New XAFS Facility for In-Situ Measurements at Beamline C at HASYLAB

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Abstract. A XAFS-experiment allowing for in-situ experiments has been set up at DORIS bending magnet beamline C. For that purpose, a new double-crystal, UHV-compatible monochromator with fast scanning capability was designed. This fixed-exit monochromator uses two crystal sets on a common central rotation axis driven by an ex-vacuo goniometer. Bragg angles range from 5° to 55.5° resulting in a total energy range 2.3 - 43.4 keV using Si(111)/(311) crystal sets. Crystal pairs can be remotely selected by translating the vacuum chamber. Energy encoding is performed using an optical encoder system. The standard XAFS sample environment is set-up in vacuo and can be adapted for special sample environments. For in-situ experiments, the beamline is equipped with twelve gas lines. An exhaust line allows toxic/reactive gases to be handled. As an initial performance test of the instrument, Ti, Cr, Fe and Cu XAFS and Ce K-shell QEXAFS measurements were performed.

Keywords: double-crystal fixed-exit monochromator, in-situ XAFS experiments.
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INTRODUCTION

Several beamlines at the Hamburger Synchrotronstrahlungslabor (HASYLAB) are dedicated to XAFS applications. The requirements of user groups are multifaceted with a steadily increasing demand of in-situ experiments and easily adjustable energy resolutions and ranges. To cover most user requirements and enlarge the available resources at HASYLAB, a new XAFS beamline was set up combining the possibility of in-situ experiments with a wide photon energy range. For that purpose a new compact double-crystal monochromator was designed which should provide a fixed-exit beam over a wide energy range suitable for step-by-step energy scans for conventional XAFS and continuous energy scans for QEXAFS measurements. The optical design is based on ideas proposed by Mills, King and Lee [1, 2].

MONOCHROMATOR DESIGN

Details of the design of the double-crystal monochromator are described in [3]. The most important features for the performance for XAFS measurements are as follows: The double crystal monochromator (Fig. 1) uses two crystal pairs mounted on one rotation axis driven by an ex-vacuo goniometer (Huber 420). Achievable Bragg angles θ range from 5° to 55.5° resulting in a total energy range of 2.3 - 43.4 keV by using Si(111)/(311) crystal sets. Crystal pairs can be remotely selected by translating the vacuum chamber relative to the beam. A change of crystal pairs including relative θ angle readjustment is implemented in the controlling software and takes less than 2 minutes. The fixed exit condition of the monochromator is realized by a vertical translation of the first crystal pair, which is also indirectly water-cooled. The second, short crystal may be translated in direction of the beam to fulfill the geometric boundary conditions at low angles and high energies. The use of a short second crystal on a translation stage instead of a long one as originally proposed by [1, 2] makes it easier to ensure perfect crystal properties. During energy scans, only the Bragg angle and the translation responsible for the fixed-exit condition are moved. The second crystal remains at the starting energy position to avoid mechanical instabilities.

For tuning of the monochromator, the second crystal is equipped with a two-axis cradle and a piezo-driven micro-goniometer. One cradle tilt enables an alignment of the crystal in ρ. This degree of freedom is responsible for the “fixed-exit” in the horizontal plane

and is especially important at large Bragg angles. The second axis allows a tilt relative to the global Bragg angle to align the two crystals.

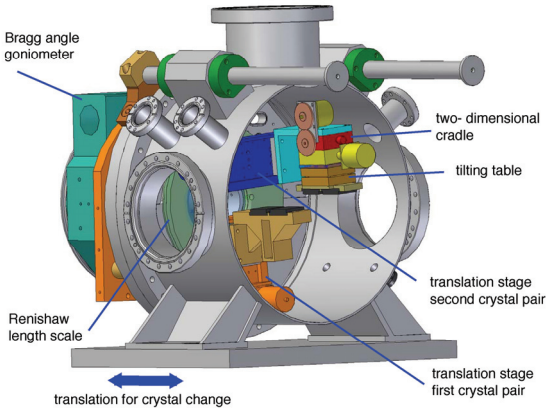

FIGURE 1. Schematic of the monochromator with the rear cover removed.

