Design and Development of compact Mini-beam Collimators for Macromolecular Crystallography at the GMCA CAT

Shenglan Xu

GM/CA CAT
Argonne National Laboratory

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GM/CA CAT Staff

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Robert Fischetti
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Sheila Trznadel
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Crystallographic Support

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Nukri Sanishvili

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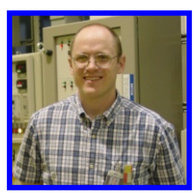


Derek Yoder

Rich Benn

Steve Corcoran

Outline

- Design history of compact mini beam collimators
  - Single collimator
  - Dual collimator
  - Triple collimator
  - Upgraded triple collimator
  - Quad collimator
- Visualization test results
- Beam flux through collimator pinholes
  - Simulation and measurements
- Material study
- Mounting and alignment
- Implementation into BluIce GUI

Design history of mini beam collimator

Feb. 2007: First use of single mini beam collimators, 5, 10 and 20μm defining apertures

Jun. 2007: Dual collimator

5 μm beam defining and 300 μm scatter guard, and

10 μm beam defining and 300 μm scatter guard

Feb. 2008: Triple collimator (5, 10, 300) with three forward scatter guard tubes

Apr. 2008: New "Uni-body", triple channel, more robust, better alignment

Feb. 2009: Two types of "Uni-body" Triple collimator installed on ID-stations

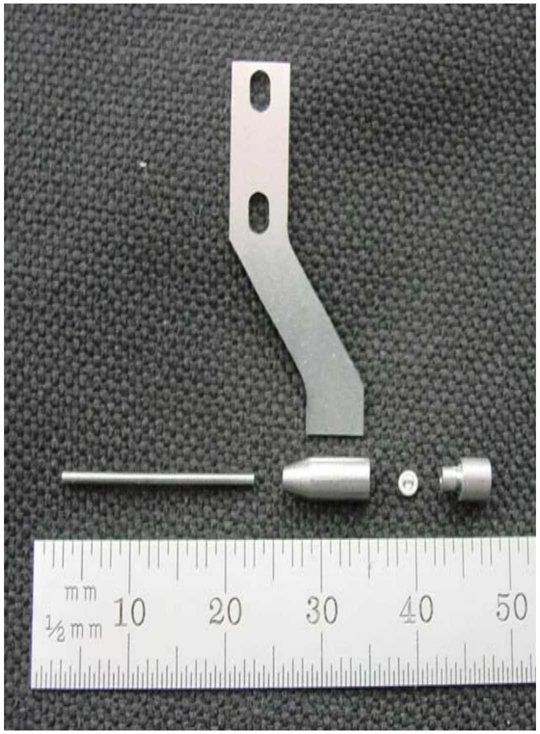
Type I: with 5, 10 and 300 μm apertures

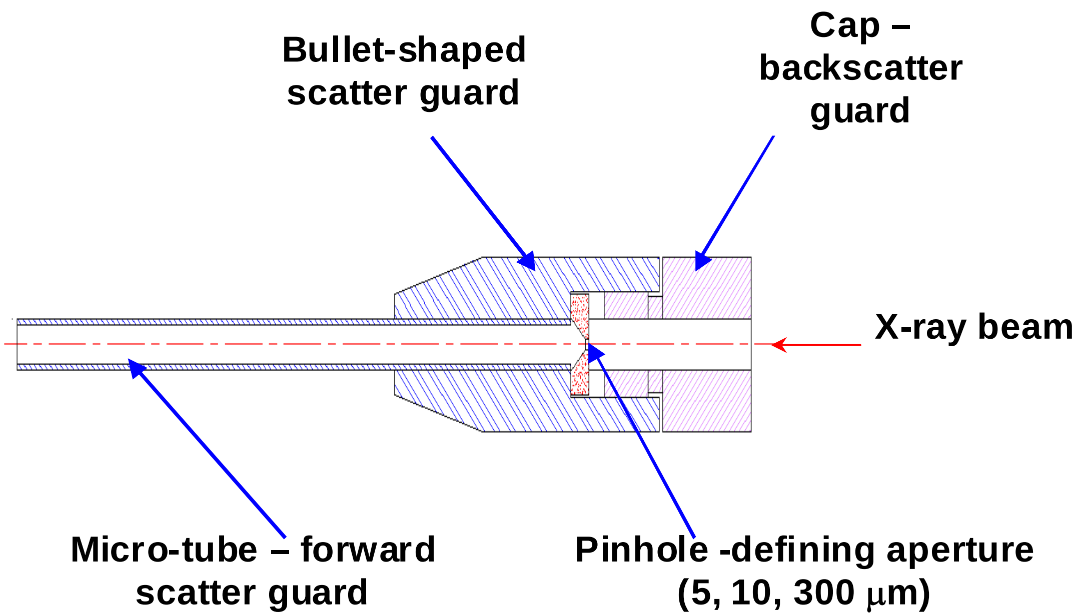
Type II: with 10, 20 and 300 μm apertures

Apr. 2009: Prototype of quad collimator designed and fabricated

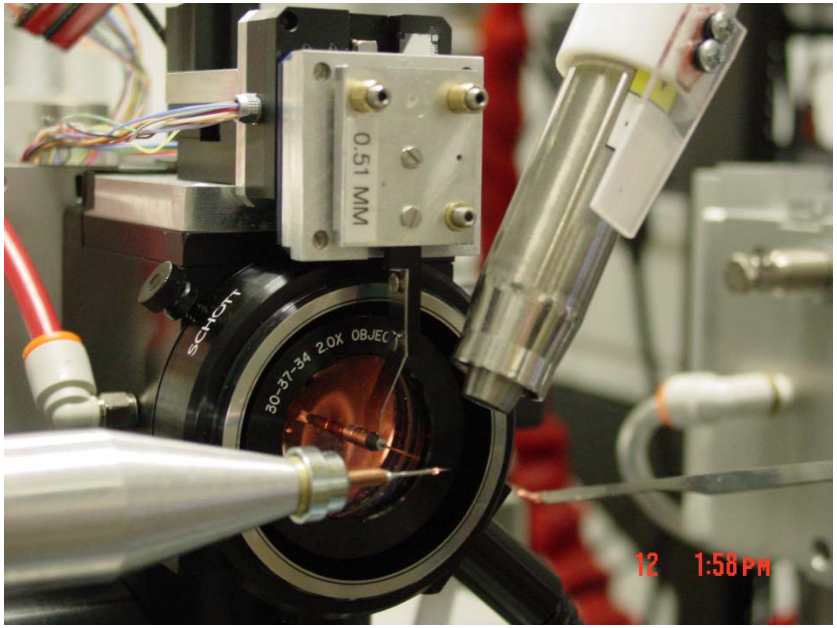
with 5, 10, 20 and 300 μm apertures

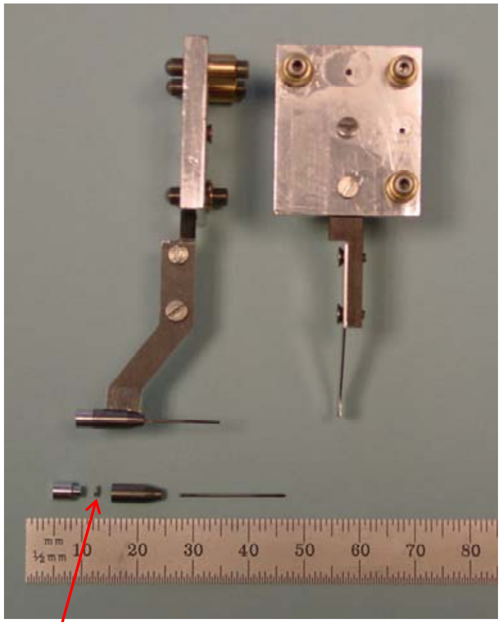
Components of the single mini-beam collimator



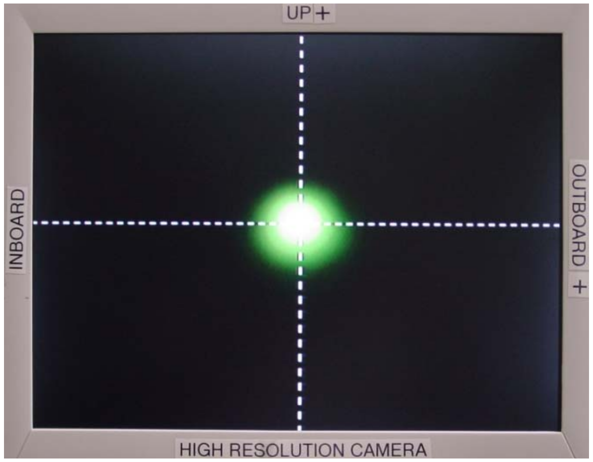


First mini-beam collimator installed Feb 2007





5, 10, 20 μm apertures



Pin hole diameter (μm)	Beam size, FWHM(VxH) (μm)	Intensity (photons/sec/100mA)
5	5.0 x 5.1	7.8×10^{10}
10	10.5 x 10.8	2.0×10^{11}
Full beam	25 x 70	2.0×10^{13}

