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Mini-beam collimator applications at the Advanced Photon Source ☆

Shenglan Xu [a,*], Lisa J. Keefe [b], Anne Mulichak [b], Lifen Yan [c], Ercan E. Alp [c], Jiyong Zhao [c], Robert F. Fischetti [a]

[a] GM/CA CAT, Biosciences Division, Argonne National Laboratory, Argonne, IL 60439, USA
[b] IMCA CAT, Argonne National Laboratory, Argonne, IL 60439, USA
[c] X-ray Sciences Division, Argonne National Laboratory, Argonne, IL 60439, USA

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ABSTRACT

In 2007, the General Medicine and Cancer Institutes Collaborative Access Team (GM/CA CAT, Sector 23, Advanced Photon Source) began providing mini-beam collimators to its users. These collimators contained individual, 5- or 10-μm pinholes and were rapidly exchangeable, thereby allowing users to tailor the beam size to their experimental needs. The use of these collimators provided a reduction in background noise, and thus improved the signal-to-noise ratio [1,2].

Recent improvements in the collimator design include construction of the device from a monolithic piece of molybdenum with multiple pinholes mounted inside [3]. This allows users to select from various size options from within the beamline control software without the realignment that was previously necessary. In addition, a new, 20-μm pinhole has been added to create a "quad-collimator", resulting in greater flexibility for the users.

The mini-beam collimator is now available at multiple crystallographic beamlines and also is a part of the first Mössbauer Microscopic system at sector 3-ID.

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1. Introduction

The advent of high quality third-generation X-ray sources, such as the Advanced Photon Source, European Synchrotron Radiation Facility, and SPring-8, has provided new advantages to protein crystallographers. One such benefit is the use of smaller mini- or micro-beams. This can be accomplished by two different approaches: to use optical elements to reduce the focus size of the incident X-ray beam or to use collimating devices to sample portions of a focused beam. The hard X-ray mini-beam collimator system was developed to exploit the latter approach.

The system consists of three essential components: a mini-beam collimator, a magnetically indexed kinematic mount and a precision motion system (Fig. 1). Further descriptions of each component are provided below. The function of the system is to provide micron-sized beams of various sizes to a sample. It must be compact, durable, economical and its motions reproducible and precise at the sub-micron level. This set-up can be placed on beamlines or on a home X-ray source.

1.1. Mini-beam collimator allows for a clean beam of desired size

The quad collimator design includes a robust monolithic "Uni-body", four "caps" and four pinholes (Fig. 1). Four stepped beam path channels were machined into the monolithic body. This has significantly improved the robustness, the ease of initial alignment and the reduction of background scatter. It ensures the alignment of the forward scatter-guards that are smaller than Ø250 μm. The collimators capture the low angle scattering, thus keeping background scatter to a minimum. The compact footprint was a requirement to minimize obstruction of the high resolution, on-axis sample visualization microscope. The beam size is defined by a 2-mm diameter platinum disk with a pinhole in the center (Ted Pella, Inc.). The disk is 600 μm thick and tapers to 150 μm at the position of the aperture [3].

1.2. Kinematic mounting allows for easy adaptation to other station

A key design specification was to provide easy access for removal and replacement of the collimator from its mount. This is to accommodate changing experimental conditions, as well as maintenance/customization of the surrounding instrumentation. The collimators are then required to be replaceable within sub-micron tolerances. A kinematic mounting system has been designed to

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* Corresponding author.
E-mail address: sxu@anl.gov (S. Xu).

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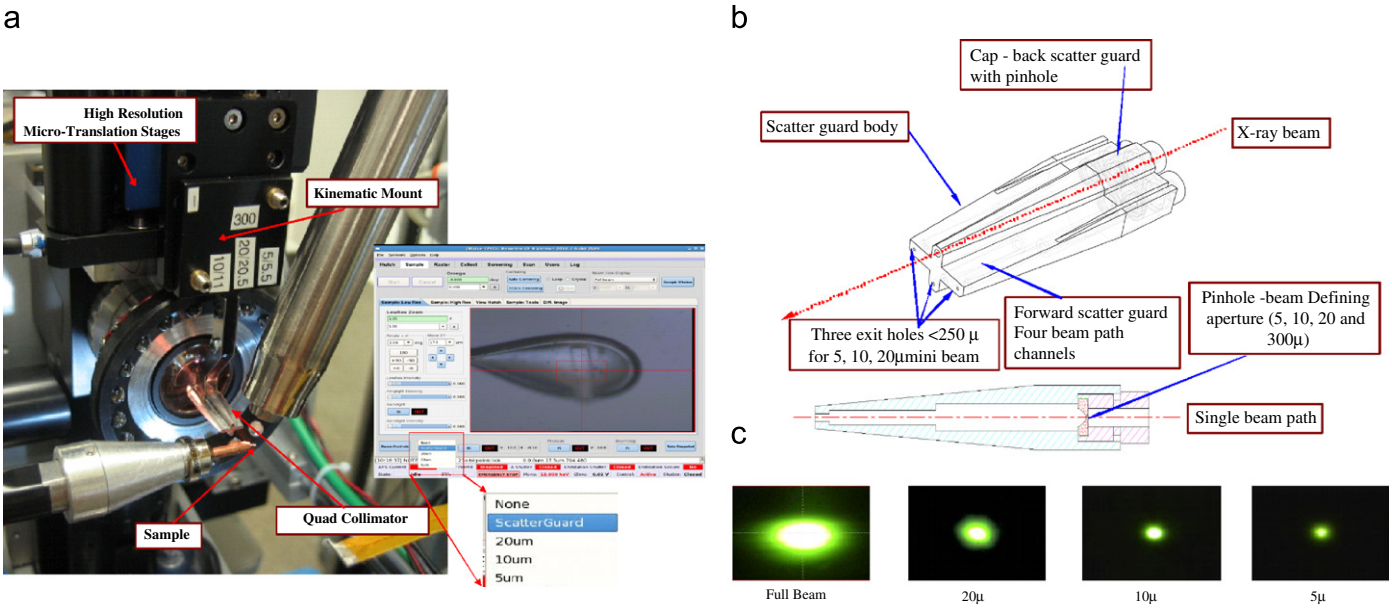

