Compact Fixed-exit UHV DCM for XAFS

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Abstract. A double-crystal, UHV-compatible monochromator for XAFS applications at bending magnet beamlines has been designed. It uses two crystal sets, Si(111) and (311), on a common central rotation axis driven by an *ex-vacuo* goniometer. All mechanical and electrical components are mounted on a 400 mm UHV flange which is attached to a compact vacuum chamber. The first crystals are water cooled using connector- and bellowless tubing through the fluidic sealed feedthrough of the central rotation. The first crystal set is mounted off-axis and can be translated vertically to keep the fixed exit condition. The second crystal set uses small crystals of the same size as the first. In order to accept the reflected beam of the first crystal at small Bragg angles, it is tangentially translated along the beam. The angle can be varied from 5° to 55.5° resulting in a total energy range 2.4 - 43.4 keV for Si(111)/(311). Crystal sets are interchangeable by translating the vacuum chamber. Angle encoding is achieved by a Renishaw incremental optical encoder *in vacuo*.

Keywords: Double crystal monochromator, XAFS

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INTRODUCTION

At the DORIS III storage ring, several bending magnet beamlines are dedicated to X-ray absorption spectroscopy. Depending on the beamline optics and the main fields of application, the energy range is either preferentially "soft", i.e. starting from the Sulphur K-edge at 2.4 keV going up to approximately 11 keV, or "hard", i.e. from around 5 keV up to beyond 40 keV. High photon energies are available at DORIS III bending magnets with considerable flux due to their high critical energy ($E_c = 16.6$ keV). The beamlines and monochromators have been implemented many years apart and their layout is quite different. Based on the long standing experience with a variety of different double crystal monochromators at the laboratory, the goal was to design a new device which incorporates as many of the advantages and features of the other instruments as possible. At the same time, it should be compact and UHV-compatible to allow its straightforward integration into existing bending magnet beamlines without their major modification.

DESIGN AND PERFORMANCE

The device should be used primarily, but not exclusively, for XAFS experiments at bending magnet beamlines. Therefore, the main design criteria were a wide energy range, fast tunability as well as fixed-exit operation. To cover the energy range from just below the Sulphur K-edge and to above 40 keV, it seemed advisable to implement two different Si crystal pairs, (111) and (311), interchangeable *in-situ* by remote control. Fast and precise tunability is best reached using a common rotation stage for both crystals which has proven to be suitable for fast continuous scanning absorption measurements (QEXAFS)[1]. A corresponding (non fixed-exit) monochromator has been in operation at HASYLAB for many years [2] and is very successfully used for in-situ catalysis XAFS studies.

For the new device, an optical design proposed by Mills, King and Lee [3, 4] was adapted which allows fixed-exit operation. This is accomplished by an off-rotation-axis mount of the first crystal, which is translated perpendicular to the beam. The original design uses a fixed second crystal which is long enough to reflect the beam at all accessible Bragg angles. Here it was decided to use a shorter crystal and to implement its tangential translation along the beam instead. A short crystal makes it easier to ensure perfect crystal properties across the entire surface. The sizes used here are 40 mm x 40 mm for both crystal pairs. It is noted that during a typical XAFS energy scan covering 1-2 keV, the second crystal is not moved to minimize mechanical instabilities. Prior to an energy scan the crystal is positioned appropriately by the software to maximize the available scan range (for decreasing Bragg angles during the scan). The Bragg angle range was limited to 5° - 55.5° which corresponds to an energy range from 2.4-22.6 keV for Si(111)

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FIGURE 1. Schematic of the monochromator, shown in its "service" position. All components are attached to an O-ring sealed 400 mm UHV-flange (1). The outside goniometer (2) is coupled by a hollow-shaft ferrofluidic® sealed feedthrough to the common rotation *in-vacuo*. The first crystal pair (3) can be moved perpendicular to the beam and is indirectly water-cooled. The second crystal pair is mounted on a two-axis cradle (4) for alignment relative to the first, the unit can be translated along the beam. A piezo-driven micro-goniometer (5) allows to fine tune the relative Bragg angles. The angle position is read out by a Renishaw® optical encoder system (6).

and 4.6-43.4 keV for Si(311). To keep the dimensions of the vacuum chamber small, the central rotation is driven by a goniometer (Huber 420) from the outside, coupled by a hollow-shaft ferrofluidic® vacuum feedthrough [5] with 70 mm inner diameter (see Fig. 1). Precise angular encoding is achieved by an *in-vacuo* Renishaw [6] incremental optical encoder system. All mechanical components are mounted on the central rotation *in vacuo*, electrical connectors and feedthroughs are provided on the common 400 mm UHV flange. The entire unit can be easily retracted for maintenance.