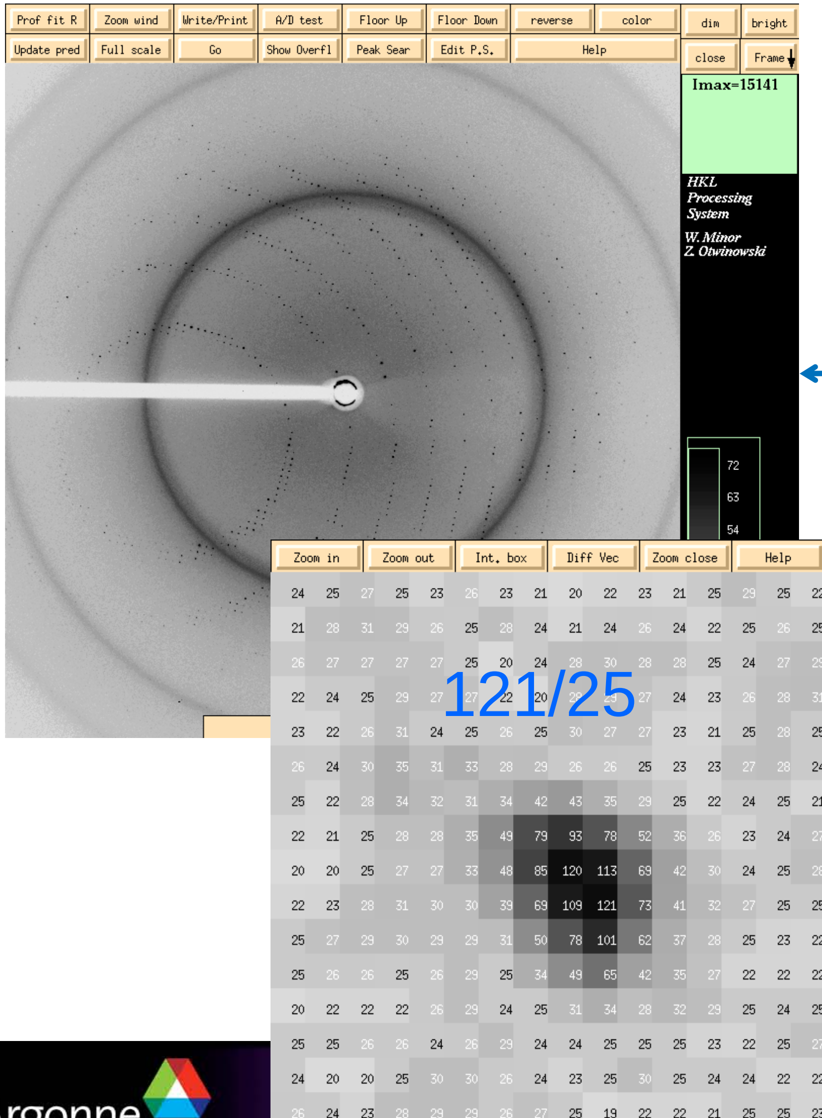
Advantages of Mini-beam

- Background reduction due to the better size match of the beam and crystal
- Collect useful data on projects that produce only small crystals
- Select best part of crystal – mosaicity or macro twining
- Rastering on large crystals during data collection reduces effects of radiation damage

Improved Signal/Noise ratio from Thioesterase Sample - ~8x8x150 μm

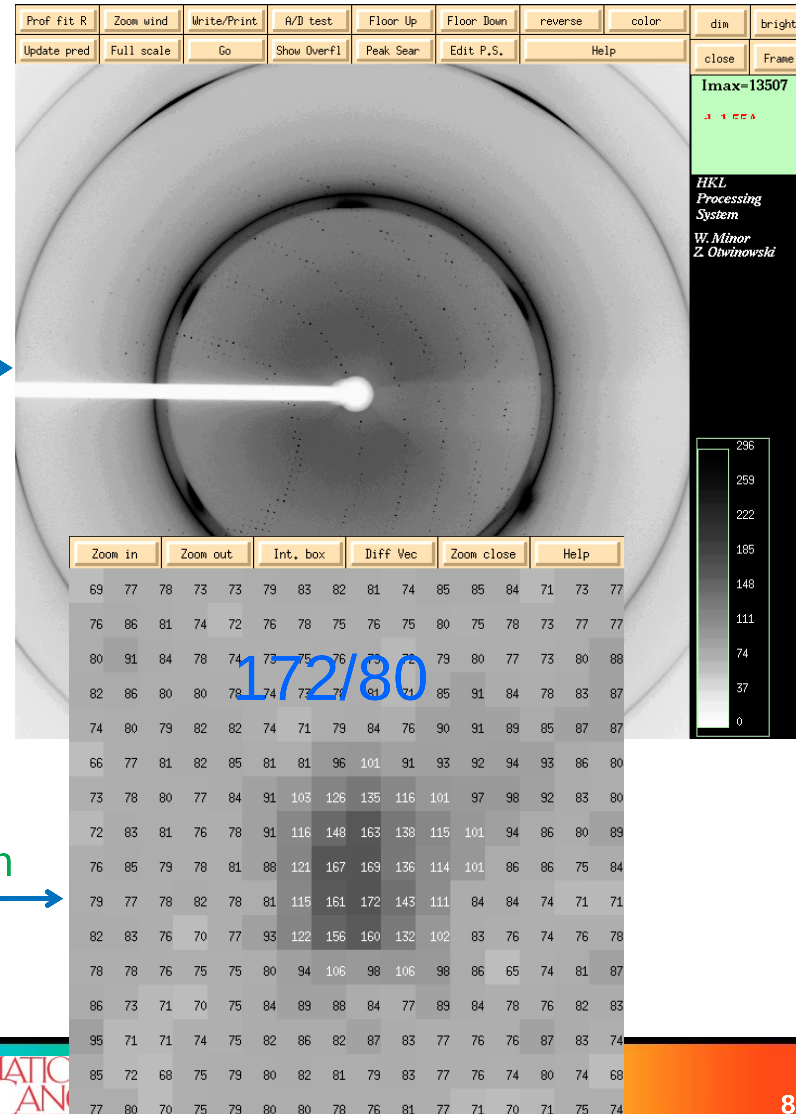
7-μm mini beam

d_{min} = 2.4 Å



75-μm beam

d_{min} = 2.6 Å



Same crystal & setting

Same reflection

Dual collimator installed June 2007
Reliable user operations with mini-beam - quick switch between full-beam and mini-beam

Dual collimator:
- one click exchange
- auto-align routines

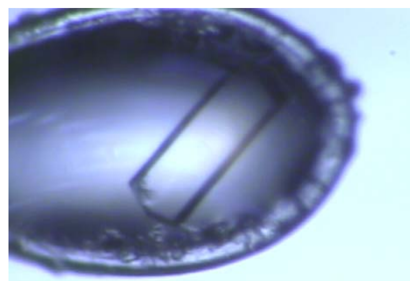
5 or 10 µm

300 µm

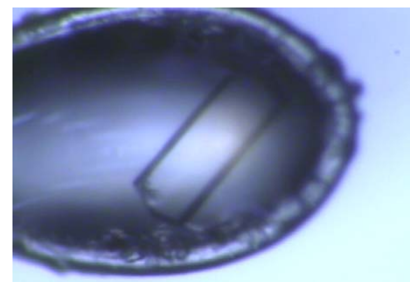
Double collimator can be used for optimal data collection from large crystals as well:

Loop area can be scanned with the larger beam to locate the crystal then finely scanned with the mini-beam to locate the best part of the crystal for data collection .