Fig. 1. Left—the quad collimator installed on the beamline; middle—collimator selection menu in JBluIce; right—(a) schematic diagram of the assembled mini-beam collimator. Excellent scatter protection is achieved by nesting of the pinhole. The pinholes are held into the uni-body with a cap that also serves as a back scatterguard. The exit holes are smaller than Ø250 μm for mini-beams. (b) Cross-sectional view of an individual collimator. (c) Image of the various beams from an Yttrium Aluminum Garnet crystal (YAG) scintillator mounted at the sample position and viewed with the on-axis-visualizer.

Table 1
List of mini-beam collimator applications at the Advanced Photon Source.

Location at APS	Beamline	With defining aperture Ø (μm)	Type of collimator	Application
Sector 23	ID-B	5, 10, 20 and 300	Quad	Micro-crystallography
Sector 23	ID-D	5, 10, 20 and 300	Quad	Micro-crystallography
Sector 23	BM	50, 70, 100 and 300	Quad	Micro-crystallography
Sector 17	ID	5, 10, 20 and 300	Quad	Micro-crystallography
Sector 17	ID	10, 20, 50, and 300	Quad	Micro-crystallography
Sector 3	ID	5, 10 and 300	Trip	MÖssbauer Microscope
Sector 31	ID	50, 100, 150 and 300	Quad	Micro-crystallography

reach these specifications. The collimator can be removed and remounted on the kinematic mount with a reproducibility of +/−0.4 μm in the two orthogonal translation directions [3].

1.3. High resolution translation stages and control system allow for easy selection of beam size

Two high resolution micro-translation stages are combined to form an XY system for the collimators alignment. The travel range is 15 mm for both stages. The design resolution is 0.007 μm with a repeatability of 0.1 μm. The whole system stability is +/−0.1 μm [3].

The new quad collimators and modified control software to accommodate them are available on the beamlines. In addition to enabling a change of beam size with just two clicks in JBluIce (Fig. 1), two other software features have been added to take advantage of the mini-beam. Rastering software uses the mini-beam with low dose to map the diffraction quality of a crystal. Vector collection enables the collection along an arbitrary 3D vector to mitigate the effects of radiation damage. These new capabilities have been well received by the user community.

2. Mini-beam collimator applications in the Advanced Photon Source (APS)

There has been an increasing interest in this device around the APS. This can be seen in the growing list of beamlines that now incorporate the mini-beam collimator (Table 1). The quad collimator on Sector 31 is planned for installation in the near future.

2.1. Quad collimator at GM/CA beamlines Sector 23 for micro-crystallography

The quad collimator system has been in use on both of the GM/CA CAT beamlines since October 2009. More than 80% of user groups use one of the mini-beams to match the beam size with the crystal size or rastering to find the best part of the crystal or collecting along a vector as described previously.

Mini-beam collimator enables micro-crystallography experiments on the focused ID-beamlines at GM/CA CAT. The beam sizes, intensities and flux densities are shown in Table 2.

2.2. Quad–mini-beam collimator implemented on Maatel OAV for micro-crystallography at IMCA-CAT Sector 17

The Industrial Macromolecular Crystallography Association Collaborative Access Team operates a data collection facility at APS Sector 17 to satisfy the needs of pharmaceutical researchers for high-throughput diffraction experiments. The IMCA-CAT insertion device beamline was recently upgraded to a micro-focused, high-flux beamline with all new state-of-the-art experimental components, including a Dectris pixel array detector to allow ultrafast, shutterless data collection, as well as a high-precision

Table 2
Beam sizes and intensities are listed for the focused and mini-beams. The pinhole selects the central part of the focused beam.

