a good choice though one of 150 mm would be better concerning the reduction of the fast neutrons transmission. However, such larger thickness also reduces considerably the thermal-neutron flux in the monochromatic beam. The authors pointed out that the advantages of using a thinner sapphire filter are: a higher thermal-neutron transmission, a lower neutron background at the vicinity of the instrument and a lower cost. It should be noted that the study was done taking as model a reactor and a generic neutron instrument very similar to our reactor and diffractometer. For such reasons, we have assumed a 75 mm thick filter as the better choice for a sapphire filter to be installed in the Aurora diffractometer. Taking into account the dimensions of the polychromatic beam as well as of the channels in the beam shutter we ordered (Crystran Ltd., UK) a sapphire filter formed by three pieces. Description of each piece is the following: *Sapphire window* (*optical quality*); *orientation C plane* ±0.5°; 100 mm *dia.* ±0.1 × 25.4 mm *thick* ±0.1; *polished both faces* 80/50; *chamfered both faces*.

Fig. 8 is a photograph of the three pieces of sapphire together and inserted into a cylindrical support made of aluminum. The three pieces are inserted into the support in order to maintain them centered when installed in the in-pile collimator.

10. The main neutron shield

In order to avoid creation of a large ambient background in the diffractometer, a massive shield was designed and constructed at

IPEN. This main neutron shield accommodates the beam shutter, the focusing silicon monochromator and the incident-beam collimator. On the top of the shield is located the pedestal that indicates the beam shutter status.

The main neutron shield is composed by several boxes. The boxes are made of 1/8 in. steel plates and filled with barite cement in order to shield the radiation. They are different in size and shape, in such a way that when joined together they do not allow channeling of radiation. Three horizontal layers form the shield each one formed by several boxes. Beam shutter, focusing Si monochromator and monochromatic-beam collimator, all three are installed in the central layer of the shield. The main shield of the old diffractometer is now used as an additional shield to the new one. It is placed in front of the new shield. Both are supported by movable platforms to allow access to the beam port in the reactor wall.

Fig. 9(a) is a photograph of the new diffractometer installed in the "beam-hole" no. 6 (BH-6) of the IEA-R1 reactor. In the photograph, the two shields, the old and the new, are seen together. Supported by the platform of the new shield is the spectrometer. It is formed by several parts: the PSD shield (with the PSD installed inside), the ROC (installed at the entrance of the PSD shield) and the extra shield for the PSD. The assembly is sustained by two 4 in. steel I beams fixed to a large 25 in. dia. rotary table that provides the instrument with the angular movement 2θ. A smaller 9 in. dia. rotary table, placed underneath and concentric with the larger one, provides the instrument with an $\omega(\theta)$ sample rotation (if required). This small rotary table is not visible in the figure. Both tables are driven by computer controlled stepper motors. The box containing the stepper motors and the rotation motion gears for the 2θ and $\omega(\theta)$ movements is also seen in Fig. 9(a). The spectrometer is counterbalanced by numerous lead bricks placed on the opposite side of the steel I beams. In Fig. 9(b) the three racks with the associated electronics and control modules, used for control and data acquisition in the instrument, are seen.

11. Calibration of the PSD

In order to transform the raw spectrum obtained with the PSD from *position channel vs. neutron counts* to *scattering angle* (2θ) *vs. scattered intensity* an absolute position calibration of the detector is required [3]. Calibration is obtained using a slotted absorbent mask which is inserted into a narrow opening at the top of the PSD shield. The mask used in the new instrument is made of aluminum with one of its surface covered with the same gadolinium oxide neutron absorbing paint used in other parts. Thirteen coatings were required to obtain a uniform layer about 1 mm thick. The mask has equally spaced and equally dimensioned vertical slots that allow neutrons to pass and illuminate uniformly the eleven linear PSD elements. The painted surface is placed directly in front of the detector elements. To calibrate the PSD, neutrons are scattered incoherently by a plastic rod (in general a small cylinder 10 mm dia. × 60 mm height made of HDP) placed in the sample position. Scattered neutrons, traversing the slots and reaching the linear detectors, give rise to the formation of a series of sharp peaks each one corresponding to one slot. All peaks are fit by Gaussians to determine its central electronic channel. As the absolute position (in cm) with respect to the detector center is known for each of the elements, a calibration plot can be drawn and fit to a straight line. Piecewise linear interpolation between the data points with extrapolation at the ends is used to obtain a more accurate calibration than would be provided by the simpler linear fit. The mask can be seen in the photograph of Fig. 10 still with the tapes used during painting with the gadolinium oxide paint.

