

# Focusing near and far with Bimorphs Mirrors

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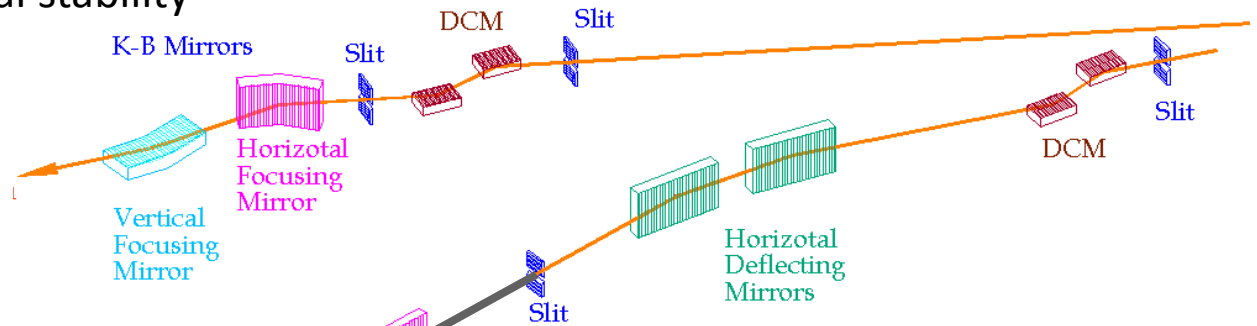
# Outline

- Current microcrystallography capabilities
- 1- $\mu\text{m}$  beam development for macromolecular crystallography
- In-situ, at wavelength metrology
- Far field beam profile
- Future plans

# GM/CA-CAT dual canted undulator beamlines at the APS

Rapid energy tunability  
High intensity and positional stability

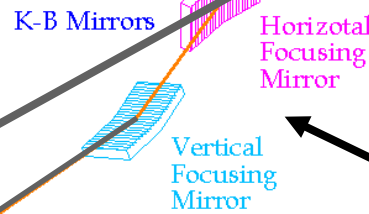
23-ID-D  
5 – 20 keV  
20 x 65  $\mu\text{m}^2$



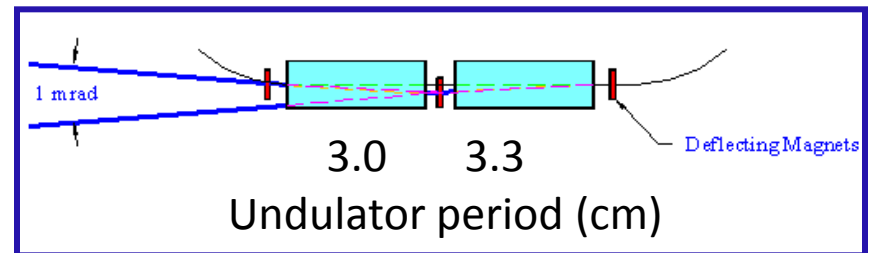
Zone plate

1  $\mu\text{m}$   
5, 10, 20  $\mu\text{m}$

23-ID-B  
3.5 – 20 keV  
25 x 120  $\mu\text{m}^2$



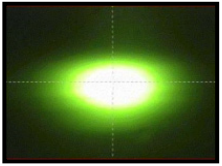
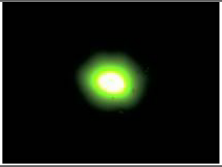


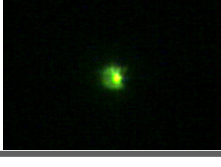
Bimorph mirrors



Large unit cells of biomolecules  
→ low convergence optics

3.0 cm device optimized for Se MAD phasing

# Beam properties

Beam		Size at sample, FWHM ( $\mu\text{m}$ )	Intensity (Photons/sec)	Flux density (Photons/sec/ $\mu\text{m}^2$ )	Convergence ( $\mu$ -radians)
Full		25 x 120 20 x 65	1.0 x 10 <sup>13</sup> 2.0 x 10 <sup>13</sup>	3.3 x 10 <sup>9</sup> 1.5 x 10 <sup>10</sup>	176 x 95 305 x 172
20- $\mu\text{m}$		20 $\emptyset$	5.0 x 10 <sup>11</sup> 1.0 x 10 <sup>12</sup>	2.0 x 10 <sup>9</sup> 3.0 x 10 <sup>9</sup>	
10- $\mu\text{m}$		10 $\emptyset$	1.3 x 10 <sup>11</sup> 5.2 x 10 <sup>11</sup>	1.1 x 10 <sup>9</sup> 4.6 x 10 <sup>9</sup>	103
5- $\mu\text{m}$		5 $\emptyset$	2.7 x 10 <sup>10</sup> 5.4 x 10 <sup>10</sup>	9.1 x 10 <sup>8</sup> 2.1 x 10 <sup>9</sup>	
1- $\mu\text{m}$		1 $\emptyset$	3.0 x 10 <sup>9</sup>	2.2 x 10 <sup>9</sup>	310 ( $<0.02^\circ$ )

