A Beamline for Macromolecular Crystallography in ALBA

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Abstract. ALBA is a third generation 3 GeV storage ring being built near Barcelona and foreseen to be operational in 2010. Out of the seven beamlines already funded in ALBA, one will be dedicated to macromolecular crystallography (MX). The beamline, dubbed XALOC, shall cope with a broad range of crystal structures and sizes. To this aim, a flexible optical design involving variable focusing optics has been incorporated into the beamline optics. The photon source will be a 2 m long, in-vacuum undulator with a period of 21.3 mm. The optics will consist in a Si(111), double-crystal monochromator cryogenically cooled, and a pair of mirrors placed in a Kirkpatrick-Baez configuration. The beamline will deliver a high flux beam in the 5-15 keV energy range, with an energy resolution of $\Delta E/E \sim 2\times10^{-4}$. In addition to the main beamline, it is being considered the possibility to use a diamond laue monochromator to provide photons at a fixed wavelength to an ancillary branch. This report shows the present status of the beamline design.

Keywords: Beamline, Macromolecular crystallography, ALBA, CELLS.
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INTRODUCTION

ALBA synchrotron, managed by the CELLS consortium (Consortium for the Construction, Equipment and Exploitation of the Synchrotron Light Source), will be located near Barcelona. This is a 3 GeV, 4.5 nm·rad emittance storage ring able to run in top-up mode, and operating with users at a current of 250 mA, although the ring is designed to store 400 mA [1]. One of the seven beamlines to be implemented in the first phase will be devoted to macromolecular crystallography (MX) experiments. The beamline, called XALOC, has to be able to cope with large complexes, which usually crystallize in large unit cells. At the same time the more conventional work involving small crystals has to be ensured to satisfy the needs of the user community. To this aim, a flexible optical design involving variable focusing optics has been considered into the beamline optics.

The requirements to be fulfilled by the beamline are listed in table 1. The guide lines in the design have been the simplicity of components and the beam stability, which should be granted through efficient beam monitoring. The experimental station, which is out of the scope of this report, will include all the equipment needed to perform automated MX experiments. XALOC is now in the procurement phase, and the commissioning is planned mid 2009.

TABLE 1. General requirements to the beamline.

Source	In-vacuum undulator
Main optics	Symmetric Si(111) monochromator + KB focusing system
Photon energy range	5 – 15 keV
Photon flux at sample	10^{12} ph/s in 0.1×0.1 mm^2
Energy resolution	$\Delta E/E \sim 2\times10^{-4}$
Energy stability	± 0.1 eV for 3 hours
Beam size at sample (FWHM)	Adjustable 50-200 μm × 20-100 μm (H×V)
Beam divergence at sample (FWHM)	<0.5 mrad, < 0.2 mrad for large unit cells

SOURCE

The photon source of the XALOC beamline is an in-vacuum undulator [2], placed in the 5^{th} medium straight section of the ALBA storage ring (Table 2). The undulator is designed so that the energy of the 7^{th} harmonic is close

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to the Se K-edge (12.658 keV) at minimum gap. This criterion optimizes the undulator at the most used energy in MX experiments, while providing good tunability, high brilliance and flux density in the whole useful energy range.

The front-end accepts the beam in an angular aperture of 0.4×0.2 $mrad^2$ (H×V), transmitting 1.48 kW of power out of the 2.87 kW emitted by the undulator when the current of the storage ring is 400 mA. The angular aperture can be further reduced by the cooled white beam slits, which are included in the front-end.

TABLE 2. Source parameters.

Type of ID	FeCo pure permanent magnet, in-vacuum undulator
Period	21.3 mm
Number of periods	92
Deflection parameter (K) at minimum gap (5.5 mm)	1.5949
Photon source size (FWHM)	309 × 18 μm^2 (H×V)
Photon source divergence (FWHM)	112 × 28-22 μrad^2 (H×V)

OPTICS

XALOC optical elements are a CVD-diamond vacuum window, a removable diamond filter, a channel-cut Si(111) monochromator and a Kirkpatrick-Baez system (Fig. 1). The 300 μm thick, CVD-diamond vacuum window separates the beamline vacuum from the front-end and the storage ring, and filters the radiation arriving to the optical elements placed downstream. After the vacuum window, the beam is further filtered by a 300 μm thick diamond crystal, which will be removed from the beam path when working at energies below 7 keV to avoid excessive absorption. The filter could be operated also as a Laue monochromator to feed an ancillary branch. The properties of this branch, whose implementation is still under discussion, are discussed below.

