

In-house XAS measurements using a von Hamos curved crystal spectrometer and an X-ray tube

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X-Ray Absorption Spectroscopy (XAS) has become a routine technique at synchrotron radiation sources where the bright, coherent, energy tunable and monochromatic X-ray beams permit to investigate a variety of samples from different disciplines such as physics, chemistry, environmental sciences, geology, cultural heritage, archeology and biomedicine. For external users, however, the access to such advanced research facilities is not so easy and the available beam time is limited. In this context, a laboratory-based setup offers the advantages of lower costs and better accessibility. Such a setup based on a von Hamos curved crystal spectrometer [1] and a side-window X-ray tube was therefore developed at the University of Fribourg for in-house XAS measurements.

In the present setup the focal line of the electron beam on the X-ray tube anode, the sample and the center of the spectrometer crystal are aligned along the direction determined by the Bragg angle corresponding to the measured absorption edge (see Fig. 1). To preserve the energy resolution of the spectrometer, a narrow rectangular slit (width of 200 μm) is placed between the sample and the crystal. The X-ray tube is operated at low voltages in order to reduce the scattering background and the contributions from higher orders of diffraction.

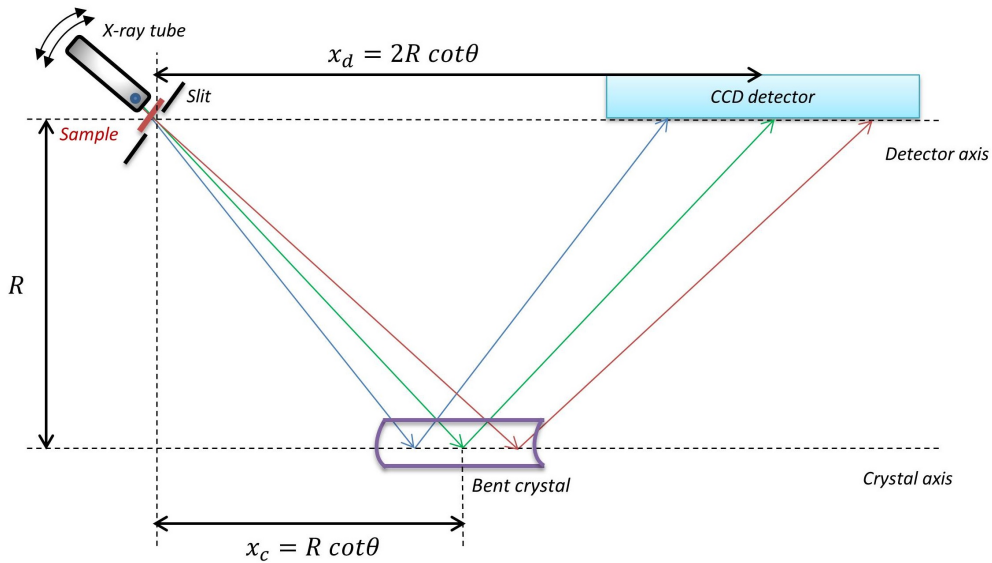


Figure 1: Schematic drawing of the laboratory-based XAS setup. R stands for the crystal radius of curvature and θ for the Bragg angle.

The K-edges of Ti, Fe, Cu and Ge and the L_3 -edges of Mo, Ag, Hf, Ta and Pt were measured. To probe the sensitivity of the setup, the less pronounced L_2 - and L_1 -edges were also measured but only for Mo and Ag. Precise edge energies could be determined and compared to existing experimental and theoretical values. The effect of the sample thickness on the experimental edge energy was carefully investigated. The L-edge energies corresponding to the first inflection point were found to be systematically lower than the values commonly used as references, the deviations being particularly pronounced for samples whose absorption edges are characterized by strong white lines.

References

- [1] J. Hoszowska, J.-Cl. Dousse, J. Kern and Ch. Rhême, Nucl. Instrum. Meth. Phys. Res. A 376, 1996, 129