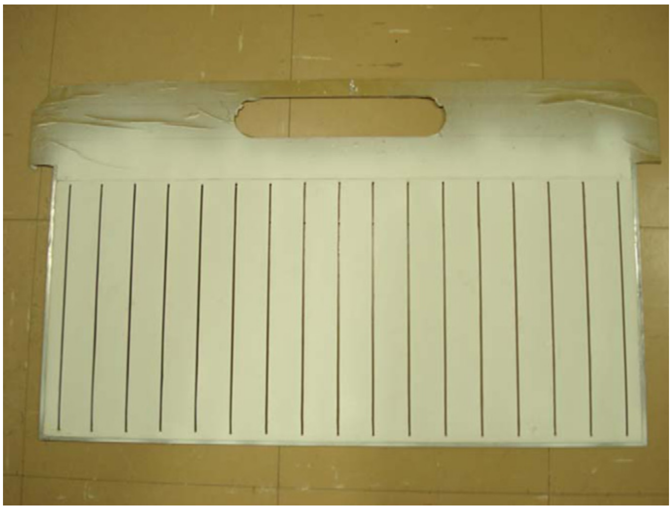

Fig. 10. The slotted absorbent mask used in the 2θ calibration of the Aurora HRPD.

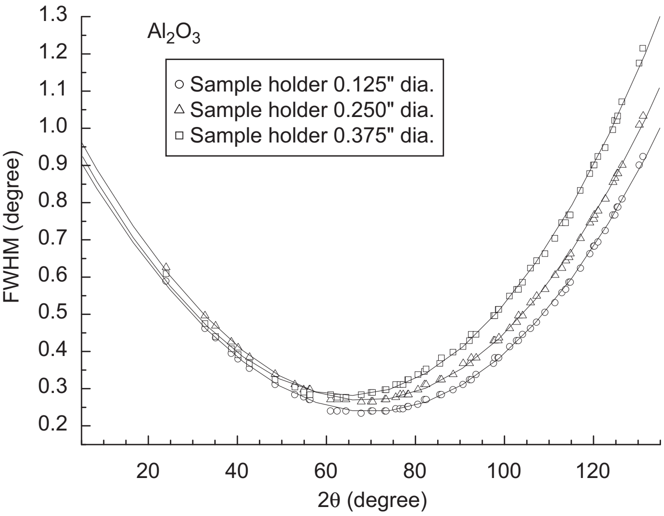

Fig. 11. Resolution curves full width at half maximum (FWHM) vs. scattering angle (2θ) for the Aurora HRPD obtained with alumina (Al_2O_3) in three different sample holders.

12. Results obtained with the new instrument

Results presented below were obtained with the new instrument after calibrations of the 2θ scale and wavelength. The 2θ scale calibration was carried out using the procedure above described. On the other hand, the wavelength calibration was carried out by first measuring a pattern of a silicon standard sample. A vanadium sample holder (0.005 in. wall thickness) with dimensions 0.25 in. internal diameter (i.d.) × 2.6 in. long was used. Assuming the cubic parameter for Si, at room temperature, fixed to 5.431021 Å the Rietveld program GSAS [15] was used to obtain the value for the monochromatic beam wavelength. It resulted $\lambda = 1.4119$ Å. This wavelength corresponds to reflection 511 from the monochromator.

The three resolution curves in Fig. 11 were obtained by measuring powder diffraction patterns with a standard powder sample of alumina (Al_2O_3). Three different vanadium sample holders were used in the measurements. They had the following dimensions: 0.125 in. and 0.250 in. i.d., both 2.6 in. long;

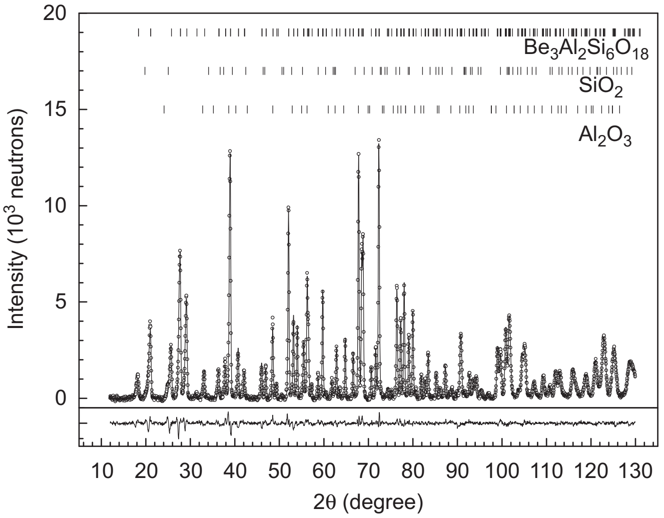

Fig. 12. Neutron diffraction pattern obtained with a mineral sample of $Be_3Al_2Si_6O_{18}$ (Beryl) in the Aurora HRPD. A Rietveld analysis of the pattern has shown presence of two other phases SiO_2 and Al_2O_3.

