J. Synchrotron Rad. (1998). **5**, 491–493

A versatile X-ray diffraction station at LNLS (Brazil)

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(*Received 1 September 1997; accepted 12 January 1998*)

Versatility was a major consideration in the project to provide an X-ray diffraction station at LNLS. At least two techniques are possible at the station: powder diffraction and multiple single-crystal diffraction. A two-crystal monochromator based on monolithic elastic translators, developed at LNLS, with sagittal focusing by the second crystal, allows 10 mrad of a $\geq$2 keV monochromatic beam to be used on the diffractometer. The station is equipped with a fast scintillation detector, imaging plates, a high-energy-resolution pin-diode detector, an ionization chamber and a high-angular-resolution soller slit. The data collection and control hardware and software were also developed at LNLS. The θ–2θ goniometry for powder diffraction on this 1 m-diameter diffractometer is based on commercial rotation tables. The multiple single-crystal goniometry is realized by an independent elastic axis driven by differential micrometers for both high- and low-resolution angular movements. At least four independent axes can be positioned as necessary on the diffractometer table. Powder diffractograms and double-crystal rocking curves collected with the synchrotron beam show the expected quality.

Keywords: X-ray diffraction; diffraction station; diffraction beamline.

1. Introduction

The beamline (Fig. 1) installed at the bending magnet B12 of the Laboratório Nacional de Luz Síncrotron (LNLS), in Campinas, Brazil, is devoted to different techniques, mainly involving X-ray diffraction, and was conceived to be versatile. The beamline includes a two-crystal monochromator and, alternatively, a scanning four-crystal monochromator, which serve a dual-purpose diffractometer for powder diffraction and multiple single-crystal techniques, such as double-crystal rocking curves, reciprocal-space mapping, standing waves, back-diffraction, grazing-incidence diffraction, interferometry *etc.* This beamline is already operative and some experiments have been performed. A brief description of the beamline follows together with the results of preliminary tests.

2. Beamline description

At present the 1.37 GeV LNLS ring runs with 100 mA current and 6 h lifetime. The X-ray diffraction beamline is installed at the 1.7 T field B12 bending magnet. The measured emittance of the source is 100 nm rad. The slit systems accept 11 mrad of the emitted beam horizontally.

The beamline is expected to operate in the 2–15 keV range. A double-crystal monochromator with elastic translations, developed at LNLS (Correa *et al.*, 1992), is located at 7 m from the source and will use a sagittal-focusing second crystal to accept 10 mrad of the horizontal divergence. It can be easily substituted by a dispersive four-crystal scanning monochromator, also developed at LNLS (Tolentino & Rodrigues, 1992) for higher energy resolution. The monochromator (and the rest of the beamline) can be moved as close as 5.3 m from the source, if necessary. Also, the first crystal of the monochromator can be moved in and out of position very easily to let the white beam go through to the diffractometer, as necessary.

A 70 $\times$ 10 mm beryllium window, 125 μm thick, which will eventually support atmospheric pressure, is located 4.4 m from the source. This window is substituted by a 7.6 μm Be window, mounted in a VAT valve to separate the machine vacuum from the 10^{-2} torr pressure in the beamline, when a low-energy beam (say 2 keV, when transmission is 90%) is used in the diffractometer. For energies above 4 keV, the beamline operates at high vacuum from the 125 μm window to the ionization chamber located between the monochromator and the diffractometer. The working pressure in the diffractometer can vary from 760 to 10^{-2} torr.

Between the beam-defining slits and the monochromator there is a removable fluorescent screen to visualize the white beam, and after the monochromator there is another screen for the monochromatic beam. Before the diffractometer there is a second slit system to avoid the scattered radiation. Both beamline slit systems have four independent tantalum blades, each with sufficient range to stop the beam completely.

3. Diffractometer

The diffractometer is in a 1 m-diameter airtight vessel with a 90 cm-diameter 2θ table that can be detached from the driving goniometric table and screwed to the fixed part of the diffractometer. Then, it can be used to accommodate independent axes for a multiple single-crystal set-up, slits, *etc*.