Visual obstruction of the double collimator is minimal



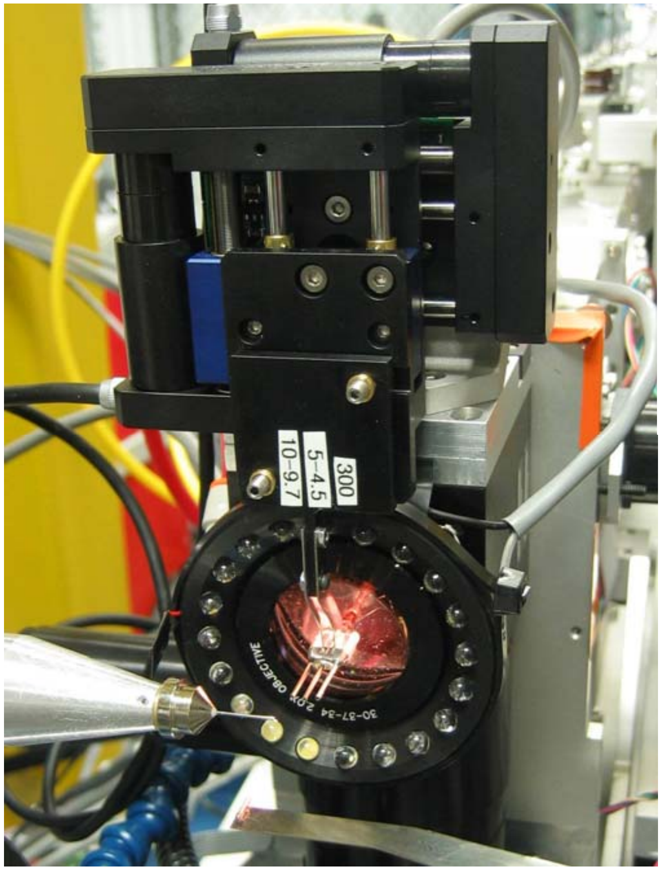
No collimator

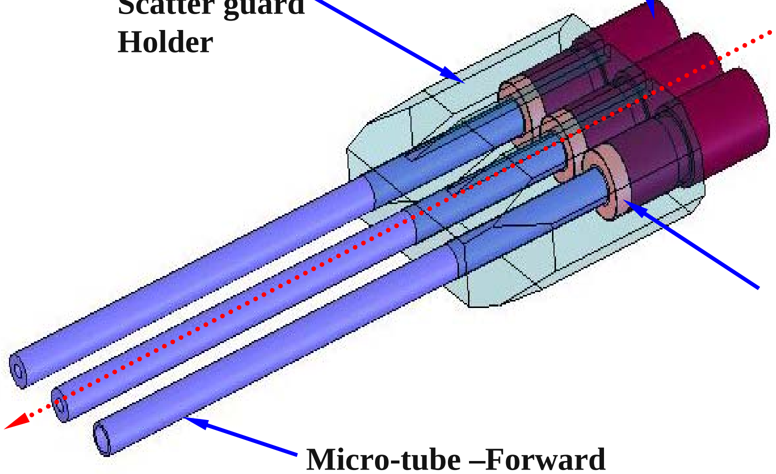


Double collimator

Triple collimator with three tubes installed Feb 2008

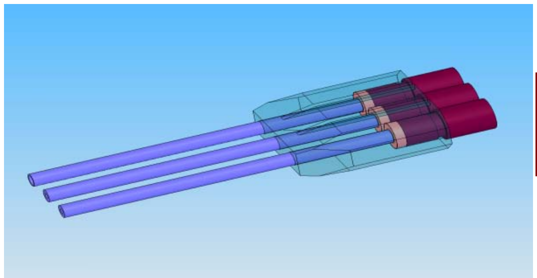
quick switch between full-baem and two mini-beams (5, 10 microns)



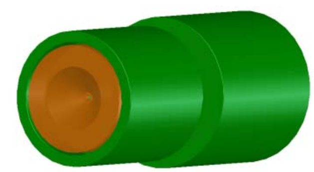


Upgraded design of triple collimator

--- to overcome the assembly and alignment difficuties



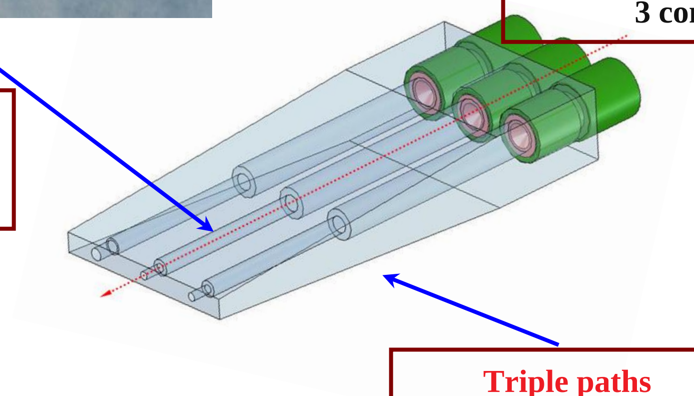
Assemble 10 pieces and
align 9 component



Pinhole-defining aperture
Glued on cap (back scatter guard)