The beam is monochromated by a symmetric Si(111), channel-cut monochromator, with a narrow gap between crystals (<6 mm) to reduce the variation of the vertical beam offset to less than 1 mm within the useful energy range. The monochromatic beam is focused onto the experimental set-up (either the sample, the detector, or at some point along the optical axis nearby) by a vertical focusing mirror (VFM) and a horizontal focusing mirror (HFM) in a Kirkpatrick-Baez configuration [3]. The VFM and HFM, meridionally bent in an elliptical cylinder shape, focus the beam with a demagnification of 3.84 and 6.58, respectively. The incidence angle of the beam onto the mirrors, which are coated with Rh, is 4.1 mrad. The reflectivity is high in the energy range of interest and shows a cut-off just above 15 keV. The useful optical length of the mirrors is 300 mm and 600 mm for the VFM and the HFM, respectively, which allow collecting the whole beam vertically and one FWHM horizontally.

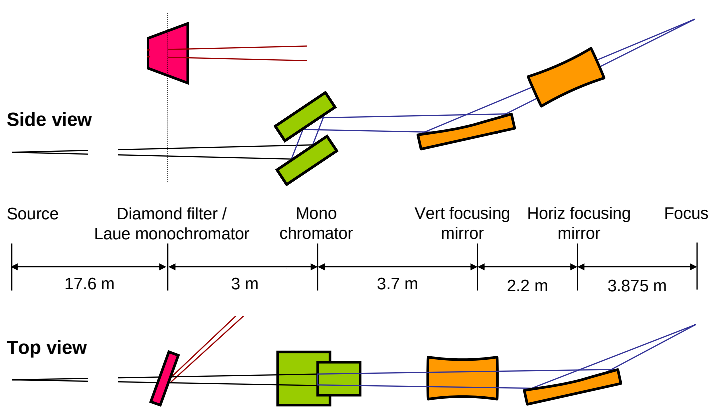

FIGURE 1. XALOC layout (vacuum window not shown). Diamond filter could be used as Laue mono for an ancillary branch.

BEAM CHARACTERISTICS

Regarding spot sizes, in the absence of slope errors, raytracing calculations using SHADOW [4] through a Matlab interface show that the focal spot size is 52×5 μm^2 (H×V) FWHM (Fig. 2), which agree with analytical calculations. The detector is assumed here to be in the focal position, so the spot size at sample will then change upon the sample-detector distance. For example, to have data at 2 Å resolution using photons at Se K edge and a detector diameter of 315 mm, the sample-detector distance must be 379 mm, resulting in a beam size and divergence at sample of 195×33 μm^2 and 0.57×0.09 mrad^2 FWHM, respectively. The horizontal divergence is limited by the acceptance of the HFM. Beam dimensions, calculated at 12.658 keV, are almost independent of the photon energy.

Beam size will be adjusted to crystal dimensions by focusing the beam out of the sample position, preferably close to the detector. However, when working out of focus, the beam profile is very sensitive to the long-period slope errors of the mirror surfaces. Homogeneous beam profile, which is important to uniformly expose the crystal, is especially difficult to reach in the vertical plane due to the small vertical emittance. Raytracing (not shown) shows that the beam homogeneity is specially affected by slope errors with periods larger than 20 mm in the vertical plane and larger than 75 mm in the horizontal one. In case the vertical unhomogeneities affected the quality of data, the VFM would be removed from the beam path. The vertical beam size would then increase to 710 μm FWHM, but would preserve the gaussian profile given by the source. Beam should be adjusted using slits close to the sample.

The flux of the beamline is calculated to be above 3×10^{12} ph/s in the whole energy range (Fig. 3). The energy resolution, contributed by the source divergence in the dispersive direction and the Darwin width of the crystal, is less than 2×10^{-4} in the 5-15 keV energy range, as agreed by both raytracing and analytical calculations (not shown).

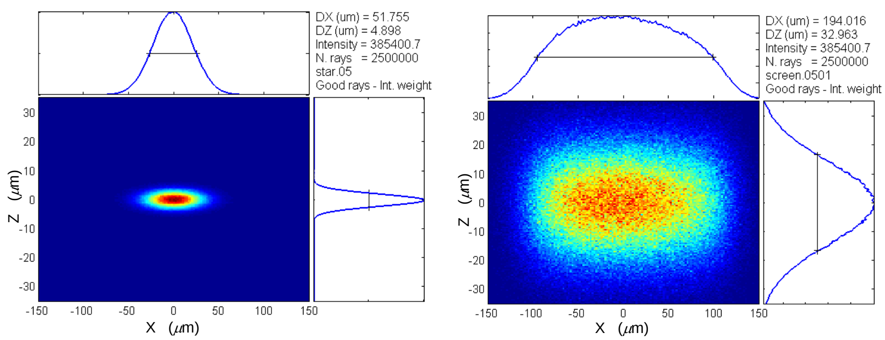

FIGURE 2. Simulated beam spots at focus (left), and 379 mm before (right). Photon energy is 12.658 keV.

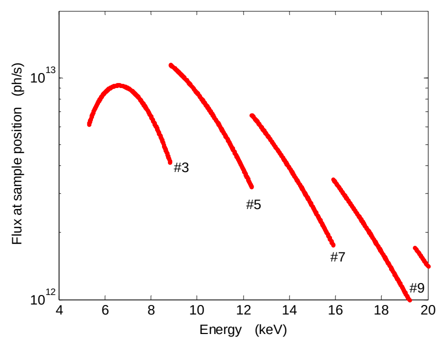

FIGURE 3. Calculated flux at sample position assuming a current of 250 mA in the storage ring. Calculation takes into account filter transmission (300 μm thick, diamond), monochromator bandpass, and reflectivity and acceptance of the mirrors.