23-ID-B 23-ID-D

# Comparing LTP and at wavelength metrology

## Slope error specification

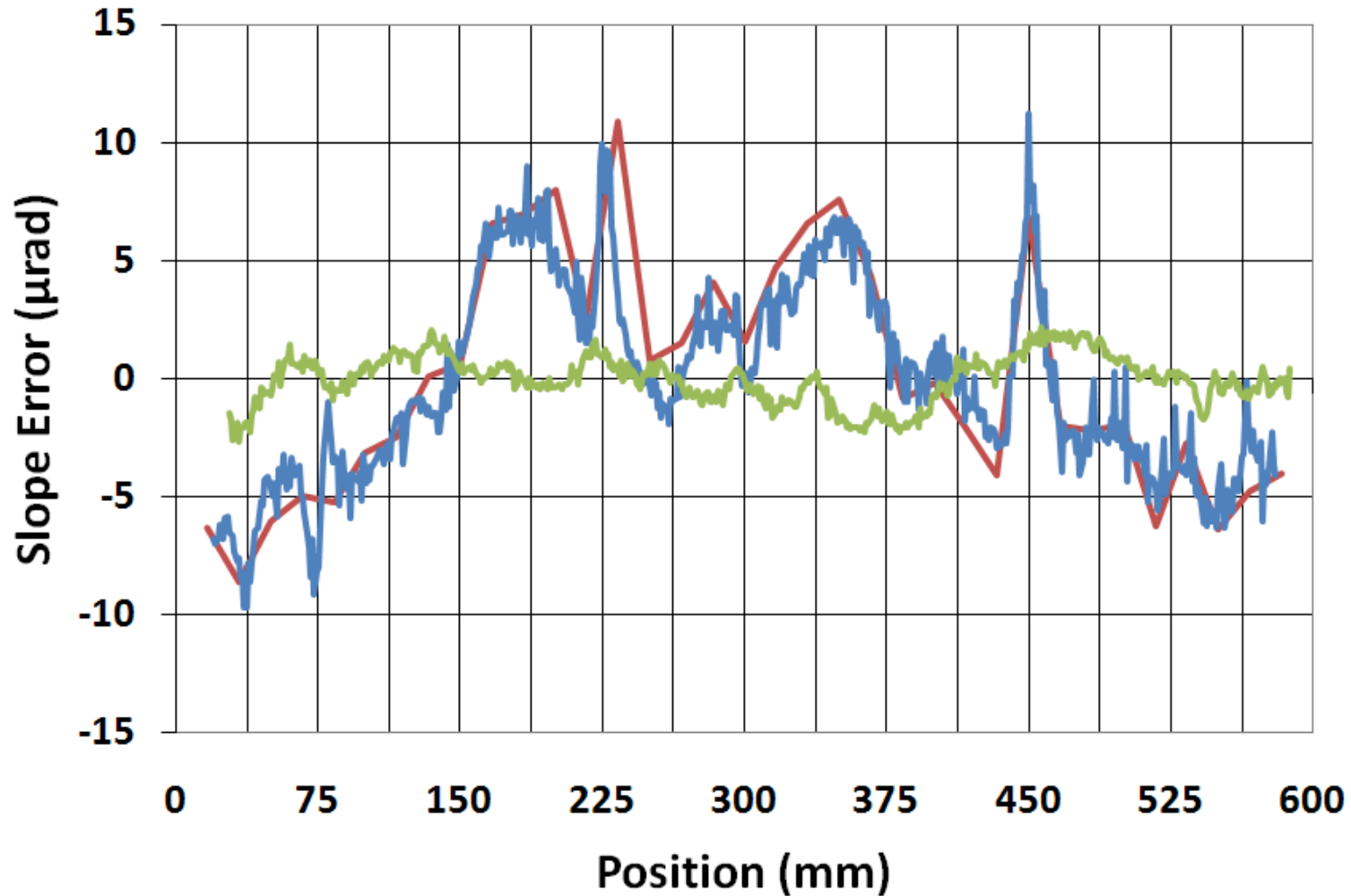
Original specification: 2.5  $\mu\text{rad}$  in 2003

Repolished : 1.0  $\mu\text{rad}$  in 2007

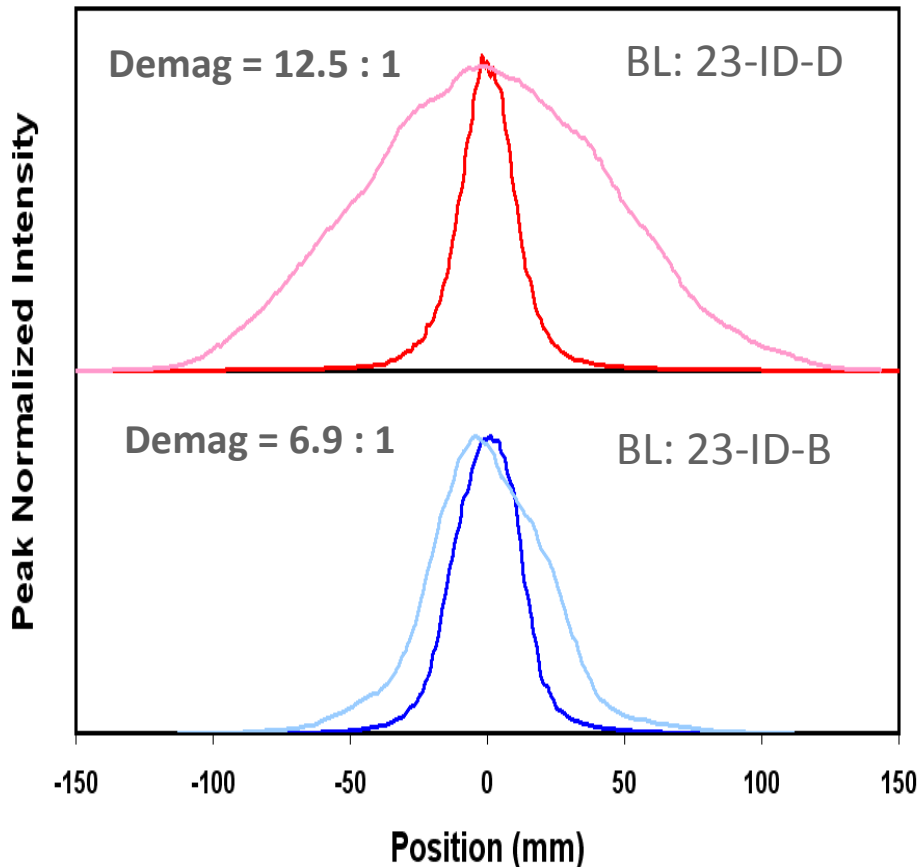
Blue – LTP before repolish

Red – in situ; before repolish

Green – LTP after repolish



# Vertical focusing with “bi-morph” mirrors: unstructured profile at and off focus, automated focusing



- State-of-the-art slope error for 600mm long mirrors
  - <math><1.0\ \mu\text{rad}</math> RMS – uncorrected
  - $\sim 0.5\ \mu\text{rad}$  RMS – corrected
- Auto-focusing algorithm implemented
- Gaussian beam shape on/off focus
- Residual slope error
  - Determines minimum beam size
  - Effects beam positional stability
- GM/CA-CAT designed gravity compensator is better than the one provided by SESO