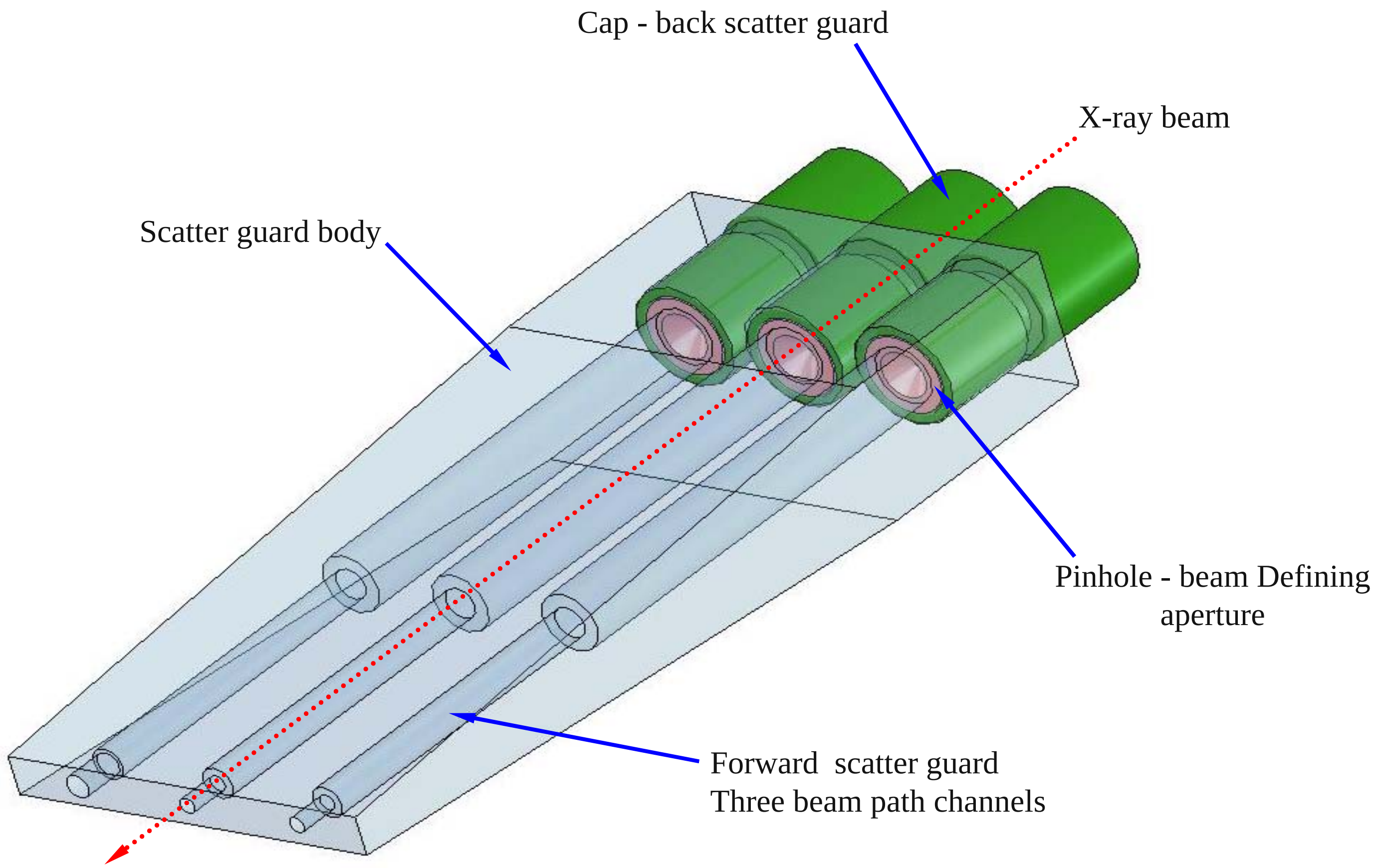
Assemble 4 pieces and align
3 component

Merge to
One body
Scatter guard

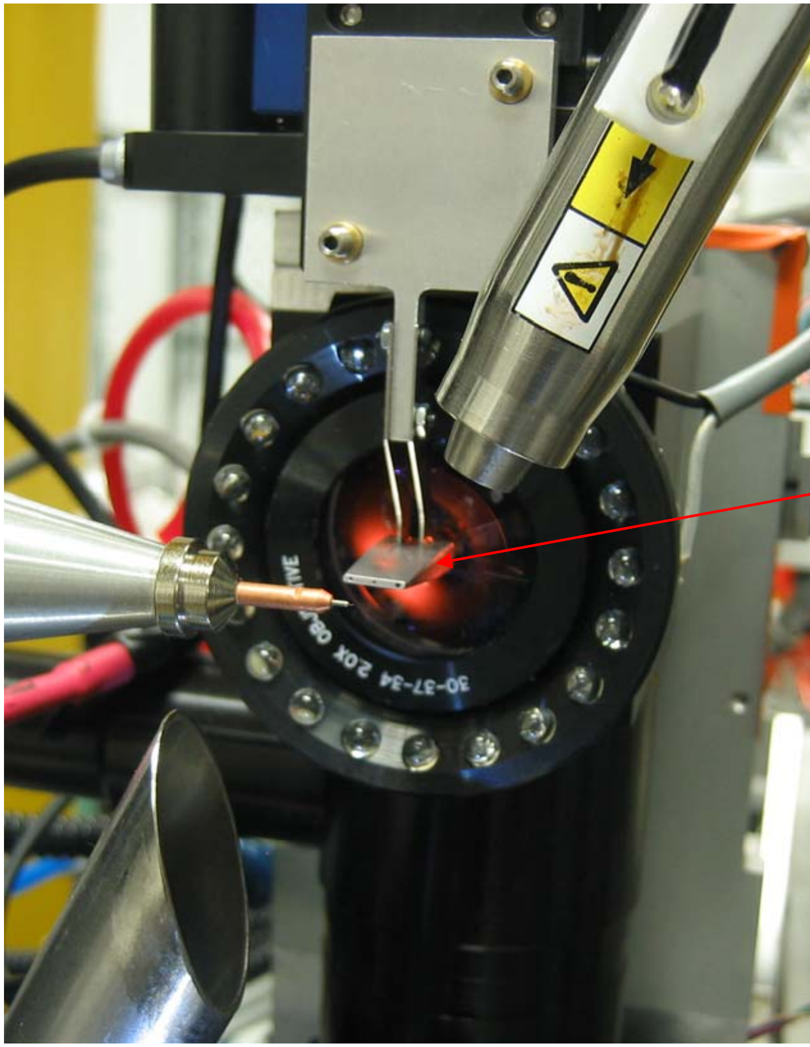


Triple paths
Scatter guard

“Uni-body” Triple collimator – significantly improves the robustness, ease of initial alignment, and reduction of background.



“Uni-body” triple collimator installed Feb. 2009



Type I:
5 and 10 micron mini-beam defining apertures, 300 micron scatter guard aperture

Type II:
10 and 20 micron mini-beam defining apertures, 300 micron scatter guard aperture

User selectable via BluIce buttons
Prealigned
Highly reproducible

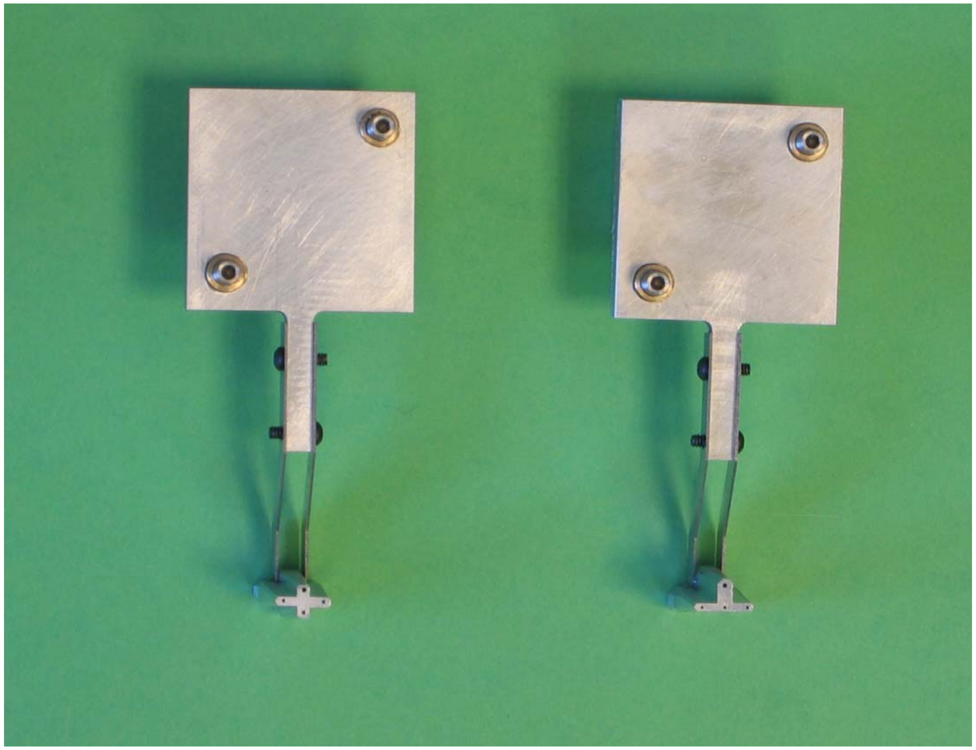
Advantages of "Uni-body" Triple collimator

- Easy to assemble
- No tubes to pre-align using a microscope
- Robust – no tubes to bend
- Pinholes can be removed for cleaning
- Easy to change pinhole size
- Easy to align - machined forward scatter "tubes" all point in the same direction
- Smaller exit aperture - Ø300μ
- Reduced scatter around the beamstop

Type I and II triple collimators had to be exchanged for 5 or 20 micron beam

Solution: Designed quad-mini-beam collimator with 5, 10, 20 , 300 micron pinholes

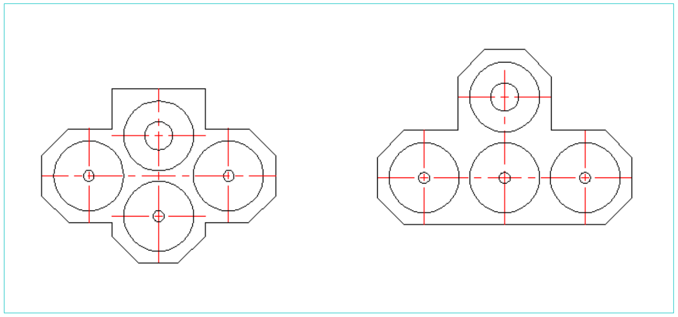
Prototype Quad Collimators - April 06, 2009



D - Quad Collimator

T - Quad Collimator

5, 10 and 20 micron mini-beam defining apertures, 300 micron scatter guard apertures on one collimator



D

T

Back side views of Quad Collimators

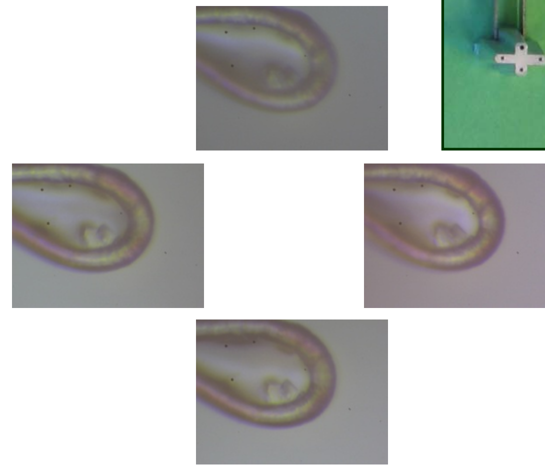
Visualization test results:

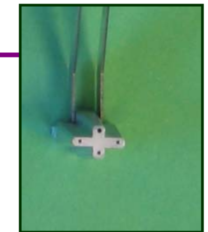
Visual obstruction of the triple collimator is marginal,
While Visual obstruction for the top position of the quad collimator is minimal.

