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An exchangeable Bragg/Laue polychromator for energy-dispersive XAFS

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An original design for a cooled exchangeable polychromator for energy-dispersive XAFS (X-ray absorption fine structure) working either in the transmission configuration (Laue case) or in the reflection configuration (Bragg case) is presented. It enables the acquisition of X-ray absorption spectra between 5 and 25 keV with a spot size on the sample that can reach to less than 20 μm FWHM for some energies. Only 1 h is needed to exchange both benders in operative mode. Parallel transmission spectra with a bandpass between 5 and 10% can be obtained in the full energy range. The dispersive optics and mechanics of ID24 (ESRF, Grenoble, France) have been designed to obtain XAFS spectra in less than 1 s and, in some cases, in the millisecond range.

Keywords: XAFS; monochromators; energy-dispersive.

1. Introduction

Energy-dispersive X-ray absorption spectroscopy enables parallel collection of transmission spectra (Matshshita & Phyzackerly, 1981; Flank *et al.*, 1983). At station ID24 (Hagelstein *et al.*, 1997), the white beam from a tapered undulator is reflected by two 1 m-long mirrors in the Kirkpatrick–Baez configuration (Kirkpatrick & Baez, 1948), reducing the thermal load and eliminating high-order harmonics. The second mirror enlarges the horizontal size of the beam to 40 mm at the position of the polychromator. There, the Laue or the Bragg polychromator establishes an energy–angular correlation and focuses the beam onto the sample. In the 2θ direction, a position-sensitive detector based on a CCD (Koch *et al.*, 1996) converts this correlation into an energy–position correlation. XAFS spectra are then recorded simultaneously for all energies in a single X-ray shot.

To satisfy the best energy resolution requirements without limiting the energy bandwidth too much, Si(111) crystals are used in Bragg geometry when working at energies from 5 to 12 keV and in Laue geometry from 12 to 25 keV (Hagelstein, Ferrero, Hatje *et al.*, 1995). The crystals have to be bent with a radius of curvature ranging from 1 to 20 m and as close as possible to an elliptical profile to avoid X-ray optical aberrations. Vacuum conditions have to be combined with the capabilities of orientation in three different axes (the diffraction angle and two tilt movements). In addition, a twist correction of the crystal must be included to obtain the best focusing performances. An efficient cooling system to evacuate the thermal load (up to 50 W) is also included.

2. Technical description

2.1. *Goniometer*

The polychromator assembly comprises the goniometer (Fig. 1), which is responsible for accurately positioning the crystal and holding the benders, the benders (Figs. 2 and 3), which give the crystal the requested radius of curvature, and a large vacuum vessel equipped with a Be window (Fig. 1) that houses the benders and creates the necessary vacuum environment (in the low 10^{-6} mbar range). The goniometer sits outside the vacuum vessel while the bender is supported on the goniometer by a specially developed differentially pumped rotary feedthrough. The polychromatized beam exits the vacuum vessel through a wide polished Be window covering a total angle of 120° around the polychromator vertical axis. Special care was taken when polishing the 250 μm-thick window in order to suppress the generation of unwanted contrast due to the interference of partially coherent wavefronts at rough surfaces. The Bragg angle of the goniometer can be varied from 0° to 360°, whereas the two tilt angles, χ and ψ, have a stroke of ±2° with a resolution of 0.01°. The goniometer supports the bender *via* the differentially pumped rotary feedthrough, which is itself cooled to stabilize its temperature and dimensions. The feedthrough is a 30 mm-diameter rod entering the vacuum vessel and sealed by two V-ring polyimid gaskets. The volume between the two gaskets is pumped by a membrane pump. By design, the ±2° circle segment positioners are exactly centred on the polychromator crystal centre.

2.2. *Benders*

In the Laue configuration the bender (Fig. 2) is based on the monolithic flex-pivot structure principle, already developed and tested at the ESRF (Hagelstein, Ferrero, Sanchez *et al.*, 1995). The bending of the crystal is induced by two independent micrometre

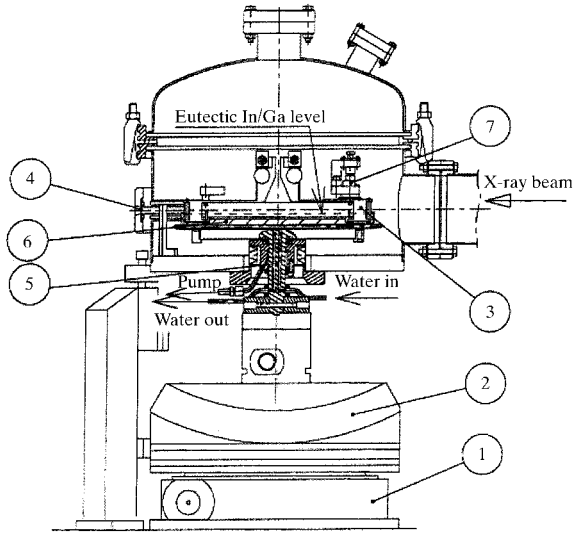

Figure 1
Assembly drawing showing the Bragg bender installation. (1) θ circle, (2) χ and ψ circles, (3) Bragg bender, (4) Be window, (5) rotary feedthrough, (6) eutectic cooling bath, (7) tilt piezo-adjustement.