Derek Yoder

# Relative Dimensions of small to largest Macromolecules Studied by X-ray Crystallography

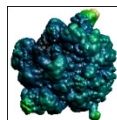
Science August 27, 2010  
 3.5 Å resolution  
 Vijay Reddy and Glen Nemerow  
 The Scripps Research Institute

$a=854.03 \text{ \AA}$   
 $b=855.17 \text{ \AA}$   
 $c=865.24 \text{ \AA}$

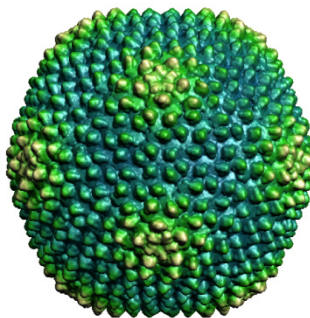
**Myoglobin**  
(2W6X)



**Ribosome**  
(70S; 2X9S)

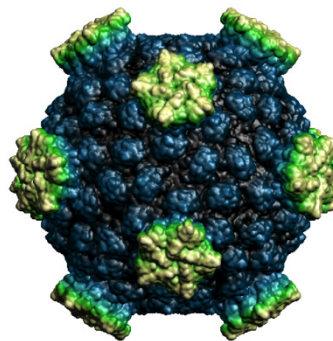


**Bacteriophage**  
PRD1; 1W8X



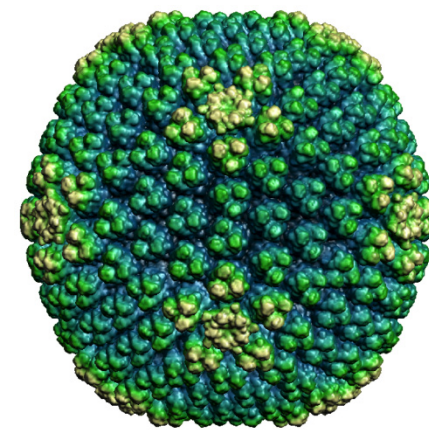
pT=25

**Reovirus core**  
(1EJ6)



T=1

**Adenovirus**  
(1VSZ/2BLD)



pT=25 ← T number

<b>Dia. (Å):</b>	42	280	702	754	960
<b># a.a. in IAU:</b>	153	3,394	4,915	4,370	15,155
<b>DNA/RNA (Kbp):</b>	N/A	3.3	14.9	23.5	35.9

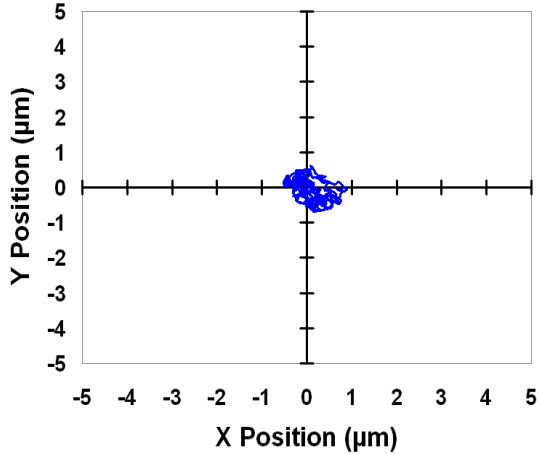


# Micro-crystallography developments

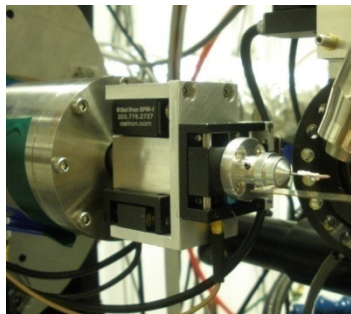
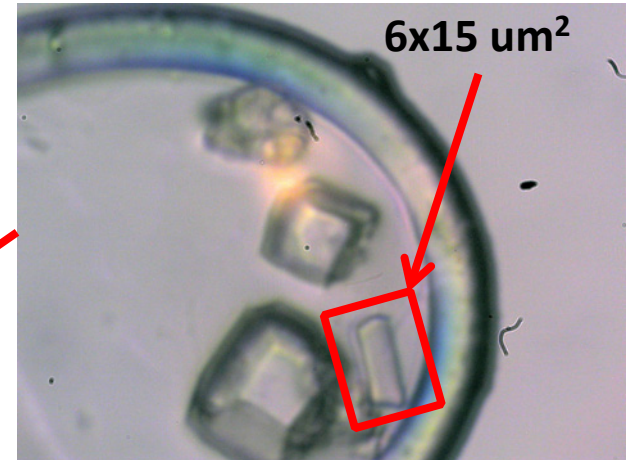
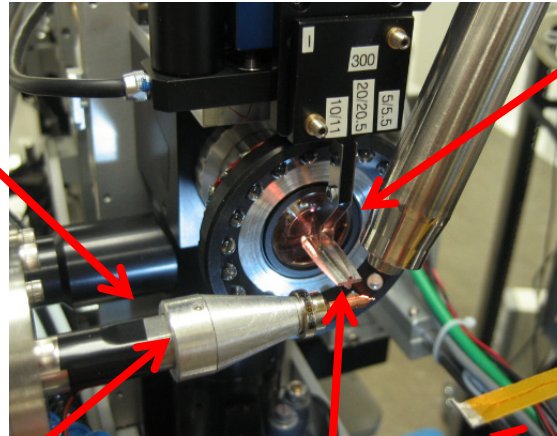
On-axis sample visualization