No Collimator

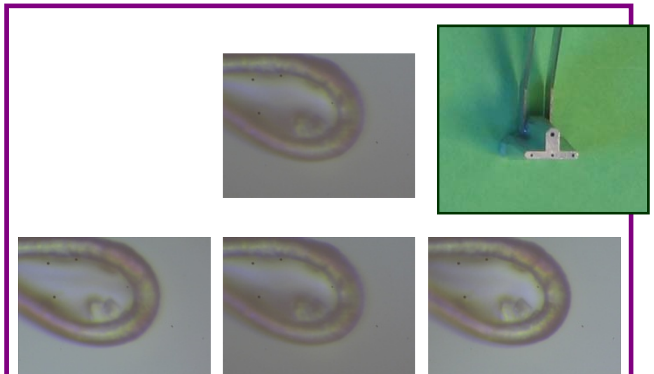
Triple Collimator

Quad Collimator I





Quad Collimator II

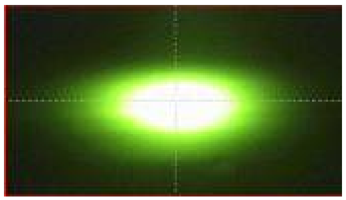


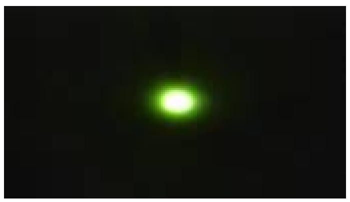
Sample: crystal size: 18 X 18 microns approx

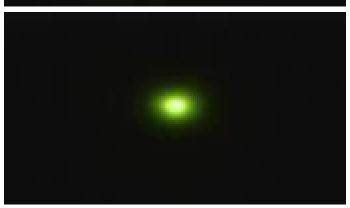
Testing at ID-D station. 2009_04_07
Zoom 15, ring light : 3.0 V,
Backlight: 4.000 V, Front light: 5.0V

Beam flux through collimator pinholes
Simulation and measurements

Beam properties for 23-ID-B and 23-ID-D





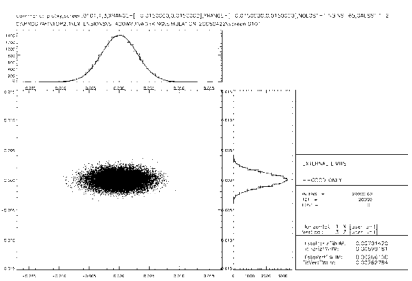


Beam	Size at sample, FWHM µm	Intensity Photons/sec	Flux density* Photons/sec/µm^2 (Intensity / beam FWHM)	Convergence µ-radians
Full	25 x 120 20 x 65	1.0 x 10^{13} 2.0 x 10^{13}	3.3 x 10^{9} 1.5 x 10^{10}	95 x 176 172 x 291
10- µm	10.6 x 11.6 10.5 x 10.8	1.3 x 10^{11} 5.2 x 10^{11}	1.1 x 10^{9} 4.6 x 10^{9}	103
5- µm	4.8 x 6.2 5.0 x 5.1	2.7 x 10^{10} 5.4 x 10^{10}	9.1 x 10^{8} 2.1 x 10^{9}	
1- µm	1.1 x 1.2	3.0 x 10^{9}	2.2 x 10^{9}	310

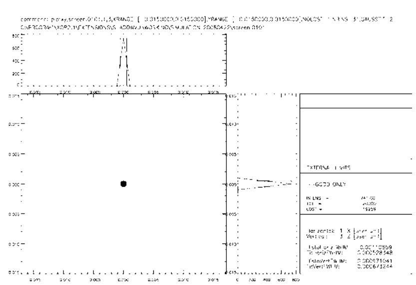
- Beam imaged on a YAG crystal mounted at the sample position.
- The pinhole selects the central part of the focused beam.

Simulation of beam flux through triple collimator pinholes (with “shadow”):

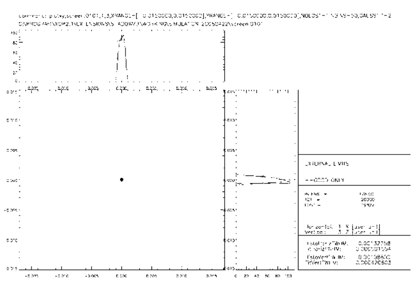
ID_IN	Flux through a 3.6mm x 1.3mm slit @ 60.98m	1.60E+14 (ph/s/0.1%BW)							
	Bandwidth defined by Si(1 1 1) (ΔE/E=1.4E-4)	2.10E+13 (ph/s)							
	Reflectivity of Rh of K-B mirror. 0.85	1.78E+13 (ph/s)*							
	Meatured beam size at Sample position	Intensity							
		no pinhole		Ø20 μ		Ø10 μ		Ø5 μ	
		rays	flux/(100mA)	rays	flux/(100mA)	rays	flux/(100mA)	rays	flux/(100mA)
	25μX 70μ at Sample position	20000	1.78E+13 (ph/s)*	2697	2.4E+12 (ph/s)*	714	6.35E+11 (ph/s)*	174	1.55E+11 (ph/s)*
	65μX 90μ (Beam focus after Sample position 300mm)	20000	1.78E+13 (ph/s)*	906	8.06E+11 (ph/s)*	226	2.01E+11 (ph/s)*	47	4.18E+10 (ph/s)*
ID_out	Flux through a 1.9mm x 0.6mm slit @ 28.35m	1.286E+14 (ph/s/0.1%BW)							
	Bandwidth defined by Si(1 1 1) (ΔE/E=1.4E-4)	1.694E+13 (ph/s)							
	Reflectivity of Rh of K-B and HDM mirror. 0.7237	1.376E+13 (ph/s)*							
	Meatured beam size at Sample position	Intensity							
		no pinhole		Ø20 μ		Ø10 μ		Ø5 μ	
		rays	flux/(100mA)	rays	flux/(100mA)	rays	flux/(100mA)	rays	flux/(100mA)
	25μX 120μ at Sample position	20000	1.376E+13 (ph/s)*	1606	1.1E+12 (ph/s)*	427	2.93E+11 (ph/s)*	111	7.6E+10 (ph/s)*



