Quick EXAFS Monochromator

Time dependent phenomena such as chemical reactions can be studied with X-ray Absorption Spectroscopy (XAS) using a quick-scanning monochromator and fast data acquisition. A quick-scanning monochromator continuously cycles through a small energy region while the absorption spectrum is measured (Quick EXAFS, or Quick NEXAFS). At the University of Wuppertal (Germany), the research group of Prof. R. Frahm developed such a special monochromator. This design is employing a channel-cut crystal that is mounted to a CAM-drive tilt table for rapid angular oscillations. The monochromator is able to record full EXAFS spectra in less 50 ms.

Bruker ASC cooperates with the University of Wuppertal and realized a commercially available QEXAFS monochromator. The first system was built for the Super-XAS beamline (X10DA) at the Swiss Light Source (SLS), (see figure 1). It is very successfully in routine operation since March 2008. The Super-XAS beamline is operating the QEXAFS monochromator in line with a conventional double-crystal monochromator from Bruker ASC. One can easily change from one monochromator to the other and back within ten minutes without any changes to the sample.

Fast Scanning Mechanism

The highly reproducible and fast movements – indispensable for QEXAFS – are realized by means of an oscillating cradle carrying the channel cut crystal. This cradle is controlled through an eccentric drive that is run by a servo motor. A sketch of the drive is shown in figure 2. The pivots of the cradle are frictionless flexural cross spring bearings. A con-rod connected to the cradle converts the rotation into an up-down move of the cradle. Various amplitudes and speeds can be selected. Maximum speed of the cradle is approx. 40 Hz (2400 rpm). Together with a photoelectric barrier a chopper provides a timing signal per cycle.

