PROXIMA 2A – A New Fully Tunable Micro-focus Beamline for Macromolecular Crystallography

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Abstract. PROXIMA 2 is the first canted beamline at the French National Synchrotron Source SOLEIL, and it will provide two independent and tunable experimental stations, PX2-A & PX2-B, dedicated to macromolecular crystallography. The first station, PX2-A, is currently under construction. The source is an in-vacuum U24 undulator, and the optical layout includes a cryogenically cooled channel-cut Si[111] monochromator, a convex horizontal pre-focussing mirror (HPM) and a pair of focusing bimorph mirrors in Kirkpatrick-Baez (KB) configuration. This innovative optical scheme, harnesses a convex mirror to produce a virtual secondary source, which permits the KB mirrors to refocus the X-rays down to 5 μm from a relatively large horizontal source size. In fully focussed mode, the cross-section of the beam at the sample position will be approximately 5.0 μm × 3.5 μm (H×V FWHM) delivering a photon flux of 1×10¹³ – 4×10¹¹ ph/s over the range of 5 – 15 keV with a desired positional stability better than 0.5 μm rms over several hours. To achieve such stability, the supports for the optical elements are designed to minimise the effects of vibrations transmitted from the surroundings, and accelerometers will be mounted in situ to monitor these effects. For long term drifts, the experimental hutch is temperature controlled to within 0.1°C, and a preparation laboratory acts as a buffer zone. Two types of X-ray Beam Position Monitors (XBPMs), single crystal CVD diamond and thin foil-diode devices, have been developed to improve their robustness and signal-noise ratio. Due to the limitations of space, three compact and modular “slit boxes” have been designed: These vessels house a variety of beam conditioning elements such as slits, XBPMs, attenuators, imagers and a fast shutter. At the end of the station, a micro-diffractometer and an area detector (ADSC Q315) have already been installed, and the first X-ray diffraction data with unfocussed beam from test crystals are of excellent quality.

1. Introduction
With the ever increasing challenges in macromolecular crystallography (MX), Synchrotron SOLEIL has foreseen the construction of two MX beamlines, plus a third for the future. The first MX beamline, PROXIMA 1, has been open to users since 2008. PROXIMA 2 is a canted double beamline, which will comprise of the second and third MX beamlines. The first canted station, PX2-A, is currently under construction, while the second station, PX2-B, will be reserved as the future third MX beamline.

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PROXIMA 1 delivers intense, parallel and tunable X-rays over the range of 5-15 keV with a moderate focal spot size of 120 μm × 80 μm (HxV, FWHM). Although these characteristics are well adapted to many MX projects, especially for large unit cells, they are not adequate for the more difficult cases where only micro-crystals can be obtained or need to be diffraction-tested. For these reasons, PX2-A has been designed and constructed for micro-crystallography experiments, and as such, it will be complementary to PROXIMA 1. Here we describe the canted beamline PROXIMA 2, and its first station PX2-A dedicated to micro-crystallography.

2. Canted Beamline Design
The possibility for expansion to second and third MX beamlines at Synchrotron SOLEIL could only be accommodated by constructing a canted double beamline. Ideally both beamlines, PX2-A and PX2-B, should be independent and tunable over a wide energy range (5-15 keV) to optimise the anomalous scattering signals in SAD & MAD experiments.

The implantation of the beamlines was complicated by the fact that although the scientific objectives of the PX2-A station are clearly defined, those for the PX2-B station are not. As many aspects required a design for both stations, this led to a complete optical scheme for PX2-A built around a basic optical scheme for PX2-B station.

As the canting angle of 4.5 mrad does not separate the two undulator beams enough to house two MX stations, one beam must be further deviated. The toughest spatial restrictions for passage of a beam transport pipe occur at the goniometer of an MX station and require the canted beams be separated by at least 500 mm. To accomplish this, the optical scheme for PX2-B proposed will employ a series of horizontal deflecting mirrors to further deviate its X-ray path away from the PX2-A station. As the PX2-A station is designed for micro-crystallography, it will be the shorter of the two beamlines in order to minimise lever arm effects. This canted beamline scheme was inspired by the design on GM-CA CAT at the APS [1].