Full focused beam



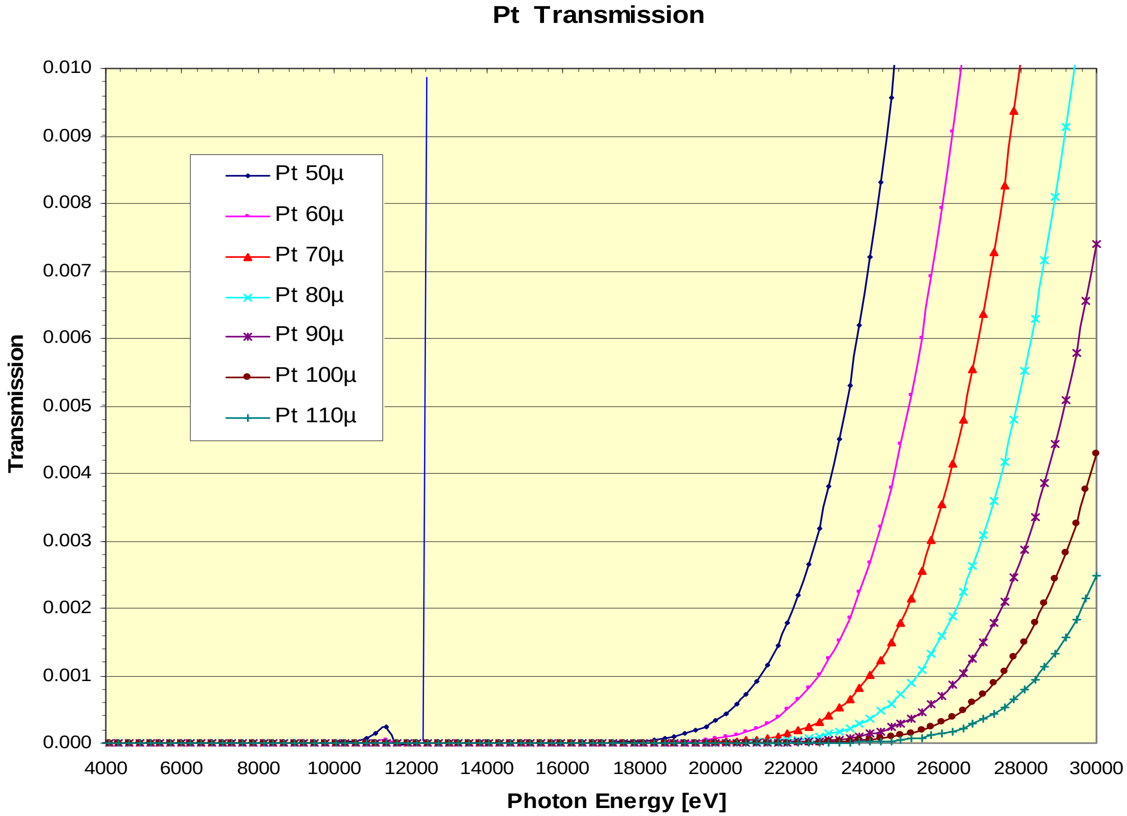
10μ beam

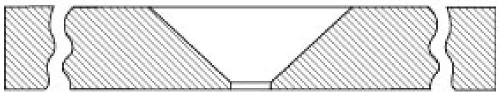


5μ beam

Material study

Material study of pinhole



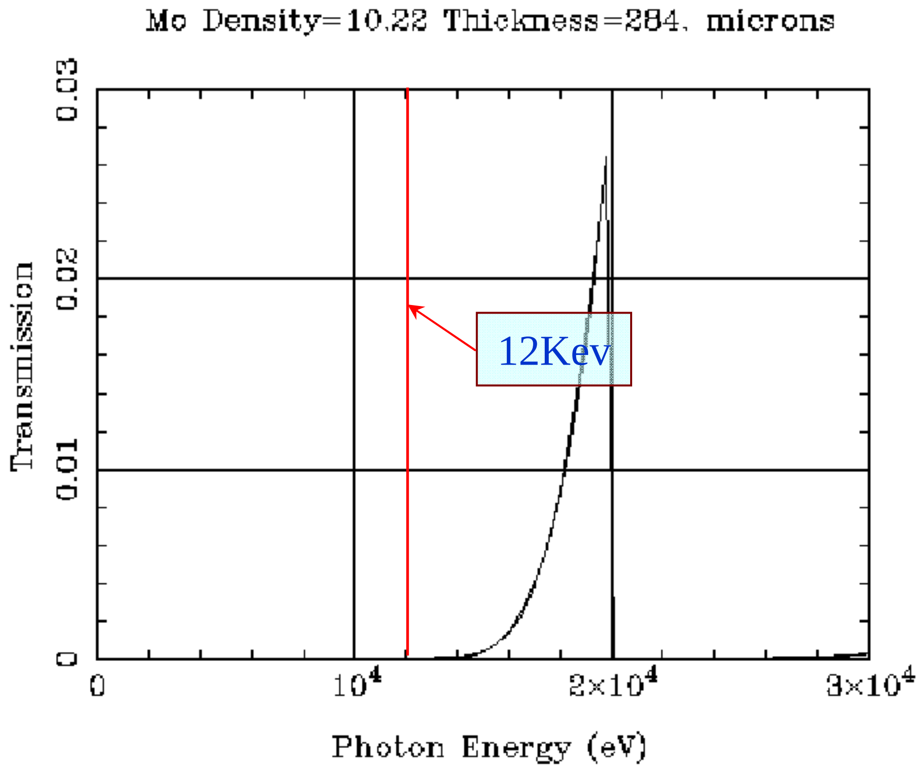


Source: Tedpella Inc.

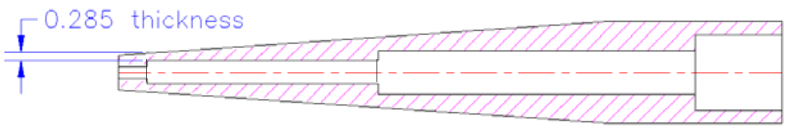
5µ pinholes only in Pt
Bigger ones in Mo

The mini beam size is defined by a 2mm diameter platinum disk with a pinhole in the center. The disk is 600 µm thick and tapers to 80-150 µm at the position of the aperture. The calculation results on the top, show that the transmission is negligible at 12 keV.

Material study of Molybdenum



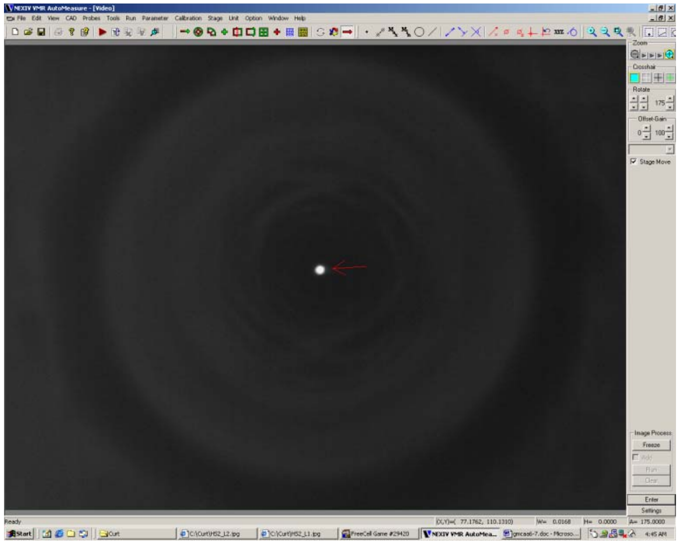
The minimal wall thickness of "Uni-body" is 0.28 mm

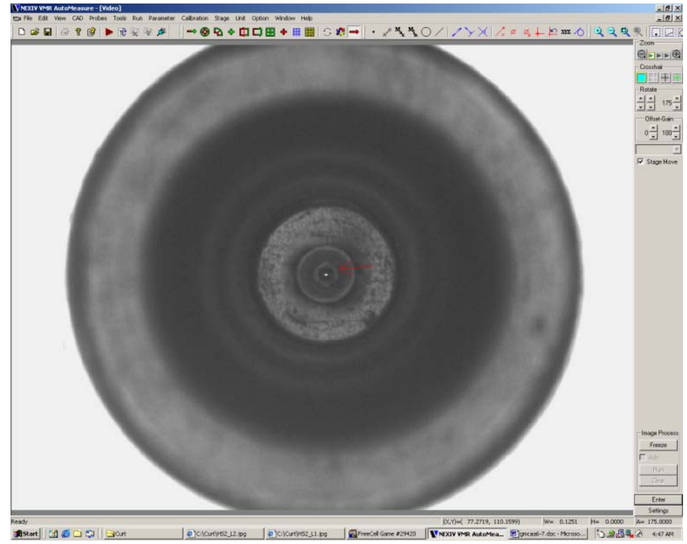


Transmission of Mo at 12Kev: 0.51E-6, is negligible

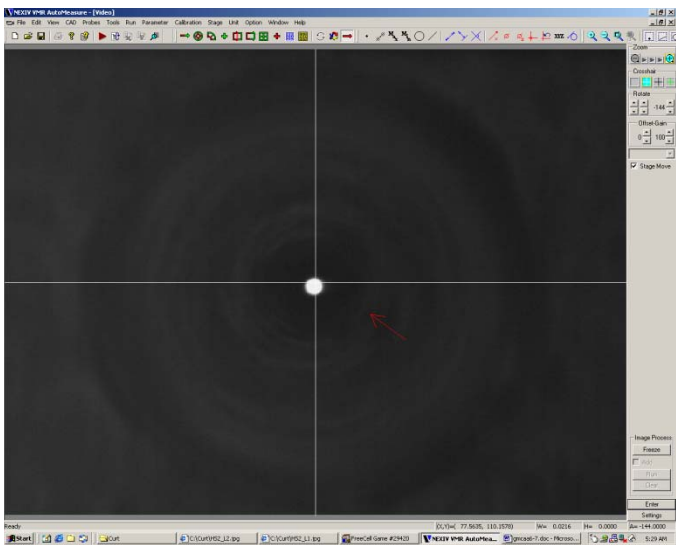
Inspection of aperture size
Nikon VMR – 3020 (CNC Video Measuring System NEXIV)

5µ A





10µ E



Pinhole (µ)	Number	Measurement results		
		1	2	3
Ø5	5-A 6-7	Ø4.2 µ	Ø4.3 µ	Ø4.3 µ
Ø5	5-E 6-7	Ø4.1 µ	Ø4.1 µ	Ø4.2 µ
Ø10	10-A 10-11	Ø8.5 µ	Ø8.5 µ	Ø8.4 µ
Ø10	10-C 10-11	Ø9.0 µ	Ø9.0 µ	Ø9.0 µ