	23-ID-B		23-ID-D	
	Beam size	Intensity (photons/s)	Beam size	Intensity (photons/s)
Natural focus	25 μm × 120 μm	5×10^{12}	20 μm × 70 μm	1×10^{13}
Mini-beam collimator	Ø20 μm	5×10^{11}	Ø20 μm	1×10^{12}
	Ø10 μm	1×10^{11}	Ø10 μm	2×10^{11}
	Ø5 μm	4×10^{10}	Ø5 μm	7×10^{10}

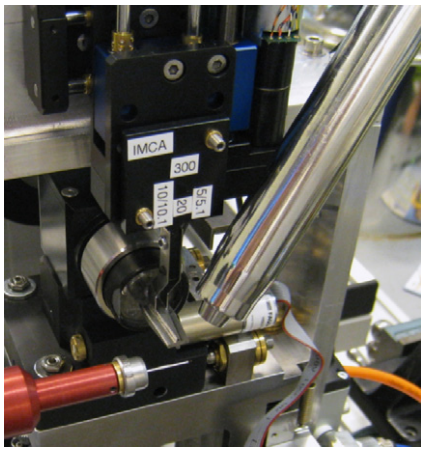

Fig. 2. Quad collimator installed on Sector 17 beamline.

goniometer and on-axis sample visualization to accommodate very small samples.

To complement these capabilities, two mini-quad collimators were developed in collaboration with GM/CA CAT for use with a Maatel on-axis viewing (OAV) microscope (Fig. 2).

Installed since February 2010, the collimators allow users to readily select the beam size most suitable for their experiment at any time, and can be retracted completely from the microscope lens optical path for optimal sample viewing and to allow access by the sample auto-mounter.

2.3. Triple collimator for Mössbauer microscope development at 3-ID

The mini-beam collimator is also a part of the first Mössbauer microscopic system development project at sector 3-ID (Fig. 3). This beamline had a micro-focus spot size of about 15 μm × 15 μm. In order to improve the resolution below 5 μm, we have installed a Ø5 μm pinhole, and scanned the incident beam, which is tuned to ^{57}Fe nuclear resonance at 14.412 keV. The first experiment results are very encouraging (L. Yan, et al., unpublished) and with the pinhole in place, the scans resulted in excellent signal-to-noise ratio.

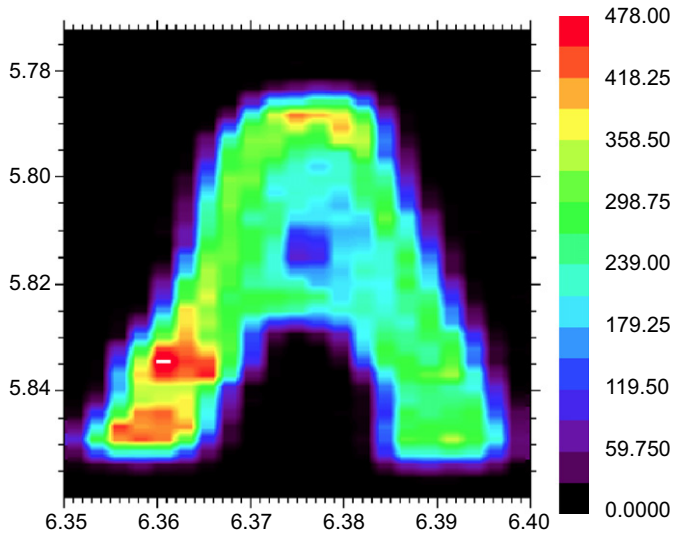

Fig. 3. Scanned image of a phantom sample. The dimensions are 50 μm × 85 μm. The image is obtained with a 5 μm pinhole, using delayed nuclear forward scattering signal; hence when there is no iron, there is no signal.

Signal:	Mössbauer signal from the sample
Sample:	^{57}Fe-enriched iron sample
Thickness of the characters:	0.7 μm
Width of features:	~15 μm
Beam size:	5 μm in diameter
Data collection time:	1 s/pt
Pixel size:	69 × 69 points, 170 × 170 μm^2

3. Summary

The quality of the mini-collimator system has enabled scientists to routinely use it to optimize their experiments and expand applications for the use of mini-beams. About 30% of our users employed the mini-beam when the collimators were single; after the implementation of multi-collimator systems, over 80% of them now use the mini-beam for data collection.

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References

[1] Venugopalan Ruslan Sanishvili, Derek Nagarajan, Michael Yoder, Shenglan Becker, Stephen Xu, David L. Corcoran, Janet L. Akey, Smith, Robert F. Fischetti, Acta Crystallographica Section D 64 (2008) 425.
[2] Robert F. Fischetti, Shenglan Xu, Derek W. Yoder, Michael Becker, Venugopalan Nagarajan, Ruslan Sanishvili, Mark C. Hilgart, Sergey Stepanov, Oleg Makarov, Janet L. Smith, Journal of Synchrotron Radiation 16 (2009) 217.
[3] Shenglan Xu, Oleg Makarov, Richard Benn, Derek W. Yoder, Sergey Stepanov, Michael Becker, Stephen Corcoran, Mark Hilgart, Venugopalan Nagarajan, Craig M. Ogata, Sudhir Pothineni, Ruslan Sanishvili, Janet L. Smith, Robert F. Fischetti, AIP Conference Proceedings 1234, 2010, pp. 897–900.