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THE IPEN-CNEN/SP PSD NEUTRON DIFFRACTOMETER

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ABSTRACT

A new IPEN-CNEN/SP neutron powder diffractometer was constructed and installed at the IEA-R1 research reactor. It is an extensive upgrading of the old IPEN-CNEN/SP multipurpose neutron diffractometer. The old diffractometer was a single-detector instrument with a boron trifluoride (BF_3) detector and a flat mosaic single crystal copper monochromator. The main modification introduced in the old instrument was the installation of a position sensitive detector (PSD). Placed at a distance of 1600 mm from sample, the PSD spans an angular range of 20º of a diffraction pattern. An extended powder diffraction pattern can be obtained by moving the detector and collecting the data in 20º segments. A rotating oscillating collimator, placed at the entrance to the detector shield, eliminates parasitic scattering from furnace or cryorefrigerator heat shields in the vicinity of the sample, while only reducing the scattered intensity by *ca.* 10%. The collimator also makes the PSD less sensitive to ambient background leaking in through the shielding entrance. In order to increase the neutron beam flux at the sample position, a double focusing perfect single crystal silicon monochromator was installed in the instrument. With a take-off angle of 84°, the monochromator can be positioned to easily produce 4 different wavelengths, namely 1.111, 1.399, 1.667 and 2.191 Å. A beam shutter protects the operator during sample manipulation or installation of any device in the monochromatic beam. In comparison to the former instrument, the new diffractometer has a better resolution and is ca. 600 times faster in data acquisition.

1. INTRODUCTION

A new IPEN-CNEN/SP neutron powder diffractometer was constructed and installed at the IEA-R1 research reactor. It is an extensive upgrading of the old IPEN-CNEN/SP multipurpose neutron diffractometer. The old diffractometer 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 allied to a 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 in the old instrument. Consequently, an extended powder pattern could take several hundred hours of reactor time to be measured. In the new diffractometer, on the other hand, the installation of a position sensitive detector (PSD) allows the measurement of a 2θ interval of 20^o all at once. An extended pattern is then formed by 20^o segments. Depending on the extension of the pattern and the time required to measure each segment, a complete pattern can be obtained in a few hours of reactor. It should also be understood the new diffractometer has a focusing monochromator and open collimators (without plates) in its configuration. This, no doubt, produces a high-intensity monochromatic beam falling on the sample, which is another reason for a quick data acquisition. It should be emphasized we are talking about a neutron diffractometer installed in a low flux reactor and 'quick' here certainly means much more rapid than in the old diffractometer. As a matter of fact, it was estimated for the new instrument that it will be *ca.* 600 times faster in data acquisition when compared to the old one. It is also expected an improvement in the resolution.

The PSD diffractometer has several new components. All those components belonging to the detector system, including its electronics, were imported from Instrumentation Associates Inc. (IA), 2 Davis Drive, P.O. Box 13169, Research Triangle Park, NC 27169-3168, USA, (RBerliner@InstrumentationAssociates.com). IA also furnished the focusing monochromator and a rotating oscillating collimator, the latter an essential component of the new instrument. Other components were mostly constructed at the IPEN machine shop, e.g. the main neutron shield, detector shield and beam shutter. In what follows, a brief description of the essential components of the new instrument is presented.

2. THE POSITION SENSITIVE DETECTOR

The PSD [1,2,3] installed in the IPEN-CNEN/SP neutron powder diffractometer Aurora is formed by eleven linear ^{3}He detector elements, clamped together at each end to form a rigid plane. Each linear detector element is a proportional counter. In operation, each end of a detector element is connected to a charge sensitive preamplifier and the detector anode is maintained at a high voltage bias. When a thermal neutron strikes the detector element it can be captured by a ^{3}He atom in the fill gas. The electrons created in the detector fill gas by the nuclear reaction of capture are drawn to the detector anode, injecting (with gas multiplication) a charge pulse on the anode. This signal propagates to each end of the detector element, is amplified by the preamplifiers and is passed to analog-to-digital converters (dualADC modules). The digital information is then processed by an appropriate software and the position of the neutron is precisely determined in the detector element. This is the basis for the use of such detectors as a position sensitive detector [4]. The software is also responsible for the analysis of the data obtained by the detector array in order to construct the diffraction pattern.

Figure 1 shows the neutron detector array and one of the linear PSD signal processing electronics elements constructed for the new diffractometer. In the middle of Fig., a junction box serves to gather together the wires connecting the detector to its electronics. Figure 2 shows in detail the detector array wiring, the preamplifier circuit boards, high-voltage coupling capacitors and detector wiring harness.

3. THE FOCUSING MONOCHROMATOR

In order to increase the neutron beam flux at the sample position, a focusing perfect single crystal silicon monochromator was installed in the instrument [5]. The unit is composed of 9 vertically stacked silicon blades, approximately 5 mm thick, 14 mm high, 190 mm length, mechanically bent in the horizontal plane and quasi-bent by segmentation in the vertical plane. At 84° take-off angle the following reflections/wavelengths (Å) can be quite easily attained: 533/1.111, 511/1.399, 331/1.667 and 311/2.191. Switching between 533, 511 and 311 reflections only requires rotating the crystal around the vertical [011] zone axis. Switching to 331 requires flipping the monochromator bottom up with a full circle stage [6].