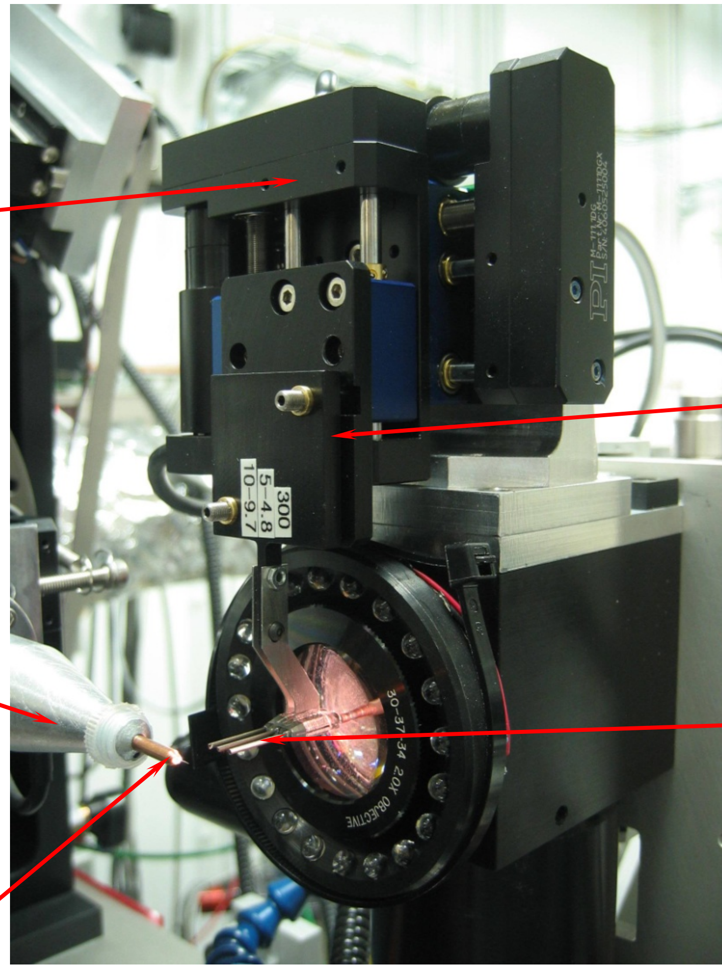
Aperture tolerance: +/- 1 µm for 5µm aperture
+/- 1.5 µm for 10µm aperture

Mounting and alignment - collimator mounting on on-axis sample-visualization (OAV) system

High Resolution Micro-Translation Stages

Goniometer

Sample



Kinematic Mount

Triple Collimator

XY Stages, Physik Instrumete

High-Resolution Micro-Translation Stages

M-110.1DG micro-translation stage (battery for size comparison).

M110, M-111 can be combined to xy and xyz systems for multiaxis Alignment applications

- 0.05 µm Minimum Incremental Motion
- 5, 15 and 25 mm Travel Ranges
- Velocity to 1.5 mm/sec.
- Closed-Loop DC Motors and Stepper Motors
- Integrated Hall-Effect Limit and Reference Switches

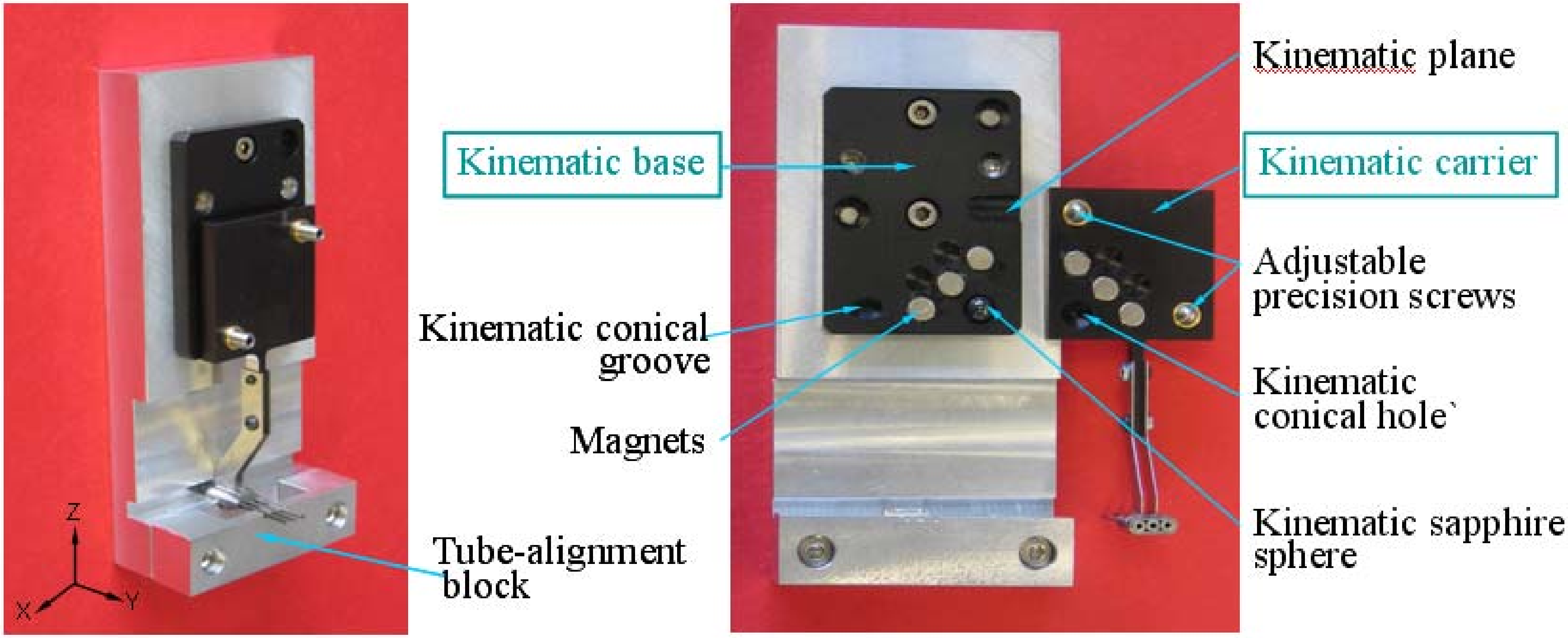
Technical Data

Models	M-110.1DG	M-111.1DG	M-112.1DG
Travel range	5	15	25
Design resolution	0.007	0.007	0.007
Min. incremental motion	0.05	0.05	0.05
Unidirectional repeatability	0.1	0.1	0.1
Backlash	2	2	2
Max. velocity	1	1.5	1.5
Max. normal load capacity	3	3	2
Max. push/pull force	10	10	10
Max. lateral force	10	10	10
Encoder resolution	2048	2048	2048
Motor resolution	-	-	-
Drive screw pitch	0.4	0.4	0.4
Gear ratio	28.44444:1	28.44444:1	28.44444:1
Nominal motor power	0.6	2	2
Motor voltage	12	12	12
Weight	0.3	0.4	0.5
Recommended motor controllers	C-843, C-848, C-862	C-843, C-848, C-862	C-843, C-848, C-862

* 2-phase stepper, 24 V chopper voltage, max. 250 mA / phase, 1,200 microsteps with C-600, C-630 control

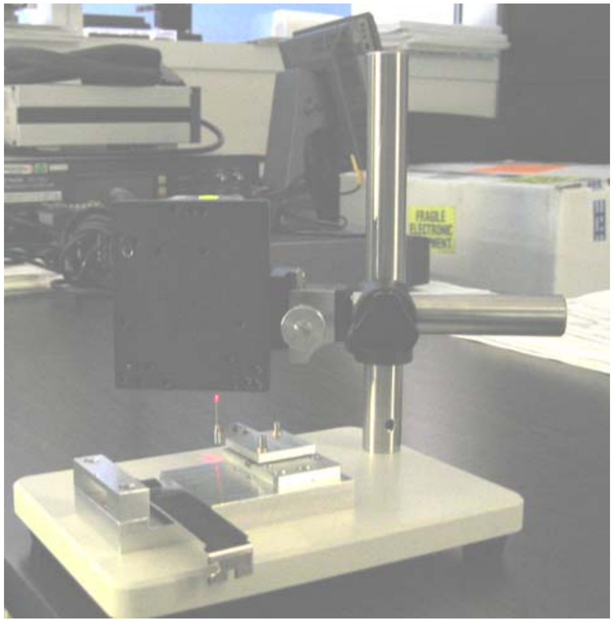
* See page 7-106 for notes and explanations.