0.375 in. i.d. × 2.0 in. long. Such sample holders are currently used in the new diffractometer. Experimental points in the curves correspond to the full width at half maximum (FWHM) obtained from the Al_2O_3 patterns after a refinement done with program GSAS.

Pattern of Fig. 12 was measured in the Aurora HRPD. Sample for the measurement was obtained by pulverizing a natural crystal of Beryl. A 0.250 in. i.d. × 2.25 in. long sample holder was used. The entire pattern is formed by six 20° segments. Each segment took about 8 h to be measured (actually, the measurement was carried out by using the neutron monitor). A Rietveld analysis of the pattern, employing program GSAS, showed presence of $Be_3Al_2Si_6O_{18}$ (Beryl) and two other phases SiO_2 and Al_2O_3, respectively, 96.9, 2.7 and 0.4% weight fraction. Two of the most common numerical criteria of fit [16], given in the output of GSAS, *R-pattern* (R_p) and *R-weighted pattern* (R_{wp}) resulted 0.022 and 0.032, respectively.

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References

[1] R. Berliner, D.F.R. Mildner, O.A. Pringle, J.S. King, Nucl. Instr. and Meth. 185 (1981) 481.
[2] C.W. Tompson, D.F.R. Mildner, M. Mehregany, J. Sudol, R. Berliner, W.B. Yelon, J. Appl. Cryst. 17 (1984) 385–394.
[3] R. Berliner, Notes and Instructions Multi-Element Position Sensitive Neutron Detector. Instrumentation Associates, 2 Davis Dr., Research Triangle Park, NC 27709, December 17, 2006 (private communication).
[4] R. Berliner, D. Charlton, W.B. Yelon, A.D. Krawitz, R.A. Winholtz, C. Bjorn Hauback, Helmer Fjellfaag, O. Steinsvoll, Hardware and software system design for high resolution linear position sensitive proportional counter detector arrays, in: Proceedings of the International Workshop on Data Acquisition Systems for Neutron Experimental Facilities. Frank Laboratory of Neutron Physics, Joint Institute for Nuclear Research, June 1997.
[5] M. Popovici, W.B. Yelon, J. Neutron Res. 3 (1) (1995) 1.
[6] M. Popovici, W.B.A Yelon, J. Neutron Res. 5 (1997) 227.
[7] R. Berliner, Data acquisition/data analysis with linear position sensitive proportional counter arrays. IAEA_PC_1.fm, October 1998.
[8] C.B.R. Parente, V.L. Mazzocchi, Y.P. Mascarenhas, The IPEN-CNEN/SP PSD neutron diffractometer, in: Proceedings of the International Nuclear Atlantic Conference (INAC 2005), Santos, SP, Brazil, 2005. ISBN:85-99141-01-5.
[9] C. Domienikan, R.M. Schoueri, F. Toledo, C.B.R. Parente, Electronic module for the control of a beam shutter, in: Proceedings of the International Nuclear Atlantic Conference (INAC 2005), Santos, SP, Brazil, 2005. ISBN:85-99141-01-5.
[10] H.F. Nieman, D.C. Tennant, G. Dolling, Rev. Sci. Instrum. 5 (10) (1980) 1299.
[11] D.C. Tennant, Rev. Sci. Instrum. 59 (2) (1988) 380.
[12] P. Thiyagarajan, R.K. Crawford, D.F.R. Mildner, J. Appl. Cryst. 31 (1998) 841.
[13] D.F.R. Mildner, M. Arif, C.A. Stone, J. App. Cryst. 26 (1993) 438.
[14] I.E. Stamatelatos, S. Messoloras, Rev. Sci. Instrum. 71 (2000) 70.
[15] A.C. Larson, R.B. Von Dreele, General Structure Analysis System (GSAS), Los Alamos National Laboratory Report LAUR 86-748, 2004.
[16] R.A. Young (Ed.), The Rietveld Method, Oxford University Press Inc.—IUCr, New York, 1966.