Goniometer:

1  $\mu\text{m}$  SOC peak-to-peak



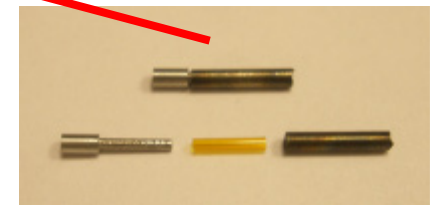
Sample environment



Goniometer head:  
nano-positioning



Quad mini-beam collimator:  
5, 10, 20- $\mu\text{m}$  beams and  
300-  $\mu\text{m}$  scatter guard



Active beamstop:  
Photoelectric effect



# Auto-beam profiling at sample

23-ID-in  
WebIce

- Blulce Configuration
- Blulce Assistant
- Active Beamstop
- Frame Audit Display
- Frame Audit advanced start
- CCD Distance Calculator
- Keithley-428 Signal Amplifier
- Cryojet Temperature Controller
- Anneal sample
- Beam intensity feedback
- Align beam or minibeam menu
- Align beam to 5um pinhole & measure beam size**
- Optimize white beam slits
- Optimize monochromatic beam slits
- Optimize Fluorescent Detector
- Minibeam tools
- Sample Mounting Robot
- Video Snapshots
- Bimorph Focusing Mirrors
- Acromag IP330 ADC
- Systran DAC-128V 12-bit
- Hytec DAC8402 16-bit
- PID Records (Feedback)
- Acromag-9440 Digital I/O
- High Voltage Power Supplies
- vxWorks & save/restore stats
- Backup/Restore Tool (BURT)
- Users Controls

JBlulce-EPICS: Be

Collect Screen

Omega 10.000 degr

10.000 A

High Res View Hutch

X 0.000

um

0.000

0.000

0.000

Collimator Controls

Beam Controls ScatterGuard IN OUT V: 10.0 H: -1.27

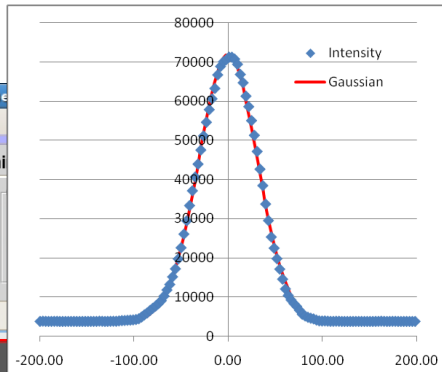
Pinodiode OUT IN V: 135

Beamstop IN OUT Take Snapshot

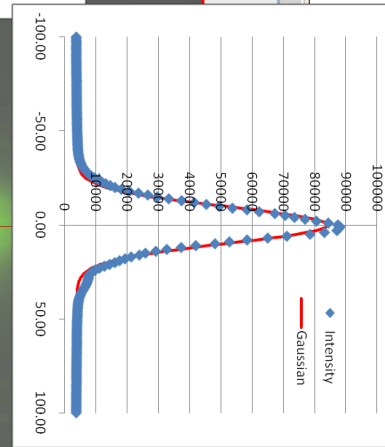
[15:26:57] NOTE: [collimatorCtrl.pl]

APS Current 89.2 Shutter Permit Enabled A Shutter Open Endstation Shutter Open Endstation Secure Yes

State: Idle ETA: --- EMERGENCY STOP Mono: 12.000 keV IZero: 0.30 V Control: Active Shutter: Open



Horizontal FWHM = 62.5  $\mu\text{m}$



Vertical FWHM = 18.8  $\mu\text{m}$

Reduce the beam size to 35 x 15  $\mu\text{m}^2$  with slits

# Rapid beam size selection

JBIulce-EPICS: Beamline ID-D Version 2010.2 Build 2618

File Network Options Help

Hutch Sample Raster Collect Screening Scan Users Log

Start Cancel Omega 10.000 deg 10.000 A

Centering Auto Centering Loop Crystal 3Click Centering Stop

Beam Size Display Sum Clmtr H: 0.0050 V: 0.0050 Sample Window

Sample: Low Res Sample: High Res View Hutch Sample: Tools Diff. Image

HighRes Zoom 8.00 x 8.00 A

Rotate +-n 1.00 deg 180 +90 -90 +n -n

Move XY 1.0 um

HighRes Intensity 0.000

Ringlight Intensity 0.000

Backlight IN OUT

Backlight Intensity 0.000

Beam Controls Collimator Controls Sum OUT IN V: 4.48 H: -1.28 Pindiode OUT IN V: 135 Beamstop IN OUT Take Snapshot

[15:31:50] NOTE: ColCtl: move OK!

APS Current 89.1 Shutter Permit Enabled A Shutter Open Endstation Shutter Open Endstation Secure Yes

State: Idle ETA: --- EMERGENCY STOP Mono: 12.000 keV IZero: 2.72 V Control: Active Shutter: Open