Figure 1. The neutron PSD array, the junction box and one of the linear PSD signal processing electronics elements.

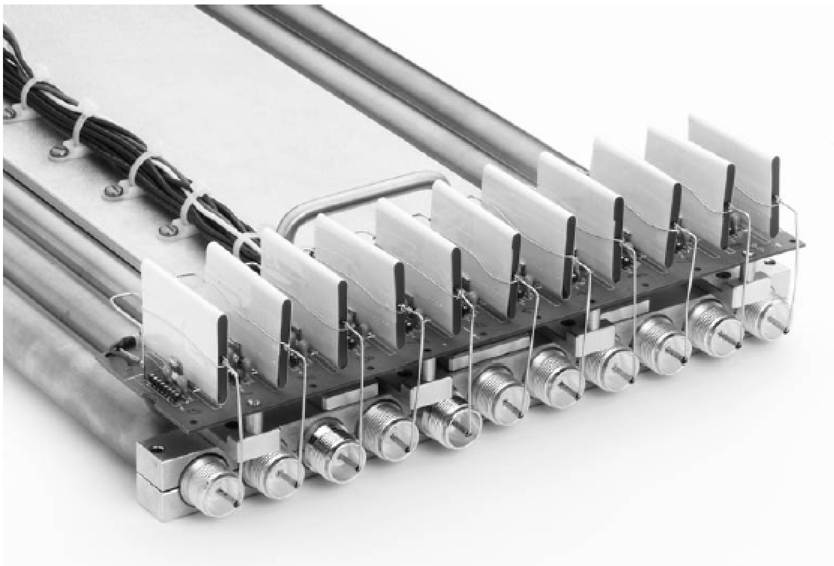

Figure 2. A back view of the PSD array showing the preamplifiers installed at one of its sides.

Figure 3 shows the monochromator installed in a goniometer used for its orientation. The goniometer has three movements: rocking, tilting and sliding. The movements are remote-controlled via the three modules seen in the Fig.

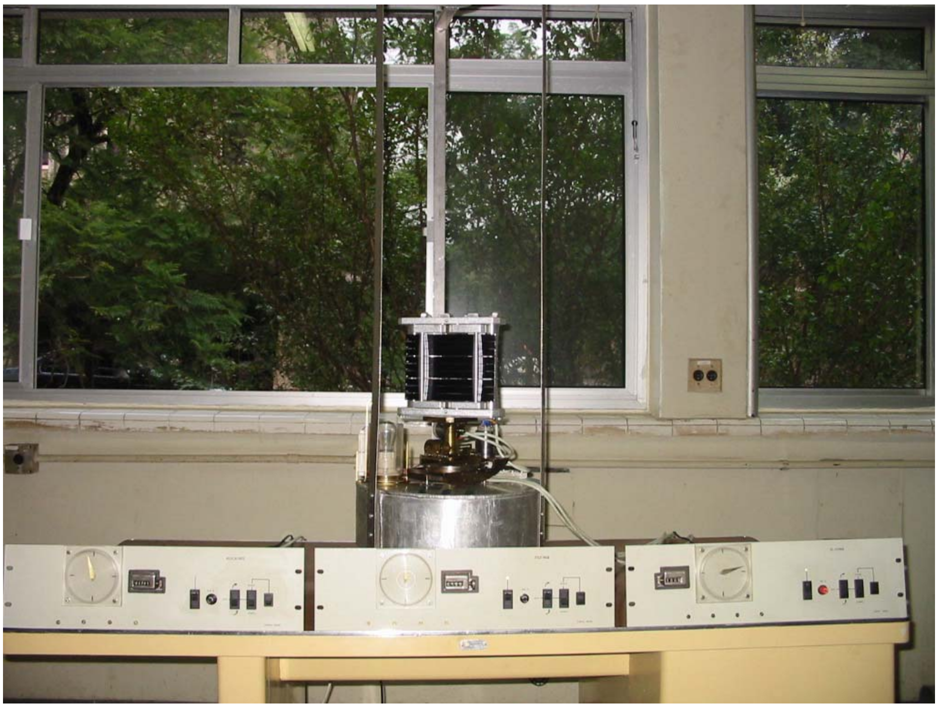

Figure 3. The focusing silicon monochromator ready to be installed in the new diffractometer.

4. THE SHIELDS

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 also accommodates the beam shutter, the focusing silicon monochromator and the incident-beam collimator. The main shield of the old diffractometer is now used as an additional shield, together with the new one. Both are supported by movable platforms to allow access to the beam port no. 6 in the reactor wall.

To cut neutrons from the ambient background, a shield for the PSD was designed and constructed at IPEN. This shield has the form of a truncated pyramid coupled with a parallelepipedal box. The PSD is placed inside the parallelepipedal box. Two arms fixed in a large 25 in. dia. rotary table support the shield. The rotary table provides the instrument with the angular 2θ movement of the detector. The movement is driven by a computer controlled geared mechanism.

