Upgrades to the XAFS2 beamline control system and to the endstation at the LNLS

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Abstract. The XAFS2 is a general-purpose X-ray absorption beamline. It is the second one built at the LNLS. After approximately 7 years in operation this beamline has been substantially updated in order to improve its experimental possibilities. Recently arrived, a 4-circle Huber diffractometer has been incorporated to perform combined experiments. This collects XRD patterns with the XAFS. Through the development of a new sampling environment it is now also possible to perform these measurements in situ/operando conditions. Other upgrades include a complete remodelling of the beamline software and its control system. The following new systems are crucial for the next steps that are currently underway at the beamline, namely, (i) enabling remote access for users and (ii) the testing of QEXAFS measurements.

1. Introduction

Upon the completion, in 2007, of the commissioned works on the XAFS2 [1], a large number of users have been using this experimental facility in order to perform several kinds of experiments [2-5]. Since 2007, and with the development of combined simultaneous characterizations [6], there are a plethora of new possibilities additional to the usual XAFS experiments. Among these numerous possibilities; a combined XAFS and XRD experiment would be especially useful in order to obtain structural information of the short range order (around a selected absorber atom) and of the long range order, thus unveiling information on the material as a whole. Here one is probing different properties of the same system in a unique experiment under the same conditions, thus bridging the complex gaps in the structure. Therefore, in order to improve those abilities of the beamline, a 4-circle Huber Diffractometer was added to the experimental hutch in order to explore both techniques [7] through user demand. Here below an example of these XAFS and XRD measurements is presented. The example shows the performance of the beamline at work while combining both techniques.

Importantly, the control system of the beamline has been renovated by the installation of a PXI. The PXI is from National Instruments (PXI-NI) and communicates with Galil/Parker controllers on an EPICS platform [8]. Note some parts of the motors were changed in order to improve performance with the upgrade and there were also important changes to the control hardware. The Windows
The operational system was replaced with Red Hat Linux and the 3-WinDCM control system [9] with EPICS. Furthermore, a new python based script (Py4Syn) was added. This provides high-level abstraction for device manipulation, scan routines, real-time plots and more [10]. This package was created with the aim of providing a simple yet powerful tool to allow scientists and users to develop their own scripts for data acquisition. For user-friendly interface builds a Control System Studio (CS-Studio) is used.

The next step with the XAFS2 upgrade, namely, towards a high-throughput XAFS beamline, will involve testing the viability of performing QEXAFS and testing the remote control at the endstation [11]. This will allow access for users to collect their data without having to travel to the LNLS. This is a work in progress and normalized operation is foreseen to commence by the end of 2015.

2. Brief overview of the beamline

The design, construction and performance testing of the majority of the elements of the XAFS2 beamline have been completed in house, save the mirrors that were purchased from SESO (France). The beamline is positioned at the 15° port of the D08B bending magnet of the LNLS storage ring. It operates with a 1.37 GeV source with a maximum current of 250 mA, a critical energy of 2.08keV and offers a beam life time close to 17 h without coupling correction [12]. The XAFS2 beamline covers the high-energy x-ray range and delivers photons from 3.5 up to 17 keV. The schematic representation of the main components of the beamline and other useful information can be obtained from the website (http://lnls.cnpem.br/beamlines/xafs/beamlines/xafs2/).

The optical hutch shapes the white beam, collimating it vertically via the First Rh-coated cylindrical mirror, which sends a parallel synchrotron beam onto the two flat Si(111) Double Crystal Monochromator (DCM). The DCM has fixed exit geometry and is the only optical element with thermal stabilization (i.e. water cooled). A second Rh coated toroidal bendable mirror refocuses the monochromatic beam, in the sample position, to a low eccentricity ellipse of approximately 450μm in diameter. The typical photon flux, in the sample position, for standard beam size and current ring is among the $10^9$ photon/s range. Harmonic content for energies higher than 6 keV is lower than $3x10^{-5}$. For lower energies, harmonic rejection is performed by detuning the second crystal. The standard beamline setup allows for transmission and fluorescence modes. For transmission experiments three ionization chambers, (for beam intensity measurements) are filled with optimal He, N$_2$ and/or Ar gas mixtures at a total pressure of 1.5 bars.

The signals of the ionization chambers are amplified by Stanford picoammeters and digitalized by a voltage to frequency converter before being finally read by the counters at the PXI-NI. A Germanium 15 elements fluorescence detector, from Canberra Inc., and an electron detector complete the detection equipment. Typical absorption data measured in transmission mode at room temperature is presented in figure 1. The single scan data were processed in Athena [13] without any filtering. Almost noise-free data are visible even for $k > 16$ Å$^{-1}$, which allows for the securing of detailed and precise information of the short range structural order.