Collimator Controls

- None
- ScatterGuard
- 20um
- 10um
- 5um

# GM/CA-CAT Bimorph Mirrors

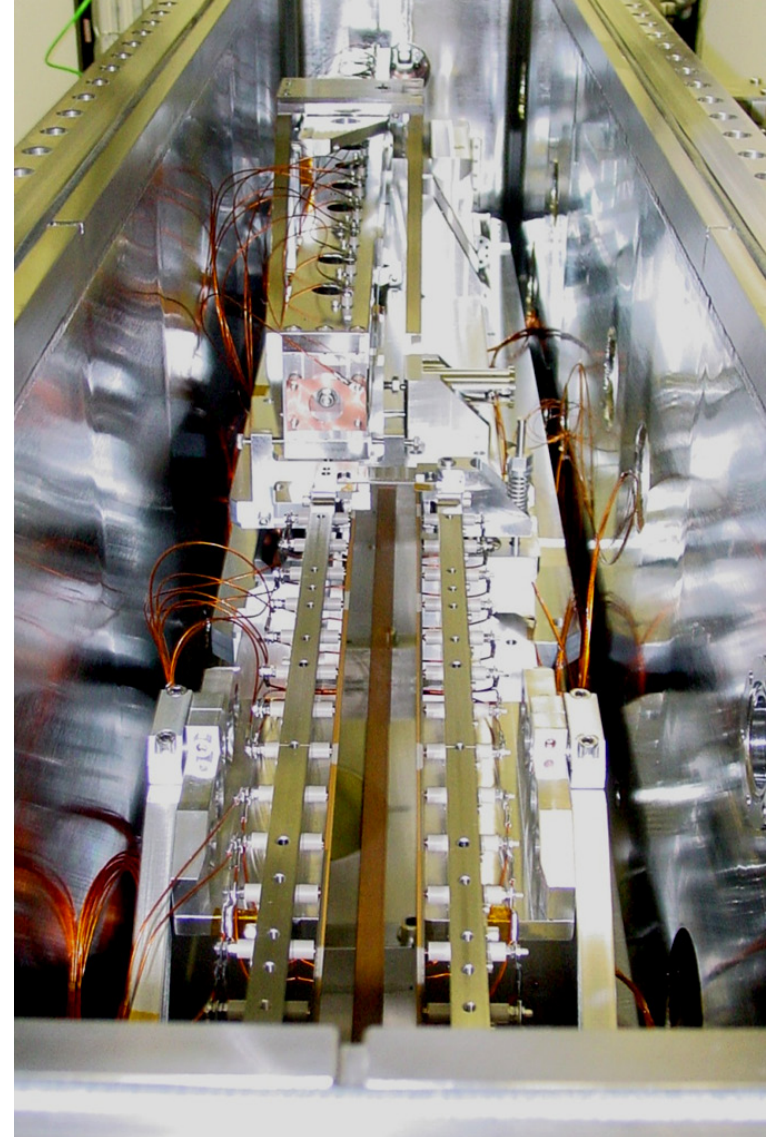
- SESO mirrors
- Kirkpatrick-Baez geometry
- $\text{SiO}_2$ , Pt, or Rh stripes for harmonic rejection

## Horizontal mirror:

- length = 1.05 m
- 3.15 mm acceptance @ 3 mrad
- 2.5 Å roughness, 2.5  $\mu\text{rad}$  slope error
- 14 electrodes

## Vertical mirror:

- length = 0.60 m
- 1.80 mm acceptance @ 3 mrad
- 2.5 Å, 2.5  $\mu\text{rad}$   $\rightarrow$  1.0  $\mu\text{rad}$
- 16 electrodes



# Power Supplies and Web GUI

- Precision, high voltage power supply from Elettra
- Bipolar output to  $\pm 2000$  V
- Protection for neighboring electrodes ( $\Delta V_{\max} = 500$  V)
- Controller includes focus software, ethernet connectivity, and web interface



High Voltage Power Supply

Group "23i:VFM" - Modify

00	01	02	03	04	05	06	07	08	09	10	11	12
Set (V)	Set (V)	Set (V)	Set (V)	Set (V)	Set (V)	Set (V)	Set (V)	Set (V)	Set (V)	Set (V)	Set (V)	Set (V)
Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)	Shift (V)
101 V	199 V	174 V	161 V	108 V	-5 V	125 V	26 V	96 V	76 V	72 V	-21 V	134 V
Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)	Trgt(V)

ALL CHANNELS

Set Voltages: Set (V)

Shift Voltages: Shift (V)

Set Target Voltages: Trgt(V)

Power:  ON  OFF

Op. Mode:  HI accuracy  Normal  Fast

Last Error:

# Control via EPICS: via Perl Utilities

- EPICS driver and MEDM screens created by ACCEL and modified by GM/CA
- GM/CA has created a perl script library to facilitate basic mirror functions and has used this library to create a number of mirror utilities:
  - Voltage backup
  - Voltage restore
  - auto-alignment of a mirror
  - Auto-focusing
- Auto-focusing
  - Implemented in 2008
  - Used routinely

**MirrorBimorph\_all\_14.a**

Channel	HctPos	RqsPos	Rqs
CH00	-136.00000	0.000	0.000
CH01	-176.00000	0.000	0.000
CH02	-83.00000	0.000	0.000
CH03	-231.00000	0.000	0.000
CH04	-368.00000	0.000	0.000
CH05	-197.00000	0.000	0.000
CH06	24.00000	0.000	0.000
CH07	-146.00000	0.000	0.000
CH08	-104.00000	0.000	0.000
CH09	-390.00000	0.000	0.000
CH10	-209.00000	0.000	0.000
CH11	-82.00000	0.000	0.000
CH12	-251.00000	0.000	0.000
CH13	-369.00000	0.000	-344.00000

**23i Mirror Focusing Matrix**

Focus profiler: gonio  
XY positioner number: default  
Mirror: VFM (selected) HFM  
Mirror angle: auto  
Segment sampling: 2  
Ignored edge beamlets: 0  
Mirror pulse: 15. V  
After-pulse delay: 300 s  
Scan Type: ScanRec (selected) HardSync  
Analyzer time/pt: 0.02 s  
Analyzer +/- range: 0.10  
Analyzer step: 0.001  
Centroid discriminator: 0.2  
Struck input for I0: 18  
Struck input for I1: 17  
Struck input for I2: 16  
Calc.correction matrix: internal  
BPM counting time: 1 s  
Attenuation Option: Use Attenuation (checked)  
MEDM Option: Show MEDM (checked)  
Video Profiler Option: Save images (unchecked)  
Output file prefix: focus

