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Upgrades to the XRD1 beamline optics and endstation at the LNLS

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Abstract. XRD1 was the first X-ray diffraction beamline to be built at the LNLS and after approximately 12 years of operation it was substantially updated to improve beam stability, increase the reliability of the monochromator movement as well as provide an experimental hutch that would meet the demands of users. The improvements included the construction of an independent concrete slab below the mirror and monochromator to minimize the vibrations originating from the floor. In addition, the installation of new monochromator mechanisms as well as the replacement of the two Si(111) crystals were performed in order to attain higher precision, stability and reproducibility during operation. Moreover, the diffractometer was replaced by a 3-circle heavy duty diffractometer from Newport to collect XRD patterns primarily in capillary geometry. A robotic arm was installed for fast and automated replacement of samples as well as to secure a cryojet or a hot air blower in front of the sample during measurements. In addition, a housing equipped with 24 Mythen detectors was installed at the beamline allowing for extremely fast data acquisition. Another upgrade was the integration of motors and control systems from PXI National Instruments and Galil controllers with Phytron. These systems are crucial for the next upgrade that is underway at the beamline: enabling remote access for users to collect their measurements without the need to travel to the LNLS.

1. Introduction

The Brazilian Synchrotron Light Laboratory (LNLS) is a 1.37 GeV machine and is the only synchrotron facility in Latin America [1]. It has been in operation since 1997 with the X-ray diffraction beamline (XRD1) being one of the first 7 beamlines initially built at the laboratory. The beamline is installed at the 1.7 T field B12 bending magnet and operates in the 5.5-14 KeV range [2]. Being the first diffraction beamline, the design of XRD1 was aimed at flexibility to accommodate a wide range of experiments including powder diffraction, reflectivity, monocrystal diffraction, multiple diffraction as well as inelastic scattering. In the following years two additional diffraction beamlines were built at the LNLS namely XRD2 (in 2003) and XPD (in 2005) with emphasis on monocrystal and powder diffraction respectively [3,4]. After approximately 12 years of operation the first X-ray diffraction beamline became uncompetitive compared to the more recent beamlines caused by lack of beam stability, low photon flux, precision and reproducibility of the monochromator motors as well as limitations to install furnaces and detectors in the diffractometer. Thus, during 2010 the XRD1 beamline was completely updated to improve beam stability, increase the reliability of the monochromator movement as well as provide an experimental hutch that would meet the requirements of users.
2. Optical enclosure upgrade

The optics of XRD1 beamline includes a vertically collimating/ cylindrically focusing bent Rh-coated mirror and a Si(111) double-crystal monochromator with developed at the LNLS. The second crystal of the monochromator is sagittally bent to focus the beam at the sample position producing a beam 3mm in the horizontal and 0.8mm in the vertical at 8 keV. Part of the beam vibrations originated due to the common floor slab supporting the optical components in the optical enclosure as well as the area outside the optical hutch. This problem was solved by building an independent concrete slab to support the mirror and monochromator. The concrete slab has a one inch gap to isolate the optical enclosure from the rest of the floor.

![Optical enclosure upgrade](image)

**Figure 1.** Monochromator mechanics design after the upgrade: (a) side view and (b) top view.

The second significant source of beam vibrations originated from the monochromator mechanics and the driver motors which wore out after many years of use. As a solution the monochromator mechanics and the support for the two crystals were completely redesigned. Figure 1 shows drawings of both the old and new design of the monochromator. The new mechanics are more robust than the old ones which now include titanium weak links, new motors and a new bender design. Further, a new Si(111) crystal from Crystal Scientific was installed to replace the first crystal. The thickness of this new crystal was increased by 5 mm to improve thermal stability. In addition, the thermal bath that is used to water cool the first crystal was also replaced by a LAUDA Proline RP845 to help avoid temperature fluctuations. Moreover, the control system of the monochromator was also renovated by the installation of a PXI National Instrument communicating with Galil controllers and Phytron ZMX+ electronics to power the stepper motor drives on an EPICS platform.

![Photon flux](image)

**Figure 2.** Photon flux as a function of energy: prior to upgrade (squares), after the upgrade (stars), and comparison with the powder diffraction beamline XPD at the LNLS (circles).

Following these upgrades in the first half of 2012 the commissioning tests confirmed significant improvements in the stability of the beam at the sample with vibrations below 100 µm after 10 h. Further,
monochromator motor reproducibility and photon flux as a function of energy became comparable to the more modern powder beamline, XPD, at the LNLS (Fig. 2).

3. Experimental station upgrade
Nowadays the XRD1 beamline has two end stations, the first one was built in 1997 and included a 3 axis Huber diffractometer on a rotational stage to change the scattering plane from the vertical to the horizontal as well as an in-vacuum diffractometer. The second endstation was built in 2010 to house a thermo-mechanical simulation facility (Gleeble) [5]. The first endstation had several vacuum and mechanical problems that became more severe after several years of operation. The diffractometer also showed limitations regarding the load capacity for different sample environments and detectors. Based on the large number of scientific proposals submitted to characterize powder samples at the LNLS as well as the positive results regarding beam stability, precision and reproducibility of the motors during operation resulting from the optical enclosure upgrade, the decision was made to upgrade the first endstation of the XRD1 beamline to provide a high-throughput beamline for powder diffraction analysis. To accomplish this task the previous diffractometer was replaced by a 3-circle Heavy Duty diffractometer from Newport [6]. The diffractometer is suitable to collect XRD patterns in capillary geometry. A unique sample holder was developed in house to allow for flat-plate geometry. The Heavy Duty diffractometer has three coaxial high precision rotatory stages, one for the sample (Θ) and the other two for detectors (2Θ and δ) (Fig. 3). The considerable load capacity of the three circles (Θ = 35Kg; 2Θ = 100Kg and δ = 60Kg) allows the installation of heavy sample holders or cell reactors and detector stages. Furthermore, to provide fast data collection, which is fundamental to rapidly characterize a large numbers of samples and to allow for time resolved experiments. This is achieved using an array of 24 Mythen detectors installed in the delta circle at a distance of 760mm from the sample and a Q resolution of the 0.02Å⁻¹. The Mythen detectors are placed inside a Huber built casing filled with helium (Fig. 3). In addition, a Yaskawa-Motoman robotic arm was installed for quick and automated replacement of samples as well as to hold a Cryojet 5 from Oxford Instruments allowing experiments from 100K to 500K as well as a soon to be acquired hot air blower from 298K -1273K. The next step of the XRD1 upgrade towards a high-throughput XRD beamline is the remote control of the endstation, which will allow access for users to collect their X-ray diffraction patterns without the need to travel to the LNLS. This is a work in progress and operation is foreseen to commence by the end of 2013.

Figure 3. XRD1 new end-station, with a Newport Heavy Duty diffractometer and a Yaskawa-Motoman robot for samples exchange and cryojet / hot air blower support.
4. Conclusions
The XRD1 beamline at the Brazilian synchrotron has undergone significant upgrades from the optics to the endstation. A noticeable improvement in beam stability and monochromator motor reproducibility was observed after the reforms in the optical enclosure. The experimental stations were replaced by a high-throughput modern powder diffraction station, reinforcing the industry focus of the beamline since the installation of the thermo-mechanical Gleeble instrument. The combination of a Heavy Duty Newport diffractometer with the 24K Mythen detector and robotic arm sample changer will allow for very fast measurements. These capabilities are desirable for in situ experiments as well as industrial applications. Future planned improvements for XRD1 include the implementation of remote access control of measurements for maximum process efficiency.

References