5. THE BEAM SHUTTER

A beam shutter, designed and constructed at IPEN, was installed inside the main neutron shield. It protects the operator during sample manipulation or installation of any device in the sample position. Movement and positioning of the shutter is controlled by an electronic control module, also designed and constructed at IPEN. Essentially, the shutter is constituted by two contrarotating drums with peripheral square channels. An idea of how it works is given in Fig. 4.

Figure 4 is a schematic drawing of the beam shutter showing it in both *beam out* and *beam on* conditions.

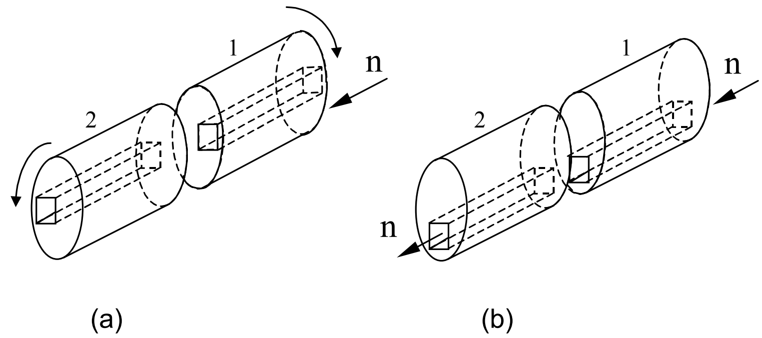

Figure 4. Schematic drawing of the beam shutter in two different situations. On the left (a), the channels are in opposite positions corresponding to the beam out condition. On the right (b), the channels are aligned corresponding to the beam on condition.

6. THE COLLIMATORS

Two open collimators were constructed in Brazil to be used in the new instrument. An in-pile collimator, to guide the polychromatic neutrons coming from the reactor core towards the monochromator. It is inserted into the beam tube no. 6 of the reactor. An incident-beam collimator, placed after the monochromator at a take-off angle of 84° from the polychromatic beam. It allows the focusing of the monochromatic beam on the sample. A third collimator, a rotating oscillating collimator (ROC), furnished by IA, is placed at the entrance to the detector shield. It eliminates parasitic scattering from furnace or cryorefrigerator heat shields in the vicinity of the sample, while only reducing the scattered intensity by *ca*. 10 %. The ROC also makes the PSD less sensitive to ambient background leaking in through the detector shield entrance.

Figure 5 shows the in-pile collimator separated into its three constituent parts (one of them a plug), Fig. 6 the incident-beam collimator and Fig. 7 the ROC.

7. FINAL COMMENTS

A new IPEN-CNEN/SP PSD neutron diffractometer is open to the brazilian and latin-american scientific and technological communities. It was designed mainly to be used in crystalline and magnetic structure determination, as well as, for application of the Rietveld method in structural multiphase analysis. Under request, other types of application can be considered.

Figure 5. The in-pile collimator.

Figure 6. The incident beam collimator.

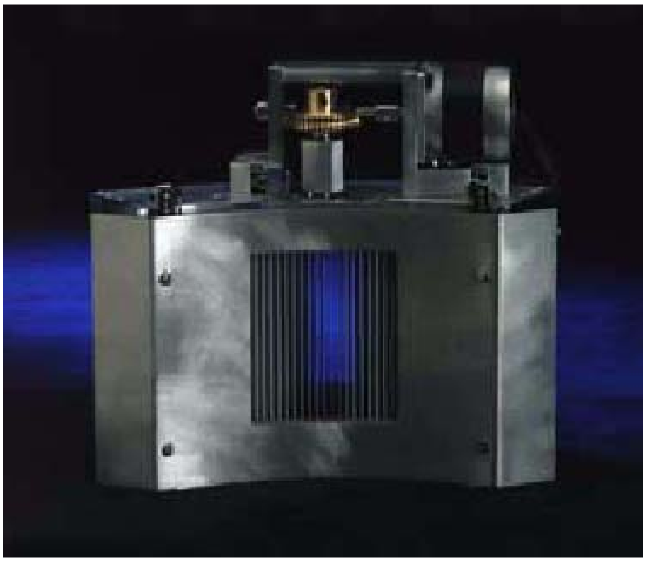

Figure 7. The rotating oscillating collimator (ROC).

Figure 8 is a view of the new diffractometer showing the main neutron shield, the incident-beam collimator, the mechanism to drive the 2θ movement and the spectrometer. The round part of the neutron shield is the shield formerly used in the old diffractometer. Behind it and touching the reactor wall is the new main neutron shield. The incident-beam collimator is inserted into this shield. A small part of it is seen in the Fig. The spectrometer, composed by the PSD shield and the ROC, both counterbalanced by a great number of lead bricks, is also seen. It is supported by two arms fixed in a rotary table. The box on the movable plataform contains the 2θ drive mechanism connected to the rotary table.

Figure 8. The IPEN-CNEN/SP PSD neutron diffractometer.

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