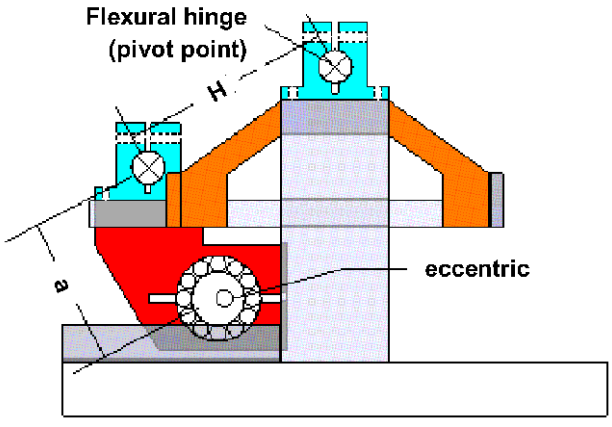

Fig. 2: Schematic of the drive mechanism

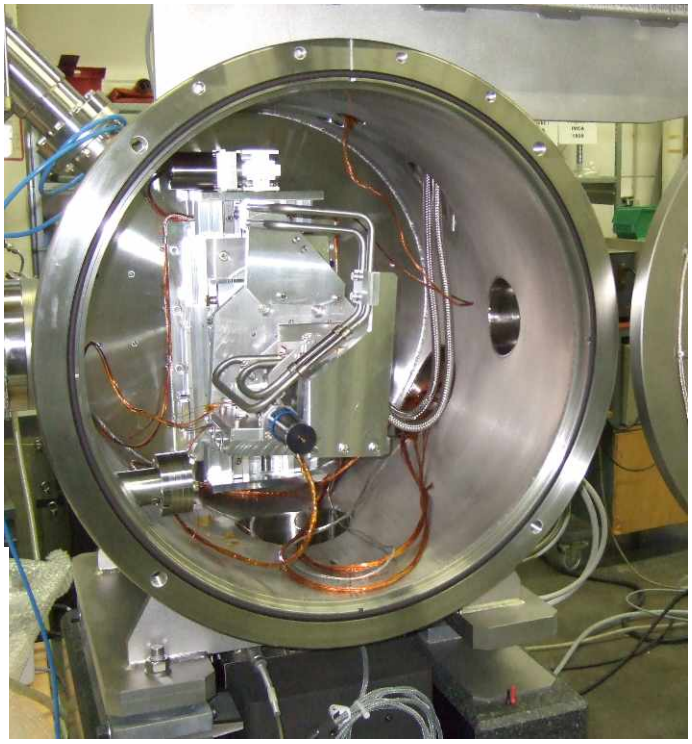

Fig 1: Bruker ASC monochromator for the SuperXAS beamline at the SLS

System Overview

Figure 3 shows the in-vacuum mechanics that are needed to control the monochromator crystal. It consists of a vertical tracking stage, a coarse goniometer to select the mean angle, i.e. the energy region near a specific excitation energy, and the oscillating cradle. Optionally an angular encoder can be connected to the cradle. The translation stage and goniometer are equipped with optical encoders. The channel-cut crystal orientation is available in Si(111) and Si(311). Together with the integrated water-cooling the system can manage thermal loads up to 200 Watts. Figure 1 shows the entire monochromator assembly as installed at the Super-XAS beamline. The vacuum vessel sits on a rigid granite support and can be manually aligned. The in-vacuum mechanics are rigidly mounted inside the vessel.

Fast Data Acquisition

Following the data acquisition that was originally developed at the University of Wuppertal, Bruker ASC has developed a stand-alone data acquisition system that is versatile to be integrated with any control environment at beamlines. The core package of the system is a high-end fast computer with PCI bus, large memory and a fast ADC board. The operating system is Linux and there is a phyton as well as EPICS interface. Combined with low noise ion chambers and amplifiers sampling rates of up to 100 kS/s are feasible. The maximum possible rate is 500 kS/s. However, such operation is very demanding on the detector electronics. The combination of detector and amplifier significantly influences the quality of the data at high speed. Please see separate product sheet for more information.

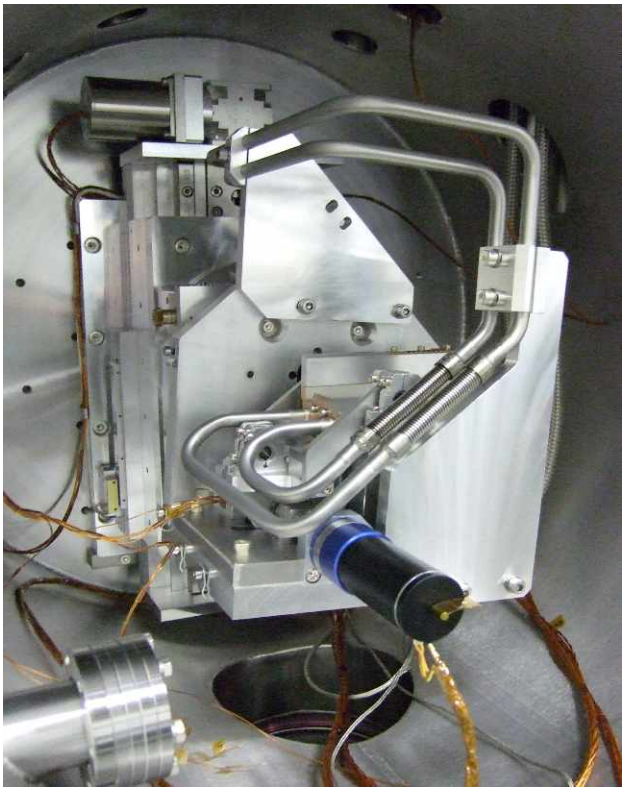

Fig 3: The in-vacuum mechanics incl. water-cooling

Basic scope of delivery:

- Massive granite support
- Vacuum chamber
- In vacuum adjustments
- Cradle mechanism for fast scanning
- Channel-cut crystal, water-cooled
- Full Testing
- On-site installation and support

Options:

- Variations of the eccentric mechanisms to cover different energy ranges
- Cryo cooled channel-cut crystal
- Fast data acquisition control system

Commissioning Results:

In March 2008 the monochromator for the SuperXAS beamline at SLS was commissioned. Within 24 hrs the system and data acquisition was integrated with the beamline, so that first users could start studies on catalytic reactions. In the figures 4 and 5 we present a comparison between two Cu K-edge spectra that were taken with our conventional monochromator on the fly (figure 4) and with our continuously quick scanning monochromator (figure 5). These data were taken with a Si(111) crystal. The scan range was 0.91 degree (approx. 800 eV). The scan speed of the QEXAFS monochromator allows taking full EXAFS scans in about 10 ms. However, such fast data accumulation requires a careful in-situ optimization of the detector electronic, we therefore have developed a special data acquisition system.