![Figure 1](image-url)  
**Figure 1** Cu foil measured at XAFS2: here is shown a $k^2 \chi(k)$ vs. $k$ and the Fourier Transform in R space. Data were collected at 1s per point from 8850 eV up to 9500 eV and then in 3 steps on $k$ space (0.07, 0.05 and 0.03) up to 10500 eV reaching 3s per point on the last step.
3. Advances in the experimental set-up and the beamline control

During the summer school program (http://lnls.cnpem.br/education/student-program/bolsas-de-verao/) at the Brazilian Synchrotron Light Laboratory, the XAFS2 beamline was adapted to perform diffraction experiments. This instrumentation development allowed us to open up a new window on the usual XAFS measurements, which is especially useful for combining the techniques to obtain information on short and long range order. Both techniques were explored by performing in situ studies with a capillary micro-reactor [14], showing the hydrogen desorption on a Mg$_2$CoH$_5$ sample (MgCo hydride) during a thermal treatment and under a helium atmosphere.

The possibility to change between XAFS and XRD experiments (in a few minutes and following the behavior with a mass spectrometer) gives a complete picture of the changes in the material under in situ conditions. This is a very promising combination to explore catalysts or functional materials, thus increasing the research potential of the beamline. In figure 2 and 3 it is possible to show the performance of the beamline with this setup. Results in the XANES region, shows slight shifts in the spectrum upon increasing the treatment temperature and characterizes changes in the oxidation state. In this scenario, we use the DCM in a pseudo-channel cut crystal configuration in order to reach an accuracy level of 0.1eV (being the mean of fluctuations at the edge energy when measuring 20 Co foil scans). With the XRD data it is possible to follow the change in the initial tetragonal phase hydride (β-Mg$_2$CoH$_5$) to the cubic (γ-Mg$_2$CoH$_5$) phase. With the displacement of the diffraction peaks this phase becomes unstable at higher temperatures and the complex hydride decomposes into Mg, Co and H$_2$.

**Figure 2** XANES spectra at Co-K edge on sample Mg$_2$CoH$_5$ under thermal treatment following the E$_0$ shift. The acquisition time was close to 300s per spectra. Sample measurements are done simultaneously with a Co foil in between I$_1$ and I$_2$ in order to have a good energy calibration (black line at 7709 eV).

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**Figure 3** MS data and XRD patterns obtained on the hydride Mg$_2$CoH$_5$ under the thermal treatment. The acquisition time for XRD measures was 300s. The energy used to measure XRD data was 11104 eV. Lines in diffractograms guide the eye onto the shifts of the identified phases (β-Mg$_2$CoH$_5$ and γ-Mg$_2$CoH$_5$).
The results obtained with mass spectrometry indicated a maximum release of H₂ at a temperature close to 250°C, an increase in energy is observed at the same temperature when plotted on a graph of $E_0$ vs. Temperature (figure 2) indicates a possible increase in the oxidation state, probably due to the loss of electrons from the cobalt upon the H₂ release. Correlating the results of diffraction, absorption and Mass Spectrometry (MS), it was noted that the initial release of H₂ is not related to change in the structure of the hydride. Rather, it has to do with the departure of the hydrogen interstitial positions in the crystal structure and without it changing per se. More detailed and careful experiments of this kind of system can be shown in the work of Zepon et al [15], differences in temperatures related to the paper are due to their air exposure.

Other changes on the beamline include the integration of motors and the control system in EPICS/Linux replacing the old Delphi/Windows platform. A PXI from the National Instruments(NI) chassis and its modules were made available to EPICS through the NI Real-Time hypervisor virtualization system. In sum, this allows the running simultaneously of the EPICS/Linux and the LabVIEW Real-Time in the same PXI controller, sharing a common memory block as their communication interface. A control software for the beamline developed in Python at the LNLS (Py4Syn) [10] completes the picture with a practical and intuitive user interface in CS-Studio. These changes were essential preparations for the next upgrades on the beamline. The first one is to implement the LabWeb project [11] aimed at allowing the remote operation of the beamlines for the user community. The implementation of LabWeb will allow researchers to use the laboratory structure through a web browser, sending the samples to the LNLS without leaving their research centres, reducing time and travel costs in a continental country like Brazil. Thereafter, is the work to develop a fast scan acquisition (QEXAFS) in order to reduce times and in tandem to test the best software and hardware solutions for our future beamlines in the next Brazilian Light Source (Sirius-
http://lnls.cnpem.br/sirius/).

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