Buttons: GO!, Simulate, ctdUpload, Cancel

Bottom status: ALL VOLT 5.000 V, ALL SHIFT 20.000 V, LastErr: 9900, GO TARGET, Reset LastErr, Err Help, mode=0, 0=Accu, 1=Norm, 2=Fast, More

# Automated Focusing

- A 14- (or 16-) dimension linear minimization problem (see Signorato, et al., *J. Synchrotron Rad.* (1998), **5**, 797-800)
- Obtain the interaction matrix describing the response of each portion of the mirror to voltage pulses
- The interaction matrix, combined with the error vector describing the current state of the mirror, provides the correction needed to focus the beam
- Typical time for collection of interaction matrix is 2-3 hours

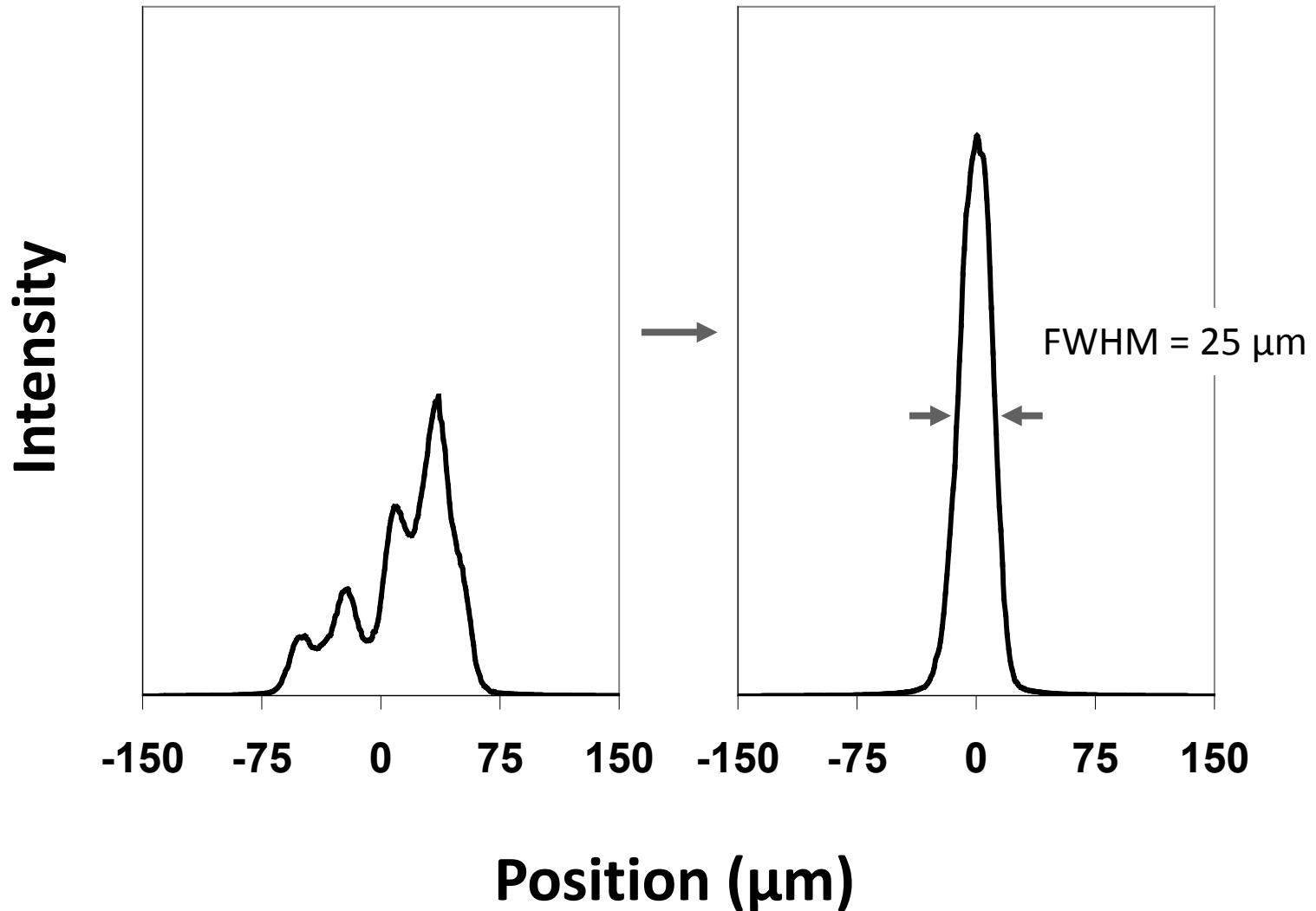
is). By knowing  $\mathbf{H}$ , the solution to the least-squares minimization of a given measured distortion  $\delta\mathbf{f}_0(x_i)$  is given by:

$$\begin{pmatrix} V_{D,1} \\ V_{D,2} \\ V_{D,3} \\ V_{D,4} \\ V_{D,5} \end{pmatrix} = (H^T H)^{-1} H^T \delta f_0,$$

$T$  is the standard notation for the transposed matrix. An inverse singular value decomposition (SVD) method is used to avoid singularities of  $\mathbf{H}$ .

**Interaction Matrix:  
Response of  
32 positions to  
16 voltage pulses**

# Automated focusing since 2008



# Crystallography with micron (and smaller) beams

What are the science drivers for micro-crystallography?