POWER LOAD

The power and power density absorbed by the vacuum window, the diamond filter and the monochromator are listed in table 3 in worst-case and working conditions. In the worst case, the white beam slits are fully open accepting all the beam transmitted by the front end at 400 mA in the storage ring. More realistic, working conditions assume a current of 250 mA and the slits being matched to the acceptance of the mirrors (112×54 μrad^2).

Regarding the vacuum window, recent measurements done at ESRF [5] show that a 300 μm thick CVD diamond of a diameter of 6 mm can withstand total absorbed powers of ~500 W and absorbed power densities of ~70 W/mm^2 using water cooling. These values are much higher than those calculated for this beamline at working conditions, indicating that the use of such vacuum window is feasible in XALOC beamline. However, total absorbed power can still be a concern under worst-case conditions. Detailed Finite Element Analysis is needed to clarify this point.

The calculated power absorbed in the monochromator under working conditions is less than 50 W, whereas maximum power density is 9 W/mm^2 at 5 keV. This heat load is well handled by Si crystals with indirect cryocooling systems without introducing a significant thermal bump [6, 7]. In the worst-case conditions the power absorbed represents a tougher requirement, but is still manageable as referenced experiments have already proved.

TABLE 3. Absorbed power and power density on optical elements.

	Working conditions with diamond filter	**Working conditions without diamond filter**	**Worst case conditions**
	250 mA, slits matched to acceptance of mirrors	250 mA, slits matched to acceptance of mirrors	400 mA, full front-end acceptance
Incoming power	91 W	91 W	1482 W
Power absorbed at the vacuum window	47 W (27.7 W/mm^2)	47 W (27.7 W/mm^2)	885 W (44.3 W/mm^2)
Power absorbed at the diamond filter	9 W (4.6 W/mm^2)	—	143 W (7.4 W/mm^2)
Power absorbed at the monochromator	35 W (5.6 W/mm^2)	44 W (7.0 W/mm^2)	454 W (9.0 W/mm^2)

ANCILLARY BRANCH

A diamond single crystal of good crystalline quality in the diamond filter produces X-ray diffraction in Laue geometry which can be used to provide photons to a side, ancillary branch. The implementation of such branch in XALOC, which is feasible in terms of floor availability, is under discussion.

No further optics other than the Laue monochromator is foreseen for this branch. As the Laue monochromator is dispersive, it would be set up at a fixed wavelength of 1.371 Å (9.041 keV), which is that of the 5th harmonic of the undulator when the 7th harmonic is tuned at the Se K-edge. Although the ancillary branch can not be used optimally when tuned otherwise, the undulator is likely to be set at this energy during a large fraction of the beam time. Using this wavelength and the strong (111) reflection, the beam collected by the ancillary branch is diffracted at 2θ = 38.90º. Using a diamond crystal cleaved in the [100] direction, the asymmetric cut angle is then 54.736º, and the tilt angle with respect to normal incidence is 15.815º.

The ancillary branch also exploits the focusing properties on polychromatic beams of the Laue monochromator [8]. The diffracted beam focuses approximately at 9 m after the Laue monochromator onto a spot of 0.75×0.55 mm^2 and a divergence of 135×24 μrad^2 FWHM (Fig. 4). The energy resolution, $\Delta E/E = 3.3\times10^{-4}$, can be readily improved by closing the horizontal slits, as it depends on the horizontal divergence. The flux is calculated to be 6.7×10^{12} ph/s. The beam is appropriate for testing instrumentation and for measurements at a fixed-wavelength.

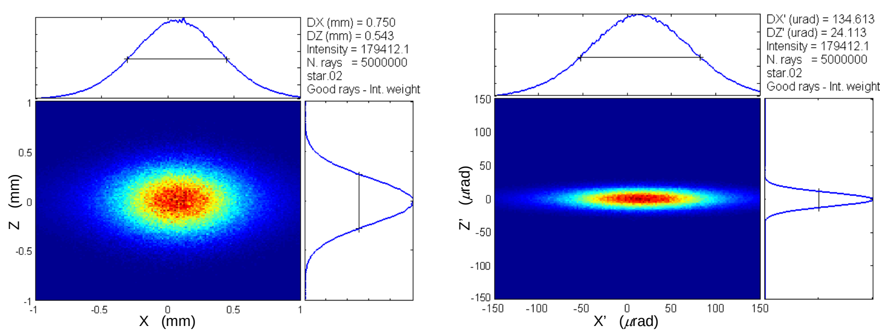

FIGURE 4. Raytracing simulation of the Laue diffracted beam size (*left*) and divergence (*right*) at focal point.

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