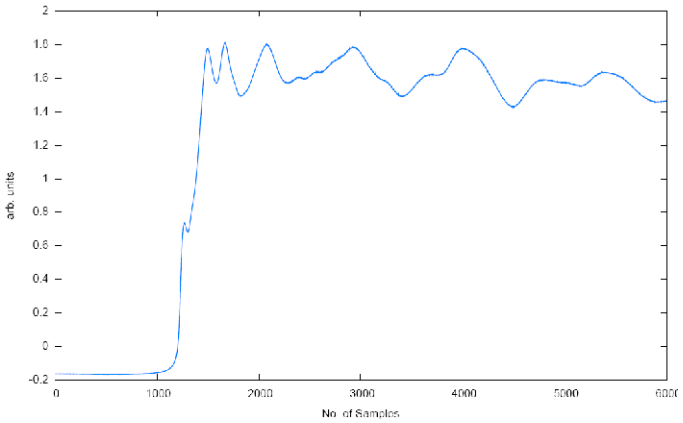

Fig 4: Cu K-edge data in 5 seconds using the conventional monochromator

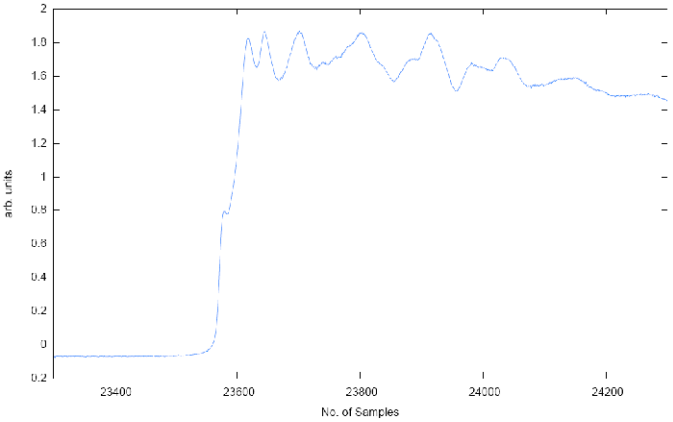

Fig 5: Cu K- edge data in less than 80 milliseconds using the Quick scanning monochromator

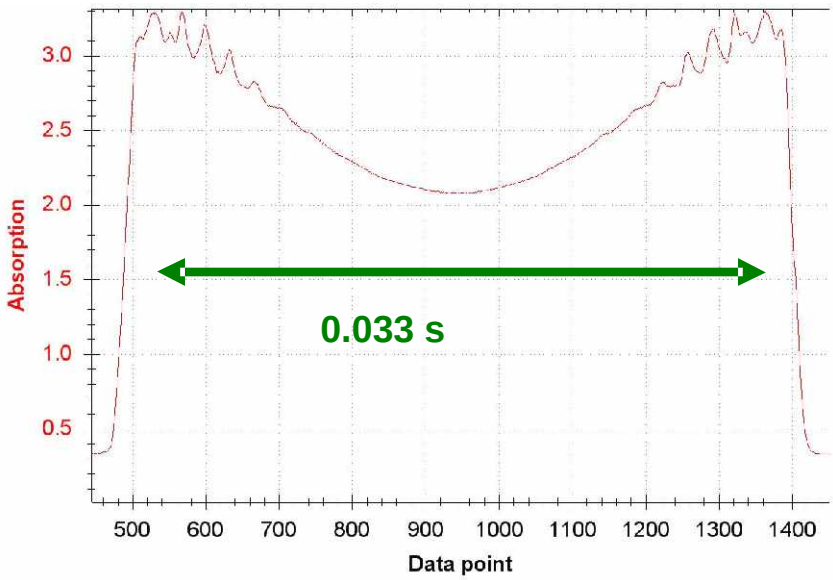

Fig 6: First try to measure at higher speed: Fe K-edge data measured in 33 milliseconds.