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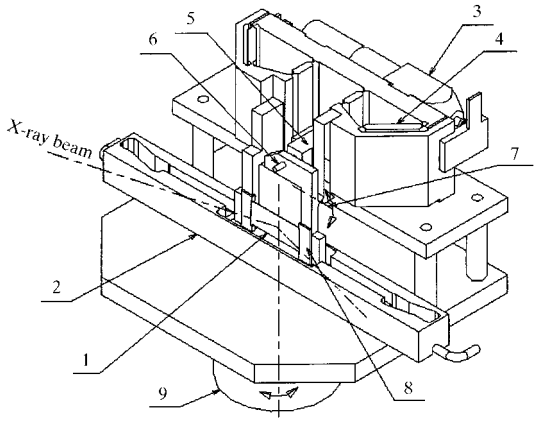

Figure 2
Dispersive Laue bender. (1) Laue crystal, (2) eutectic bath, (3) micrometric jacks, (4) monolithic bending system, (5) piezoelectric actuator, (6) its adjustment and (7) weak-link lever. The crystal is directly clamped, (8), to its support. The θ circle is indicated, (9).

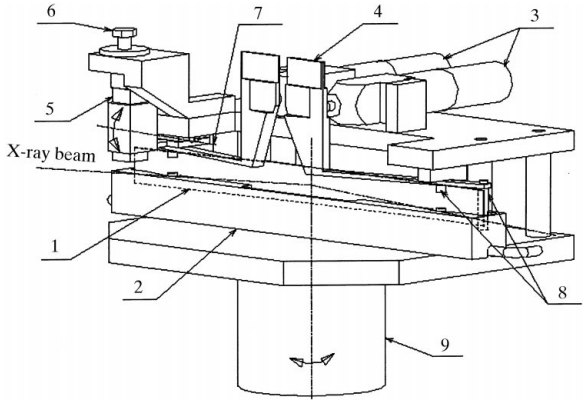

Figure 3
Dispersive Bragg bender. The 30 cm-long crystal, (1), has one-third of its vertical dimension immersed in the eutectic bath, (2). Two micrometric jacks, (3), apply the bending force through the lever arms, (4). A piezoelectric actuator, (5), with a manual initial adjustment through a differential screw, (6), allows the correction of the twist *via* a weak-link lever, (7). The two right rollers, (8), of the four-point bending system are indicated, as well as the axis of Bragg rotation, (9).

screws actuated by two stepper motors. The device allows cylindrical curvature and close to elliptical curvature. The twist correction is introduced *via* a piezoelectric actuator with a 40 µm stroke acting on a weak link. The size of the supported crystal is 60×20 mm with a typical thickness of 120 µm.

In the Bragg configuration (Fig. 3) the 30 cm-long and 1 mm-thick crystal is held between two pairs of rods situated at both extremities in what is called the four-point bender system (de Wolff, 1968). This method has already been implemented in other energy-dispersive XAFS experiments (Allen *et al.*, 1993; Lee *et al.*, 1994; Brauer *et al.*, 1996). In our set-up, the bending is induced by means of two lever arms pushed by stepper-motor-driven micrometre screws. In order to achieve a close-to-perfect elliptical curvature of the crystal, corrected for aberrations, its width follows a calculated profile (Allen *et al.*, 1993; Pellicer-Porres *et al.*, 1998). A twist correction of the crystal is obtained by means of a piezoelectric system similar to in the Laue configuration and a differential screw pre-adjustment (0.05 mm differential pitch). Because of the length of the crystal, the twist correction has proved to be extremely useful in the Bragg case, not only to correct an eventual twist of the crystal but also to correct those that could be induced by the upstream optics.

For both systems, cooling is obtained by immersion of one-third of the crystal width in a water-cooled liquid eutectic In/Ga alloy bath that has a vapour tension well under the operating pressure of the polychromator. The resulting temperature gradient along the height of the crystal is established in the direction perpendicular to that of the energy dispersion. To allow for maximum freedom of the different movements, the cooling circuit of the In/Ga bath is also supplied with water at 292 K from the cooling circuit flowing through the rotary feedthrough. As a result, no force other than the bending forces is applied to the crystal in any of its positions.

The base support that holds one or the other bender is the same, as well as all electrical connectors, enabling rapid exchange between both systems.

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