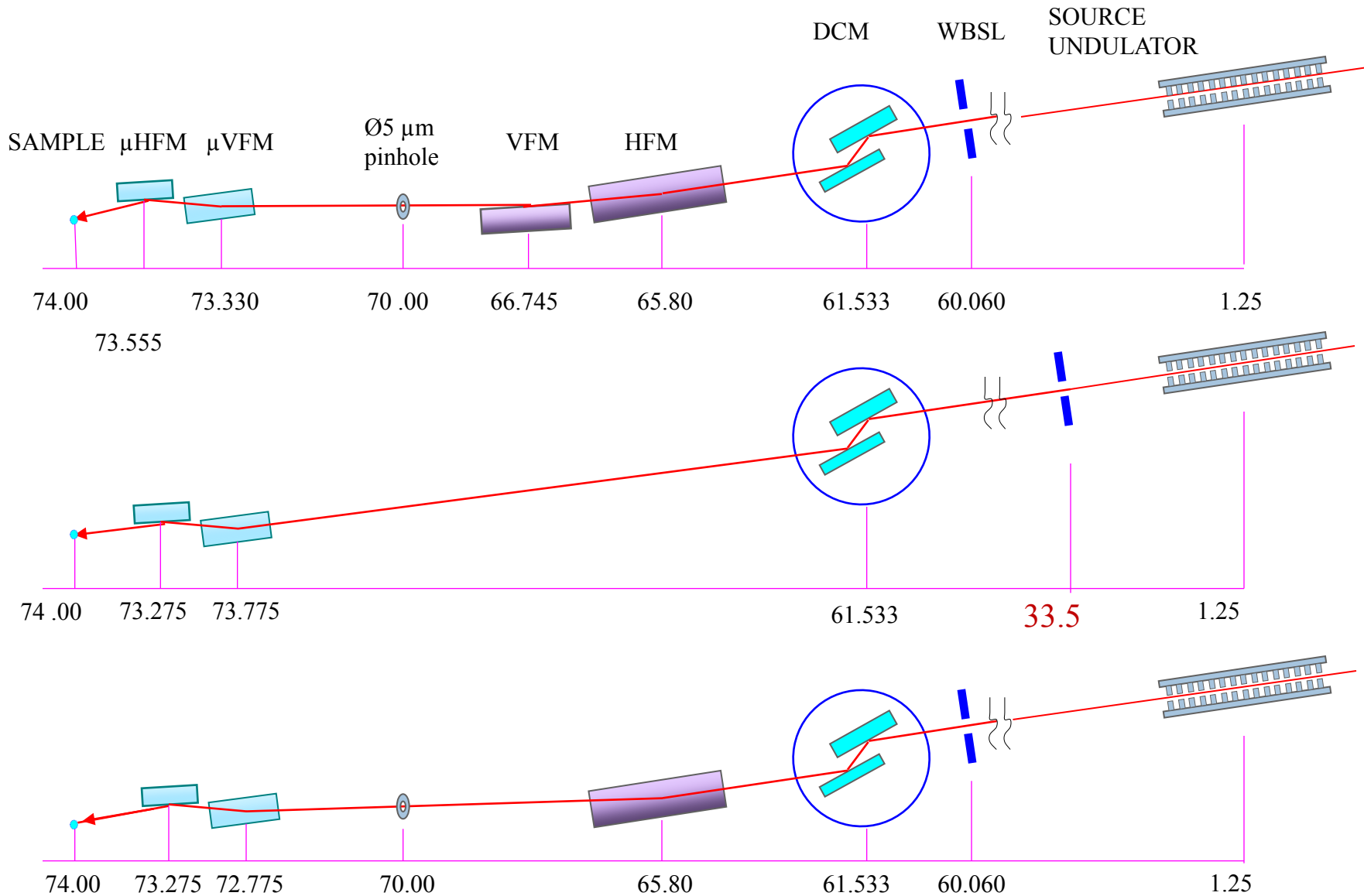
- Smaller crystals
  - membrane proteins
  - protein and RNA complexes
- Inhomogeneous crystals
  - probe to find more ordered regions
- Radiation damage
  - expose fresh crystal
  - reduce radiation damage by understanding behavior of photoelectrons



# What is so challenging about sub-micron beams?

- Achieve the desired size ***while maintaining a small divergence***
  - Large divergence prevents resolution of diffracted spots
- What is a small divergence?
  - Good cryo-cooled crystals have a mosaicity of about  $0.1^\circ = 1.7 \text{ mrad}$
  - Ideally, would like to keep divergence below 200 - 300  $\mu\text{rad}$
- Considerations:
  - Trade-off: % beam collected by focusing optic vs. beam divergence
  - Increasing focal length
    - keeps divergence low
    - reduces the demag
    - amplifies the effects of slope error
      - beam blur
      - source motion

# ID-in beamline option-1, 3 and 5



# Summary beam properties for micro-focus layouts

	Option 1a		Option 3		Option 5c	
	Horz	Vert	Horz	Vert	Horz	Vert
<b>Energy = 18.5 keV</b>						
Source size (FWHM) ( $\mu\text{m}$ )	642	21	642	21	642	21
Source divergence (FWHM) ( $\mu\text{rad}$ )	21	10	21	10	21	10
2nd source size (FWHM) ( $\mu\text{m}$ )	5	5	40		5	
2nd source divergence (FWHM) ( $\mu\text{rad}$ )	362	238			362	
Working distance (m)	0.37		0.575		0.52	
Mirror RMS slope error ( $\mu\text{rad}$ )	0.15	0.15	0.15	0.15	0.15	0.15
Focal size (FWHM) ( $\mu\text{m}$ )	0.99	0.63	0.89	0.94	0.99	0.89
Focal convergence (FWHM) ( $\mu\text{rad}$ )	894	1008	792	584	1343	612
Final flux (photons/sec)	1.39E+11		1.27E+12		1.36E+12	
Focal brilliance (ph/sec/mm <sup>2</sup> /mrad <sup>2</sup> / <b>0.0141% BW</b> )	2.45E+17		3.3E+18		1.85E+18	