2.1. Undulator Source
As the waist of the electronic beam is fixed at the centre of the section, the nominal minimum gap of 5.5 mm raises to 7.8 mm for the two insertion devices off centre. Since this breaks the continuity of the tuning curves of the standard U20 undulator at SOLEIL, the magnetic period was adjusted to 24 mm for the PX2-A undulator. An in-vacuum U24 undulator has been constructed with a phase angle error of less than 2 degrees at 8.0 mm gap. It delivers $3 \times 10^{12}$ ph/s at 12 keV and X-rays out to 28 keV.

2.2. Optical Scheme for PX2-A
The optical scheme for the PX2-A beamline is designed to minimise thermal drifts and vibrations which might provoke beam instabilities. To handle the heat load from the intense beam of the U24 undulator, the first optical element is a monochromator containing a monolithic channel-cut Si[111] crystal (SYNCHROTRON-X), which is cryogenically cooled via two copper blocks from the sides by a closed circuit LN2 system (CRYOTHERM). The monochromator vessel and mechanics (CINEL) are mounted on a granite block for rigidity, and they are especially designed to be compact to permit the passage of the adjacent PX2-B deflected beam.

Focussing of the X-rays was originally intended to be accomplished with just a pair of mirrors in Kirkpatrick-Baez (KB) configuration to yield a focal spot size of 20 μm × 20 μm, but midway through the project, the objectives of the focal spot were upgraded to the ambitious values of 5 μm × 5 μm. Achieving this new objective posed a problem in the horizontal plane because of the large horizontal source size ($\sigma_H = 182 \mu m$). An optical scheme with only a pair of KB mirrors would require a large demagnification ratio (>80x) and inflate the beam divergence incident on the sample to ~3 mrad, both of which are unacceptable for MX experiments. Since the hutches and the monochromator were already installed, a solution employing a convex mirror upstream of the KB mirrors to create a virtual secondary source was selected. This innovative optical scheme permitted a minimal re-design of the beamline and avoided repositioning those elements which were already installed. See figures 1 & 2.
Figure 1. 3D drawing of the PROXIMA 2A canted beamline.

Figure 2. (a) Creation of a virtual image/source using a convex mirror. On PX2-A, the virtual image becomes the secondary source, which is then focussed again with the KB mirror pair. (b) Optical scheme of the PX2-A beamline with the horizontal trace in green and vertical trace in blue (not to scale). The dashed line is the trace from the virtual image/secondary source. Source parameters are $\sigma_h = 182 \, \mu\text{m}$, $\sigma_H = 30.9 \, \mu\text{rad}$ and $\sigma_v = 8.27 \, \mu\text{m}$, $\sigma_V = 6.67 \, \mu\text{rad}$.

2.2.1. **Focussing mirrors.** The KB focussing mirrors are a novel type of bimorph bender (THALES-SES0) which employs piezoelectric actuators mounted laterally on a 24mm-thick block of silica. The silica blocks are polished to pre-determined radii, and the piezoelectric actuators are used to adjust the shape to the desired ellipse and minimise the slope errors for micro-focussing. A total of 12 actuators are mounted for each 450 mm mirror. The vertical focussing mirror (VFM) is polished to a radius of
714.2 m and has slope errors of 3.24 μrad rms when voltages are not applied to the piezoelectric actuators. These slope errors drop to 0.63 μrad rms over 380 mm with respect to the desired ellipse when the voltages are optimised. The horizontal focusing mirror (HFM) is polished on both sides and is double-faced. One side is polished to a radius of 479 m so that focusing down to 15 μm can be achieved without the convex mirror, whilst the other side is polished to a radius of 376.6 m for focusing down to 5 μm with the convex mirror. When no voltage is applied, the slope errors of the HFM mirror start at 1.68 μrad rms and 4.17 μrad rms and then drop to 0.40 μrad rms and 0.74 μrad rms when optimised for the sides with 15μm and 5μm focussing, respectively. Optimisation of the mirror profiles was performed using adaptive correction software. At the time of writing, the mirrors have yet to be commissioned, but we plan to have three optical modes for PX2-A: Mode 1 will use the unfocussed monochromatic beam, Mode 2 will use the KB mirrors to provide a 15 μm× 5 μm focus, while Mode 3 will combine the convex mirror, the VFM and the other side of the HFM to focus down to 5 μm × 3.5 μm.

