Nuclear Instruments and Methods in Physics Research A 649 (2011) 112–113

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

A new mini-beam device for protein crystallography

R.W. Alkire*, M. Molitsky, F.J. Rotella, K. Lazarski, A. Joachimiak

Structural Biology Center, Biosciences Division, Argonne National Laboratory, 9700 South Cass Avenue, Building 435, Argonne, IL 60439, USA

ARTICLE INFO

Available online 8 December 2010

Keywords:
Mini-beam
Micro-beam
Macromolecular crystallography

ABSTRACT

A fully motorized mini-beam device has been constructed for use in protein crystallography. This device separates the beam-defining aperture from the guard aperture into two distinct components, removing the need for pitch and yaw adjustments. Each aperture can be scanned separately using only *x* and *y* translations, allowing independent positioning of the beam-defining and guard apertures. Switching from mini-beam to the existing slit system is controlled by a single mouse click.

1. Introduction

It is becoming routine to see protein samples brought to the synchrotron that are 5–10 μm on an edge. To obtain the highest quality data from these samples requires maximizing the signal by selectively probing for the highest quality regions of the sample and minimizing the background by reducing the volume of solvent and air the beam passes through. Development of Mini-beam devices, where the beam sizes can range from 5 to 30 μm, started at the ESRF [1,2] and has continued to progress for more than a decade. GMCA-CAT at the APS has developed a mini-beam array consisting of four [3] independent beam-defining apertures. Commercially, Bruker-AXS is marketing the multi-purpose MD2 diffractometer, which has its own mini-beam device. These devices are constructed with a beam-defining aperture nested inside a tube with a much larger (100–300 μm) guard aperture. Both devices require two translations plus pitch and yaw adjustments for alignment. At present, the pitch and yaw adjustments are manual only, requiring considerable time and effort to complete the alignment.

At Structural Biology Center beamline 19ID we are developing our own mini-beam device. This device is designed to deliver beam in the 5–25 μm range with interchangeable beam-defining apertures and very small guard apertures. To alleviate the difficulty caused by alignment of a tubular style mini-beam apparatus, the SBC device was designed to separate the guard-aperture from the beam-defining aperture, eliminating the need for any pitch or yaw adjustment. Once separated, alignment can be accomplished with only vertical and horizontal adjustments.

2. Mini-beam device

SBC beamline 19ID is an undulator beamline with a Si (1 1 1) double crystal, sagittal focusing monochromator and a vertically focusing ULE mirror. Average focused beam dimensions (FWHM) at the slits are 0.25 mm horizontally $\times$ 0.065 mm vertically with a photon flux of 2×10^{12} ph/s through a 0.1×0.1 mm^2 slit at 12.66 keV. Although these values are larger than what the optics can achieve, the larger beam size and smaller slits act to smooth out short term intensity fluctuations, eliminating the need for position feedback. To take full advantage of our existing slit system, the mini-beam apparatus is installed just downstream of the primary slit scatter shield.

The mini-beam device, designed by Michael Molitsky, is composed of two independently motorized support arms, each holding two small aperture cylinders. The mini-beam device is attached to the outside of the vacuum-sealed slit assembly box and can be installed or removed in less than 5 min; a photograph of the mini-beam device is shown in Fig. 1. On installation the beam-defining aperture cylinder is positioned a few millimeters from the end of the primary slit scatter shield. Each cylinder includes an electron microscope aperture (made from Pt–Ir, 95:5, manufacturer Ted Pella, Inc., Redding, CA, aperture dimensions 2.0 mm OD $\times$ 0.6 mm thickness) placed inside a magnetic stainless steel housing. We typically use beam-defining apertures of 5, 10 or 20 μm; guard-apertures are either 20, 50 or 100 μm. Separation distance between the defining and guard-apertures is 10 mm.

In order to ensure no parasitic scattering from the apertures, each aperture cylinder is machined with a narrow (0.343 mm) downstream opening. The aperture is inserted into the holder and a second tube is inserted behind it to hold the aperture in place. A special crimping tool is then used to seal the cylinder assembly. Both tubes contain access holes, which overlap on assembly, allowing helium to flow through the cylinder when installed. The helium fill tubes located in the translation arms act as centering mechanisms for the cylinders, allowing reproducible cylinder positioning for each aperture. Small magnets in the support arms keep the magnetic cylinders secured during movement. A schematic of an assembled cylinder with aperture is shown in Fig. 2.

Accurate placement of the apertures is ensured by the 29 nm step resolution of each translator arm motor. Separation distance

* Corresponding author. Tel.: +1 630 252 3865; fax: +1 630 252 0564.
E-mail address: alkire@anl.gov (R.W. Alkire).

0168-9002/$ - see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.nima.2010.11.123

Fig. 1. Photograph of the mini-beam device.