The first crystal pair is indirectly water-cooled. The thermal coupling between the crystals and the copper heat sink is achieved by Ga-In eutecticum (Gallistan®). Side-cooling was not considered necessary because thermal gradients are small (total power on the crystal ≈ 100 W) and the simpler design was preferred. The cooling water supply was designed without water-to-vacuum joints such as brazing and connectors (Fig. 2). In order to enable crystal height tuning without the use of bellows, the flexibility of the (comparatively long) copper tubings is utilized which pass through the hollow shaft of the rotational axis.

FIGURE 2. First crystal stage with attached indirect water cooling (Si(111) and (311), crystal size $40 \times 40 \text{ mm}^2$). The tubing passes through the hollow shaft rotational axis to the left.

FIGURE 3. Schematic showing the monochromator (1) integrated into beamline C at DORIS III. The outer flange-to-flange distance of the vacuum chamber is 500 mm allowing it to be mounted onto a standard HASYLAB mirror chamber support (2) which provides additional degrees of freedom such as translation perpendicular to the beam (for crystal change) and height adjustment. The incoming white beam is passing through a fixed width carbon window (3) to separate the vacua and to cut off the low energy spectrum. Behind the monochromator, a water-cooled adjustable absorber (4) with a pinhole opening is used to either block the direct beam if the crystals are used for monochromatization or to pass the white beam through the pinhole on to the experiment if the crystals are moved out of the beampath.

Because of its compactness the monochromator vacuum chamber easily fits onto a standard (HASYLAB) mirror chamber support which offers translational (and optionally also rotational) degrees of freedom (Fig. 3). Therefore, it is straightforward to bring the different crystals into the beam by translating the vacuum chamber (50 mm) which is connected to the beamline by flexible bellows. Inlet and outlet flanges are large enough (150 mm conflat) to pass the beam through in both positions. The chamber height may be adjusted as well although this is only necessary once during the initial alignment. A complete crystal change, including relative Θ -angle re-adjustment takes about 2 minutes and is remotely done by the controlling software. An adjustable absorber with a pinhole behind the monochromator allows to pass the white beam on to the experiment. Therefore, both crystals can be easily moved out of the direct beam. A Ta wire, attached to the absorber, can be scanned through the monochromatic beam to measure its position.

In addition to pumping through the beamline, a 300 l/s ion pump on the top of the monochromator vacuum vessel is used to maintain a base pressure of $3 \cdot 10^{-8}$ mbar without bake-out in "stand-by" operation, i.e. when no motors are moving. During motor movements the pressure will increase slightly because of additional degassing due to components warming up.

The initial performance tests of the instrument were very promising. While scanning, the relative detuning of the crystals is controlled by a digital monochromator feedback system (MOSTAB). The piezo driven micro-goniometer (maximum detuning $600 \mu\text{rad}$) was mounted onto the second crystal stage, instead of the first one, to enhance the relative energy stability in the monochromatic beam under detuning conditions [7, 8].

The monochromator has been designed for fixed-exit operation which is - of course - achieved for conventional step-by-step scanning; the offset between white and monochromatic beam is 20 mm. For fast scanning measurements (QEXAFS) the motors are continuously moving and their positions are recorded "on the fly". Unfortunately, the present motor controllers cannot be programmed with non-linear step profiles. Therefore, all motors move linearly which causes a certain deviation from the fixed-exit condition. This is shown for two typical examples, Ti and Cu K-edges, in Fig. 4. It is noted that for small Bragg angles the effect becomes practically negligible. However, at large Bragg angles, e.g. at the S K-edge, the deviation is significant (may exceed 1 mm) so that continuous scanning may be problematic.

FIGURE 4. Deviation of the exit beam height during *continuous* scanning in the range of two typical K-absorption edges, Ti K-edge scanned with Si(111) (range: 4958 to 6011 eV) and Cu K-edge scanned with Si(111) and Si(311) (range: 8958 to 10760 eV). At the larger Bragg angles the maximum relative offset is below 20 μm and it becomes practically negligible ($< 4\mu\text{m}$) in the case of the Cu K-edge measured with Si(111).

To demonstrate the performance of the monochromator, a Cu metal K-edge spectrum obtained in the continuous scanning mode is shown in Fig. 5. The initial tests have shown that it is indeed performing very well as expected and it is planned to gradually replace some of the older systems at DORIS III bending magnet beamlines with this type of instrument.

FIGURE 5. Cu K-edge absorption spectrum (background subtracted and normalized) measured from a metal foil in transmission ($\mu d = 2.3$) using Si(111) in the continuous scanning mode. The sampling time per data point is 35 ms, the total scan time ≈ 30 s.

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