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Supporting information for article:

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XDS: A flexible beamline for X-ray Diffraction and Spectroscopy at the Brazilian Synchrotron

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1. Supplementary Information

1.0.1. Rietveld analysis X-ray powder diffraction data of a silicon sample was collected at XDS beamline using the sagittal Si(111) monochromator and both the VFM and VCM in the cylindrical Rh stripe (mode E). This data was analyzed using the Rietveld method. The results are shown in Fig. 1. This data was collected in Debye-Scherrer configuration at room temperature using NIST Standard Reference Material 640c. The X-ray energy was set to 20 keV, which was established measuring the absorption edge of molybdenum (E_0 = 20.00 keV, corresponding to a wavelength of 0.61992 Å). The detection of the diffracted photons was done by using a HOPG analyzer in combination with a standard LaBr scintillator in a 2θ interval of 1° up to 164°. The angular step used in this measurement was 0.01°, resulting in a total acquisition

time of 4 hours. The Si powder was mounted inside a polyimide capillary tube (0.635 mm inner diameter and 0.025 mm wall thickness), which was continuously spun to minimize grain size statistical problems and preferred orientation.

The diffraction pattern of the Si sample was fitted using the Rietveld method (Rietveld, 1969) with the GSAS suite software package (Larson & Dreele, 2000; Toby, 2001). The expected F$d\bar{3}m$ (N° 227) space group was used in the fit and the cell parameter was fixed to $a = 5.431195$ (9) Å (NIST, 2005). This also allowed the extraction of the actual X-ray wavelength employed in the experiment. In Fig. 1 we present the experimental data and the corresponding fit, which represents and excellent model to the experimental data. The wavelength obtained using this methodology was $\lambda =$ 0.61977 (1) Å, which corresponds to 20.005 (1) eV, in good agreement wit the value set by the beamline monochromator. The only crystallographic parameter allowed to vary in the fit procedure was U_{iso}, which converged to a value of 0.00630(5) Å^2. The obtained agreement factors of the fit R_{wp}, R_p and χ^2 were 14.22, 10.2 and 5.044, respectively.

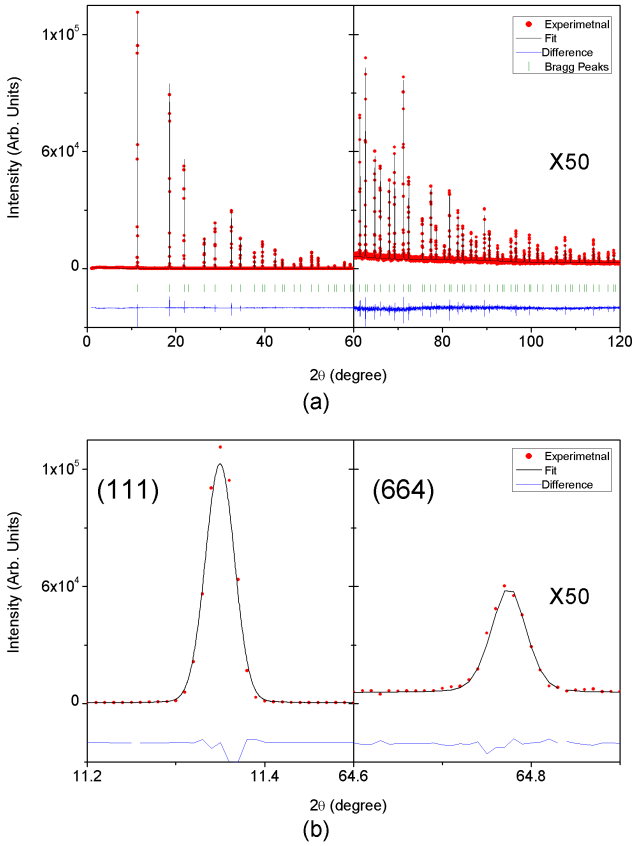

Fig. 1. (a) Room temperature X-ray powder diffraction data (red circles) and Rietveld refinement line profiles (full black line) for silicon. The (blue) line in the bottom is the difference between calculated and experimental patterns. The bars correspond to the Bragg reflections for Si in the F$d\bar{3}m$ space group. (b) Detail of the (111) and (664) peaks.