Detuning of the second crystal is achieved with a tilting table situated directly below the second crystal. The maximum tilt angle of this piezo-driven device is 600 µrad at 150 V. It is controlled by a digital monochromator feedback system (MOSTAB). Angular encoding of the Bragg angle goniometer is achieved using an in-vacuum Renishaw® [4] optical incremental encoder system and a scale attached to the circular adapter plate on which the translation stages of the crystals are mounted. This arrangement has the advantage that the encoded angle is recorded as close as possible to the crystals. The signal is transmitted to the control software, using a Beckhoff® [5] incremental encoder interface and bus terminal controller.

XAFS BEAMLINE

The double crystal monochromator is installed at 19.3 m from the bending magnet source. Mono-chromator and beamline vacuum are separated by a graphite window in front of the monochromator. Besides pumping through the beamline, a 300 L/s ion pump on top of the monochromator vessel keeps the vacuum inside the vessel at ~ $2x10^{-08}$ mbar without bake-out in "stand-by" mode i.e. without movement of motors. Towards the experiment, the monochromator vacuum ends at a Be window at 27.6 m from the source. The Be window is protected from oxidation by a ~ 0.5 m long He-rinsed area with a final capton window.

A 10 mm thick water-cooled adjustable copper block at 0.6 m from the first monochromator crystal acts as a beam stop. Collimation of the beam is achieved with two slit systems in front of the monochromator and a third one in the experimental hutch at 28.8 m from the source, directly in front of the first ionization chamber.

The XAFS experiment is installed on a 2.4 m long support table. It is movable in height with three independent translation stages. Two translation stages move the table in the direction perpendicular to the beam. The three ionization chambers have a length of 10 cm and entrance windows of 6 mm in height and 20 mm in width. Pressures and gases may be adjusted individually for each ionization chamber to achieve the desired absorption level for the used energy range. Between the first and the second pair of ionization chambers, two ISO K 160 double crosses are installed as sample chambers. The standard sample holder can carry 6 pellets with a diameter of 13 mm and can be mounted directly in the sample chamber. A pressure of 10^{-6} mbar is realized inside the sample chambers using a turbo pump. The standard sample chambers can be removed to accommodate special sample environments like an oven or in-situ cells. Besides four lines for inert gases, which are also used for the filling of the ionization chambers, eight additional lines for user gases are available at the beamline allowing in-situ experiments. An exhaust line allows the handling of toxic/reactive gases.

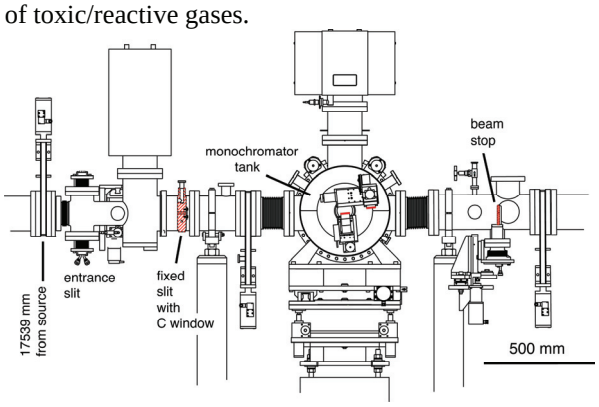

FIGURE 2. Schematic showing the monochromator integrated into beamline C at DORIS III.

In addition to the detection in transmission mode, several solid-state fluorescence detectors and a total electron yield detector are available on request. For measurements at liquid nitrogen temperature or temperatures between 4 K and room temperature, a liquid N_2 respectively liquid He cryostat are available upon request.