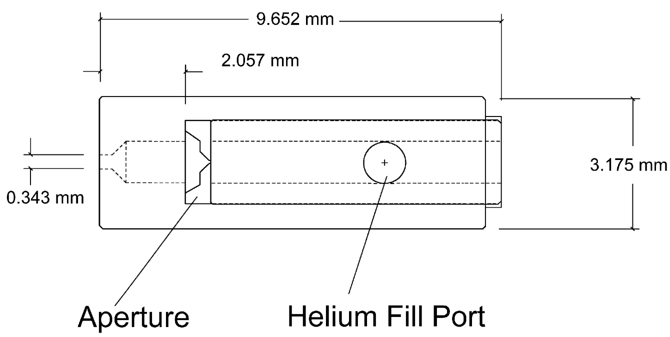

Fig. 2. Schematic of an assembled cylinder with aperture.

between adjacent cylinders on a given arm is ~4 mm, with a total travel of 6 mm available in both the vertical and horizontal directions. Certain restrictions apply when moving the two translation arms separately, but sufficient travel exists to allow all apertures to be scanned independently. Along the beam direction the beam-defining and guard cylinders are separated by roughly 1 mm, leading to a separation distance of 10 mm between the beam-defining and guard apertures.

By installing a pair of straight through cylinders i.e., without apertures, the primary slit system (which is capable of aperturing beam sizes down to $25 \times 25\ \mu m^2$) can be used without removing the mini-beam apparatus. Typical scanning time per aperture is only a few minutes. Once oriented, changing from mini-beam to the open cylinders can be done using a single mouse click. Presence of a kappa, an on-axis mirror and a minimum opening distance for the robot end-effector, requires the beam-defining aperture to be positioned 65 mm from the sample. This is a self-imposed limit since, with different sample manipulation and viewing hardware, the device could operate right up to the edge of the cold-stream. At this distance the horizontal beam size at the sample increases by about 20% over the aperture size.

3. Results

Fig. 3 shows the beam profile of the 5 μm beam-defining aperture using a 20 μm guard aperture; full-width at half-maximum is 6.2 μm. The profile was obtained via fluorescence radiation (L_3 edge) from a specially constructed blade, consisting of elemental Au deposited onto a silicon substrate. Dimensions of the Au blade [4] were 30 nm thick × 70 μm wide × 2 mm long and blade alignment was optimized using the arc motions present on the motorized *X–Y* head of the kappa device. The fluorescence profile presented is a direct measurement, showing raw data taken in 100 nm step increments. Photon flux through the 5 μm aperture was $\sim 5 \times 10^9$ ph/s at 12.66 keV. For the 10 μm beam-defining aperture a 50 μm guard aperture was employed. Photon flux through the 10 μm aperture was 3.7×10^{10} ph/s.

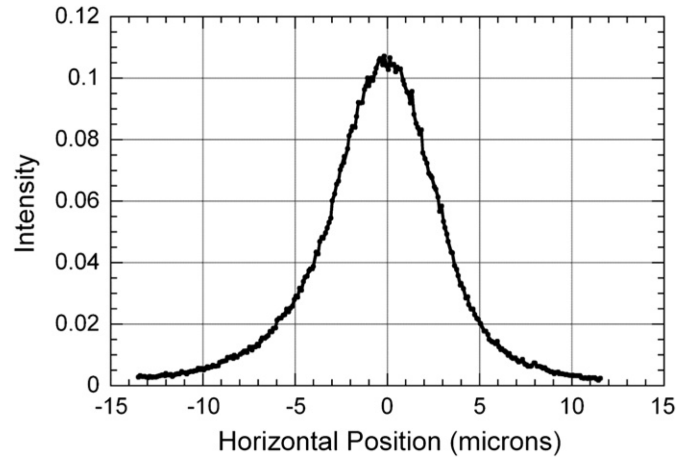

Fig. 3. Beam profile for the 5 μm beam-defining aperture using a 20 μm guard aperture; FWHM is 6.2 μm.

While the beam-defining apertures allow good quality diffraction images to be collected with the narrow guard-apertures in place, forward-scattered radiation does extend out from the beam stop to 80 Å resolution for both the 5 μm/20 μm and 10 μm/50 μm beam-defining/guard-aperture combinations. A 20 μm/100 μm beam-defining/guard-aperture combination shows a clean image out to the beam stop resolution limit of 115 Å. To completely eliminate all traces of forward-scattered radiation using the 5 or 10 μm beam-defining apertures, their respective guard-apertures would have to be increased.

This device has been constructed in order to facilitate the ease of aligning mini-beam apertures relative to the X-ray beam position. As evidenced by the ability to use small guard-apertures, rapid alignment can be accomplished using simple *x–y* translations. Future enhancements to this device will involve expanding the number of cylinders that can be permanently installed on the support arms and an extended translation range.

Acknowledgements

This work was supported by the US Department of Energy, Office of Biological and Environmental Research, under Contract DE-AC02-06CH11357.

References

[1] P. Engstrom, S. Fiedler, C. Riekel, Rev. Sci. Instrum. 66 (2) (1995) 1348.
[2] C. Riekel, Rep. Prog. Phys. 63 (2000) 233.
[3] S. Xu, O. Makarov, R. Benn, D.W. Yoder, S. Stepanov, M. Becker, S. Corcoran, M. Hilgart, V. Nagarajan, C. Ogata, S. Pothineni, R. Sanishvili, J. Smith, R. Fischetti, in: Proceedings of the 10th International SRI Conference, Melbourne, Australia, 2009, AIP Conference Proceedings, vol. 1234, pp. 897–902, ISBN 978-0-7354-0782-4.
[4] Au blade provided by Dr. Abdel F. Isakovic, BNL, currently at KUSTAR, Abu Dhabi (UAE).