First Experiment after 24 Hrs of Commissioning:

After our quick commissioning scientist of the ETH Zurich were ready to study the catalytic oxidation of carbon monoxide using platinum particles as catalysts. With the combination of the Bruker ASC monochromator, the Bruker ASC fast data acquisition and the detector electronic of the research group of the University of Wuppertal, time dependent studies of above catalytic reaction could be performed within hours. Figure 7 shows a reference spectrum of a platinum calibration foil measured in 250 milliseconds. First measurements of a Pt catalyst are shown in figure 8. Meanwhile, many results are published in different journals.

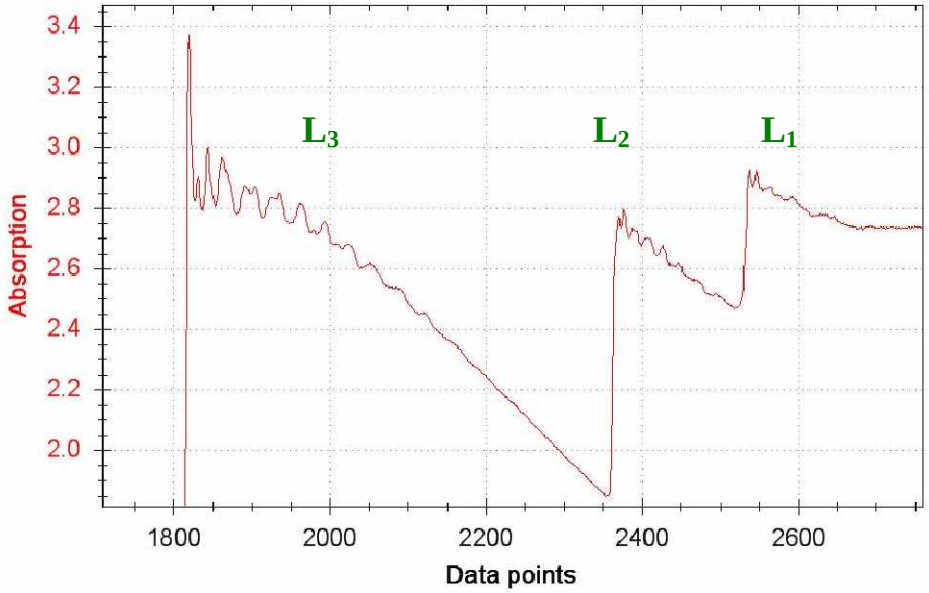

Fig 7: Pt L-edge data measured in 250 milliseconds.

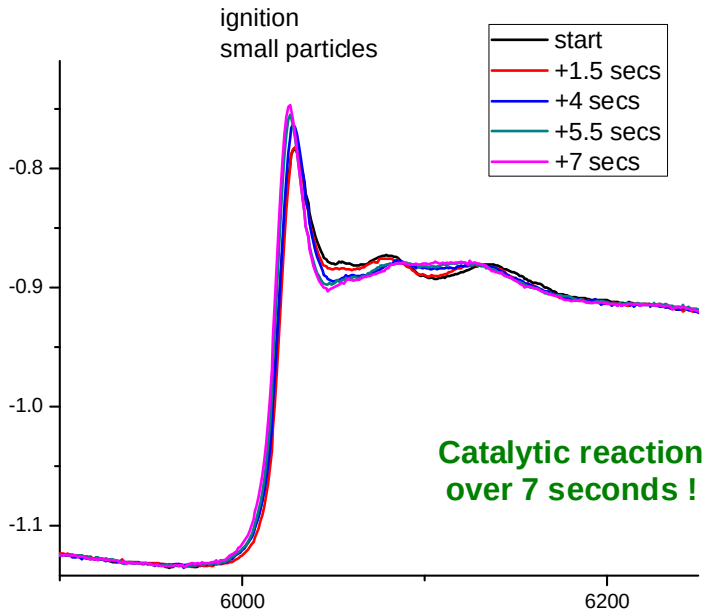

Fig 8: time dependent data of the Pt L_3-edge

Bruker ASC

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