PERFORMANCE

Several initial performance tests were done to characterize the XAFS capabilities of the beamline.

Among those were verification of the design energy ranges, the energy resolution of the device, the performance of the monochromator in continuous scan mode i.e. for QEXAFS measurements as well as the stability of the double-crystal monochromator during repetitive scans.

The calibration curve of the optical encoder was established by measuring absorption edges of four elements at significantly different Bragg angles. The K-edges of the elements Ti, Cr, Fe and Cu were chosen, covering an energy range from 5 to 9 keV and corresponding Bragg angles of 23.5 to 12.7° when using Si(111) crystals. From the measured absorption edge position [6], the corresponding Bragg angle was calculated and plotted against the determined Renishaw increments (Fig. 3). Based on this calibration curve, energies within the range of 4.9 keV to 9 keV may be determined without absorption edge reference measurements to a precision of better than 0.5 eV.

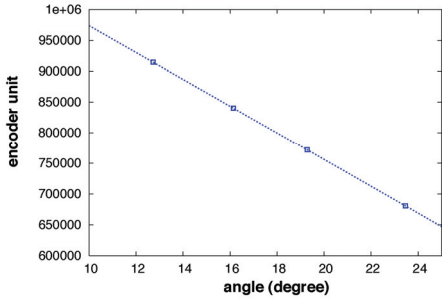

FIGURE 3. Experimentally determined calibration curve of Bragg angles using encoder values.

The energy resolution of the instrument was tested in back-reflection of a Si(111)-wafer attached to the exit window of the first ionization chamber. The diffraction peak of Si(333) at 5933 eV was scanned in stepping mode with energy steps of 0.05 eV. The full width at half maximum of the rocking curve was determined by a Gaussian fit. Divided by energy, it is a measure of the energy resolution ($\Delta E/E$) of the set-up and it was better than 8.5×10^{-5} for the Si(311) crystal pair and an exit slit opening of 1 mm. The procedure was repeated for Si(444) at 7908 eV. At the higher energies, $\Delta E/E$ was 1.26×10^{-4}.

To demonstrate the performance of the beamline at high energies and especially in continuous scanning mode, a Ce K-edge spectrum from Ce_2O_3 powder is shown in Fig. 4a. The energy scan range was 1300 eV and the sampling time of the QEXAFS scan was 220 sec. The scan was repeated five times and the merged spectrum combining the five scans is compared with the single scan (Fig. 4b). The tests have shown that the K-shell of Ce may be measured with good statistics and energy resolution up to a k value of 15 $Å^{-1}$ on a short time-scale.

Repetition precision of 15 step-by-step Cr K-edge scans revealed a stability of the position of the absorption edge better than 0.06 eV with an average energy shift of 0.032 eV.

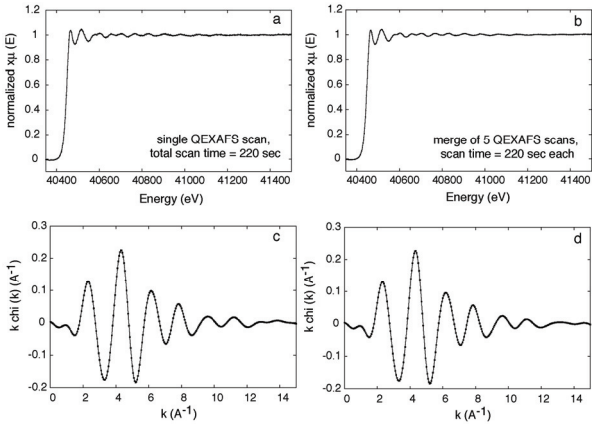

FIGURE 4. Cerium K-edge absorption spectrum (background subtracted and normalized) measured from a pressed pellet of Ce_2O_3 oxide in transmission using Si(311) in the continuous scanning mode. Results from single scan (a) and merge (b) of 5 scans and corresponding kchi(k) (c and d, respectively).

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