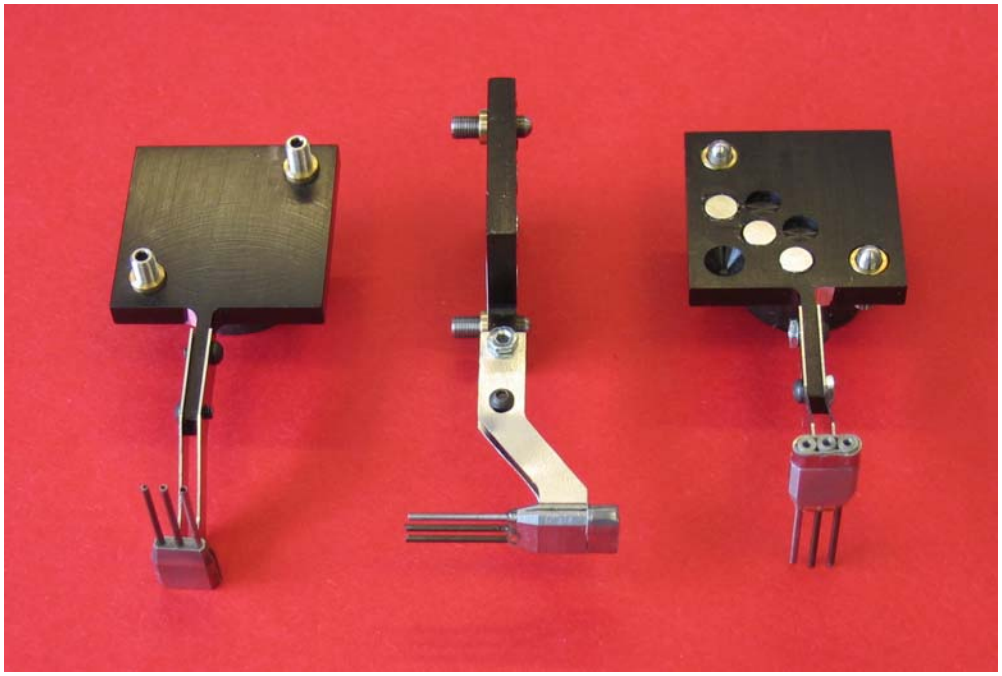
Pre-alignment jig



Pre-alignment jig: Align tubes and guard holder relative to Kinematic base

Kinematic mounting with High repeatability and stability





Measurement by Keyence Surface Scanning Lase Confcoal Displacement Meter (In Profile Mode)

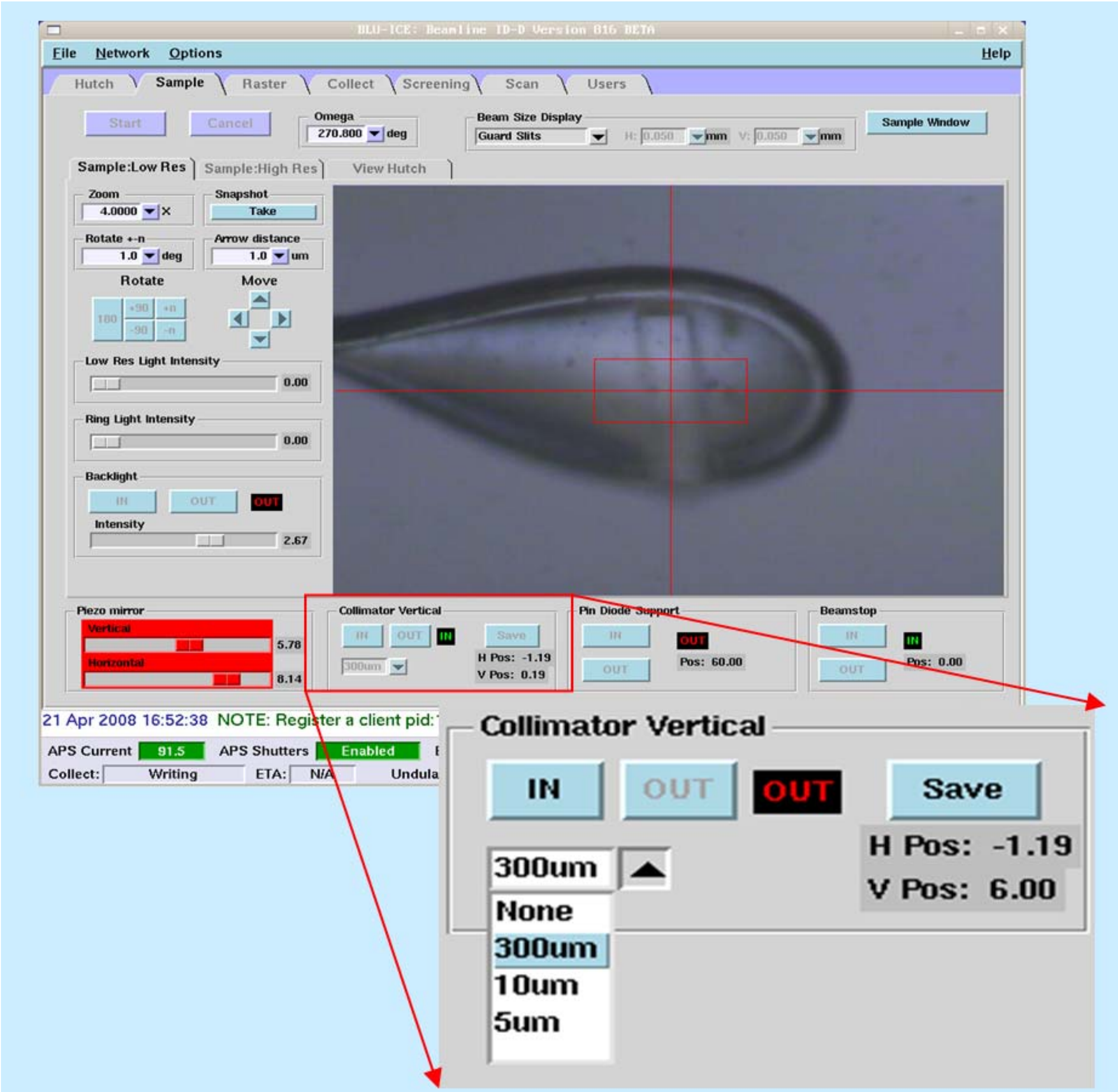
The positional reproducibility of the mini-beam collimator on the kinematic mount was measured by optical metrology. The RMS deviation from the mean position was 0.24 μm in both the X- and Z-directions for 34 repeated manual mount and dismount operations. The stability of the assembled mount was monitored in the X-direction once per minute over a period of 20 minutes. The RMS deviation from the mean X-position was 0.06 microns. The stability in the Z-direction was not measured, but is expected to be smaller than the X-direction.

Precision-alignment setting:

The optical axes of the three pinholes are adjusted to be co-parallel using the microscope before gluing.

The triple collimator is mounted on XY stages under the OLYMPUS SZX12 microscope.

User selectable beam size in Blu-Ice – pull down menu



- Recall stored positions
- Fast exchange - seconds
- Highly reproducible
- Retracted for sample mounting and alignment

User Comments on the Mini-beam

"We found that being able to use the minibeam as needed from the Blu-Ice tab was great. We had some crystals that responded better to a larger beam, and some where we got markedly improved signal-to-noise with the minibeam." - Petsko group

"We used the 10u beam. It worked very well, and many of our datasets (on small crystals, or crystals with good and bad spots) would not have been possible without it." - Kate Ferguson

"Perfect for our crystals. Data quality improved significantly by scanning the crystal using the 10 micron mini-beam" - Kornberg group

"We used the 10u beam on small and high mosaicity crystals. This resulted in better data with lower background and higher resolution." - Sylvie Doublie

"Several problematic crystals were put on. Our worse crystal was a heavily twinned plate. Before the minibeam, we were getting a smeared diffraction pattern. With the minibeam, a discernable diffraction pattern was observed due to being able to section off a thick piece. We could even index and scale the data this time!" - Sacchettini group

Quotes from end-of-run reports.

Results

Mini-beam has been a huge success at GM/CA CAT. About 30% of our users used the mini-beam when the collimators were single, after the implementation of triple collimator about 80% of users use the mini-beam for data collection. A few users come to GM/CA CAT exclusively to use the mini-beam and have successfully solved structures which might not have been otherwise possible

References:

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Thanks!