1.0.2. X-ray absorption spectroscopy During the commissioning of the XDS beamline the XAS of several reference materials were measured in the whole energy range covered (5-30 keV). Here we illustrate the beamline capabilities to perform XAS experiments by showing two XAS spectra at the extremes of operation energy of the beamline.

In Fig. 2 we show the XAS spectra of a vanadium and a paladium metal foils, which K-edges are at about 5.4 keV and 24.3 keV, respectively. These spectra correspond

to standard references manufactured by EXAFS Materials (USA) and were measured in transmission mode using N_2/Ar-filled ion chambers (vanadium/palladium, respectively) at room temperature. The main figure shows the near-edge region up to about 200 eV above the absorption edge and the inset shows the EXAFS region converted to k-space (k^3-weighted $\chi(k)$).

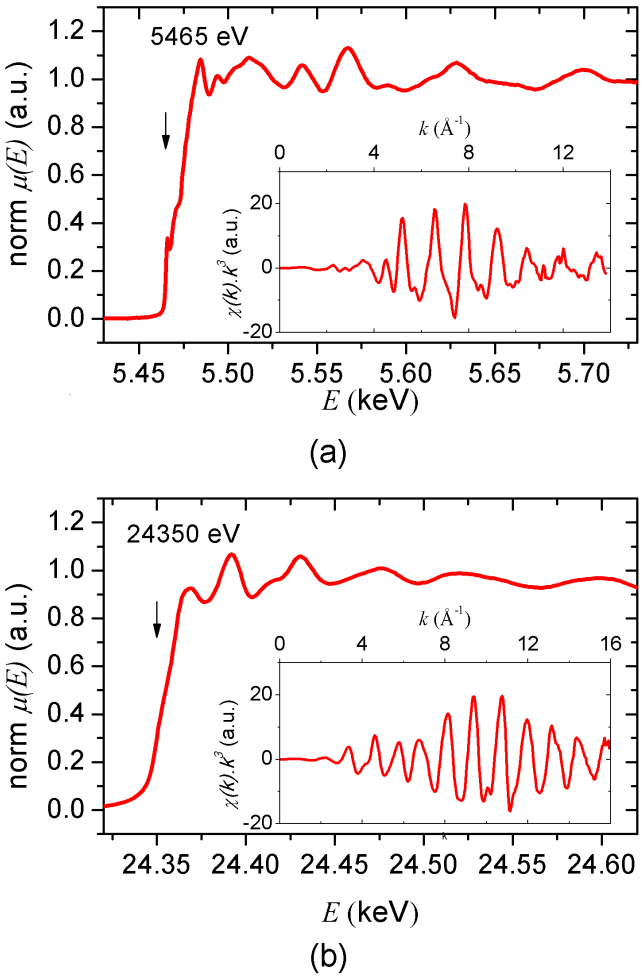

Fig. 2. K-edge XAS spectra of (a) vanadium and (b) palladium metal foils. Both spectra were measured in transmission mode and at room temperature. The insets show the extracted k^3-weighted EXAFS, illustrating the excellent data quality necessary for accurate EXAFS analysis.

The vanadium XAS was measured using the Si(111) monochromator, with the VCM in the Si stripe to minimize higher harmonics contamination and the VFM in the Rh

stripe and toroidal bending (mode B). On the other hand, the palladium XAS was measured using the Si(311) monochromator with both the VCM and VFM in the Pt stripe, the latter also with toroidal bending (mode D). The total acquisition time of the complete EXAFS up to k = 13 $Å^{-1}$ in the case of the vanadium spectrum and k = 16 $Å^{-1}$ in the case of the palladium spectrum was about 25 minutes for both data sets. The implementation of a continuous monochromator movement during data acquisition allowing faster energy scans is currently under investigation. The excellent quality of the XAS data in Fig. 2, both in terms of reproducibility of the sharp absorption features in the near-edge region as well as to the low noise in the high k- region of the EXAFS, illustrates the capabilities of the XDS beamline to perform standard XAS experiments in 3d- and 4d-metals.

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