Commercial encoded goniometers drive the θ axis and the 2θ table/detector arm. When the table is fixed and, consequently, detached from the goniometer, an arm can be attached to the 2θ goniometer to hold and move the detector. A soller slit with an angular resolution of 8×10^{-4} was developed in the LORXI laboratory (Physics Department, UFPR) and is positioned between the sample on the θ axis and the detector, to increase angular resolution when necessary for powder diffraction experiments.

An independent goniometric axis was developed based on a flexure pivot, an arm and a differential screw micrometer, with a coarse movement of 2.58 arcsec per step, range 6°, and a fine movement of 4.22×10^{-2} arcsec per step, range 10 arcmin. A piezo-motor drives the elastic φ scan to adjust the crystal parallelism on the double-crystal set-up The total size of one axis is ~18 $\times$ 18 $\times$ 7 cm and four or more axes can be screwed tightly to the diffractometer table for multiple-crystal diffraction experiments.

For X-ray detection there is an ionization chamber before the diffractometer, a fast scintillation detector (106 counts s^{-1}) and a high-energy-resolution (264 eV at 5.9 keV) pin-diode detector with a multichannel analyser board and software installed on the main microcomputer. Image-plate supports are being installed on the diffractometer covering 2θ angles of at least 0 to 120°. An 1100 × 1200 pixels CCD detector will be available soon. A 'magic eye' for alignment is also available.

A temperature-control stage for the sample is to be installed on the θ axis and it will be possible to control the temperature from 70 to 700 K within 0.1 K. The hardware and software for data collection and control were developed at LNLS. The main program is written in Delphi and runs in a Windows environment.

4. Preliminary results

A powder diffraction experiment with an alumina sample was carried out with a 1 mm slit in front of the scintillation detector and an 8 keV beam from the double-crystal monochromator (Fig. 2). In Fig. 3 a comparison is made of the resolution of this set-up with the resolution using the soller slit instead of the 1 mm slit between the sample and detector.

To test the possibility of using an elastic scanning X-ray interferometer in the LNLS experimental hall, even with the relatively high level of noise and vibration present, an interferometer was installed on the θ axis of the diffractometer and a fringe scan was performed at 8 keV, resulting in good-quality Moiré fringes allowing phases to be determined with a precision of at least one millifringe.

A double-crystal rocking curve was obtained with two Si 111 perfect crystals on the ± setting using the independent-axis fine scan. The FWHM of this curve, 9.3 arcsec, is in accord with the theoretical value. A test of the stability of the apparatus was made using a (corrected) constant-incident-intensity beam on the flank of the rocking curve; there was no variation of the detected intensity during a 2 h test.