2.3. Experimental Table & Beam Conditioning Elements
A large 5-point motorised table (BRUKER-ACCEL) houses the goniometer, the KB mirrors and beam conditioning elements, so that the whole assembly is consolidated and can be aligned as a unit to follow the changes in beam height with respect to X-ray energy. The first version of this type of table was installed on PX1 and has been working successfully since 2007. The PX2-A version has improved upon this prototype with more precise movements and enhanced stability. Metrology tests on this table have shown that its movements have mechanical resolutions well below 0.5 μm.

With the addition of the convex mirror, the space available for beam conditioning elements became tight. This lead to the development of a compact and modular design of “slitboxes” to house slits, a fast shutter, X-ray beam position monitors (XBPMs) and X-ray cameras.

2.4. X-ray Beam Stability, XBPMs & Accelerometers
As MX experiments with micro-beams are sensitive to instabilities in the X-ray beam, much attention has been given to making supports rigid, removing sources of vibration or thermal effects and monitoring the X-ray beam position. All of the main optical components are mounted on granite bases which are grouted to the experimental floor. Both radiation safety hutchies are air conditioned to within 0.1°C, and a small preparation lab, which adjoins the entry to the experimental hutch, acts as a thermal buffer zone.

To monitor the X-ray intensity and position during data collections, we have developed two types of XBPMs: foil-diode devices and single crystal CVD diamond devices. Both devices have been installed on the beamline and function in parallel. The S/N is over 50 times stronger for the scCVD diamond devices than the foil-diode devices. The long term position stability of the unfocussed beam just downstream of the monochromator giving a measured flux of 2×10^{12} photons/s at 12.65 keV (2 mm × 1 mm FWHM) is approximately 300 nm rms in the horizontal and 75 nm rms in the vertical.

2.5. MX Station
The equipment for MX experiments currently includes a micro-diffractometer (MD2, BRUKER-MAATEL), an area detector (Q315r, ADSC), an X-ray fluorescence detector (KETEK), and a Cryostream (OXFORD CRYOSYSTEMS). The MD2 is mounted at the end of the experimental table, which permits its consolidated alignment with the KB mirrors during X-ray energy changes. The area detector is mounted on a long granite base with motorised stages in XYZ. Eventually, other devices for the transfer of cryo-cooled samples and screening of crystallisation plates in situ will be installed during 2013. Software for controlling the beamline will use standard SOLEIL applications for aligning and monitoring equipment, but use MXCuBE [2] for the MX data collections.

2.5.1. Preliminary Data Collection Results. In 2011, prior the arrival of the experimental table and the KB mirrors, the MD2 and the area detector were commissioned in advance and diffraction data were
collected from single crystals in unfocussed mode. This provisional setup provided excellent diffraction data ($R_{sym} \sim 2\%$ in low resolution bins), albeit the exposure times were rather long (10-30s per degree) with a $50 \mu m$ diameter aperture.

3. Conclusions & Perspectives

Ray tracing calculations with the measured mirror profiles predict that the PX2-A focal spot size will be $5.4 \mu m \times 3.3 \mu m$ (HxV, FWHM) with over $10^{12}$ ph/s. With such an intense micro-focussing, we envisage a variety of functionalities to be made available to the MX community including automated cartography [3-9], helical scans [10] and in situ [11-13] data collections. The latter will include a concerted effort in screening samples in crystallisation plates and micro-fluidic devices [14].

Acknowledgements
The authors wish to thank many of the staff in the support groups at SOLEIL, as well as P. Gourhant, P. Legrand, B. Guimaraes & A. Thompson of the PROXIMA 1 group.

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