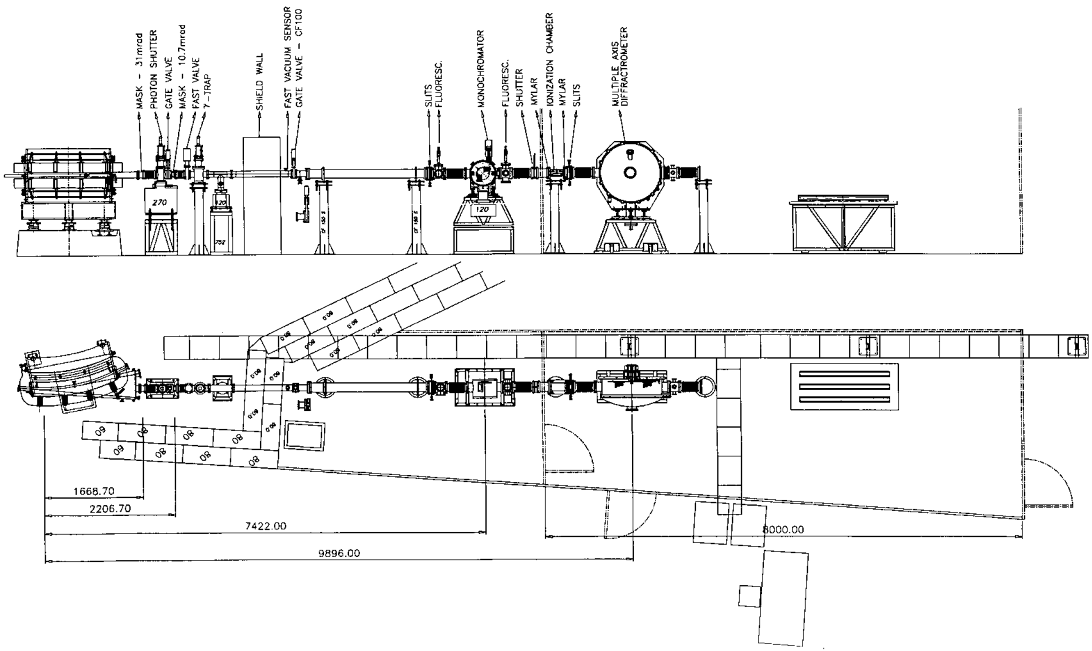

Figure 1
The X-ray diffraction beamline.

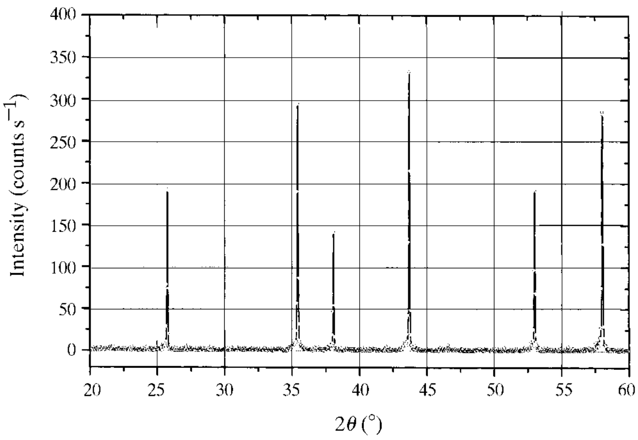

Figure 2
Intensity *versus* 2θ for a powder diffraction experiment with an alumina sample.

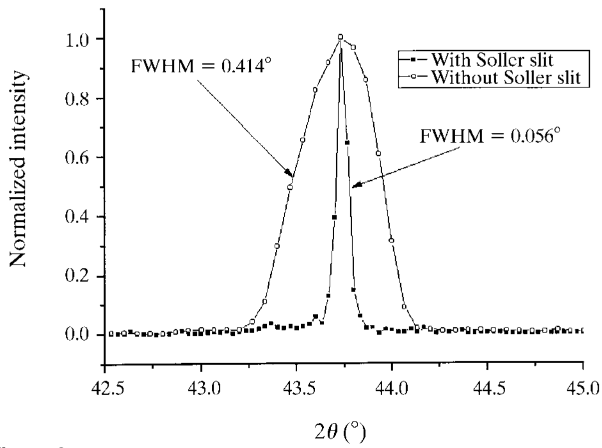

Figure 3
Comparison of the resolution using a soller slit instead of a 1 mm slit between the sample and detector.

5. Summary

An X-ray diffraction station was proposed, built and is being used at LNLS, the Brazilian synchrotron radiation laboratory. Powder diffraction and multiple-crystal preliminary experiments showed the expected results. A dedicated station for powder diffraction is to be built at LNLS in the near future and the station described here will be then devoted mainly to single-crystal diffraction.

The authors would like to acknowledge the essential help of LNLS staff members during the development and construction of the station. The support of Professor Aldo Craievich and fruitful discussions with Drs A. Ricardo D. Rodrigues and Helio Tolentino are also acknowledged. This work is supported by LNLS and PADCT-INST/CNPq.

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