# “Optimized” micro-focus configurations

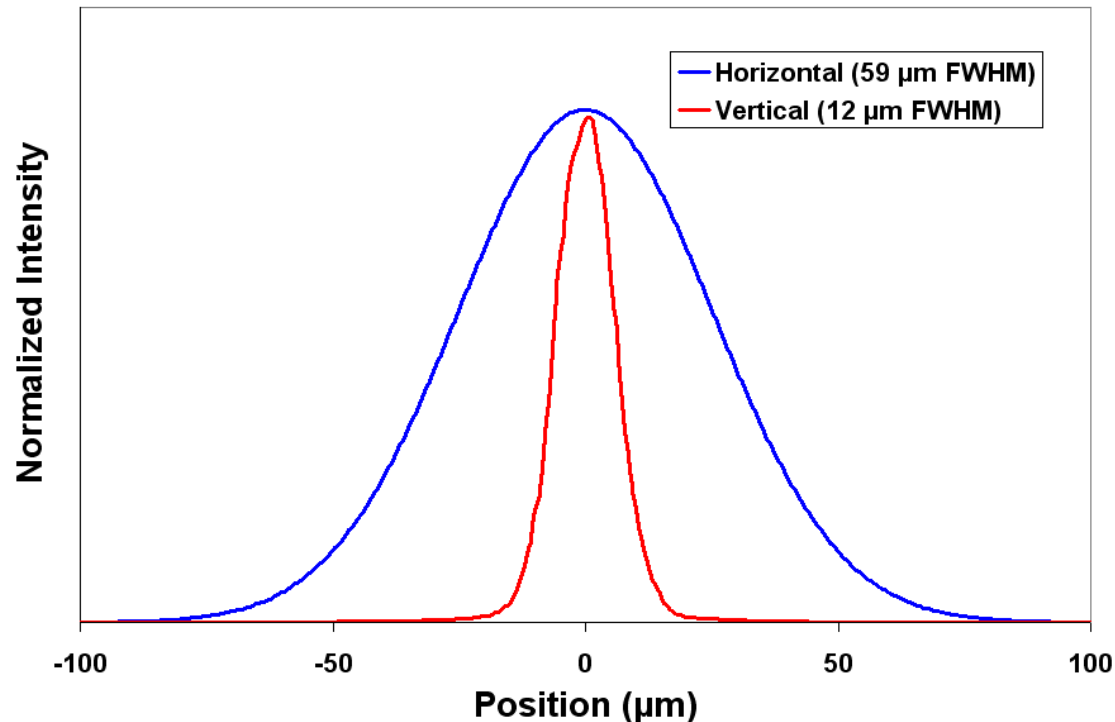
- Option 1 – KBM, MBSL 2<sup>nd</sup> source,  $\mu$ KBM
  - Pros
    - » Proven
    - » Seemed more stable than option 3 over period of hours
    - » Lowest cost
  - Cons
    - » Lowest intensity and brilliance
    - » Very short working distance
- Option 3 – WBSL 2<sup>nd</sup> source,  $\mu$ KBM
  - Pros
    - » Lower horizontal convergence
    - » Variable horizontal beam size is easy
  - Cons
    - » More expensive - requires adding a WBSL
    - » Experience showed alignment tended to drift
- Option 5 – HFM, MBSL 2<sup>nd</sup> source,  $\mu$ KBM
  - Pros
    - » Highest intensity
    - » Variable horizontal beam size is easy
  - Cons
    - » Highest convergence

# “Dynamic” micro-focus configurations

- Option 6 – similar to option 5 but push HFM focus downstream
- WBSL 2<sup>nd</sup> source issues
  - Options:
    - » Fixed or movable mask(s) in the FE at 22 m
    - » Invert existing IDA mask, then add a fixed mask for IDout somewhere
    - » New, shorter, IDin mask at 27.313 m for 150 mA
    - » New mask at >33.5 m, possible only horizontal defining
  - Add water cooling and monitoring into EPS
- MBSL 2<sup>nd</sup> source issues
  - Need to design precision adjustable slits

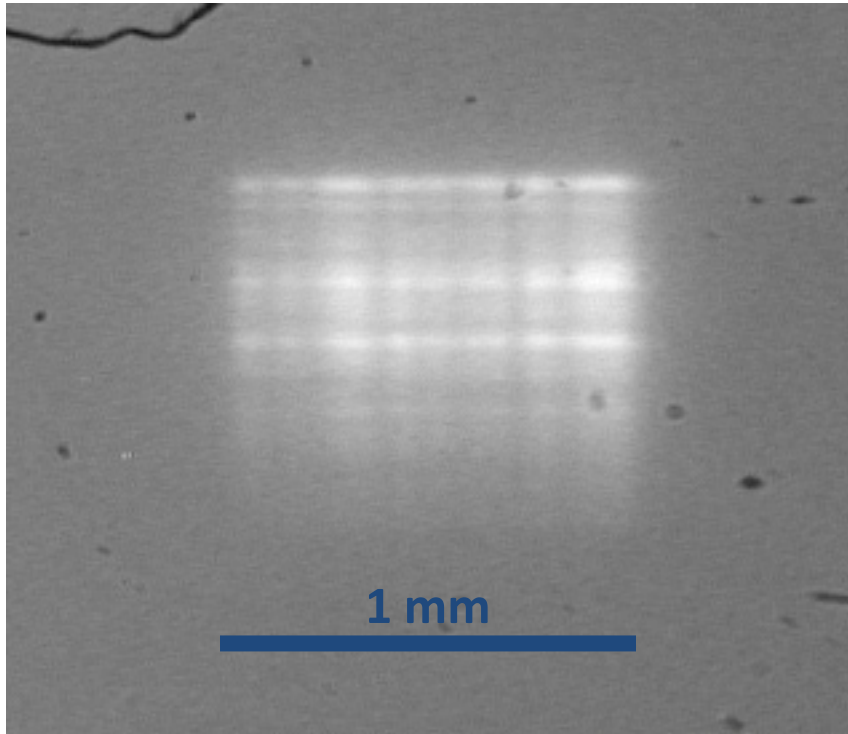
# Large K-B bimorph mirrors focused tightly

2<sup>nd</sup> source:  $59 \times 12 \mu\text{m}^2$

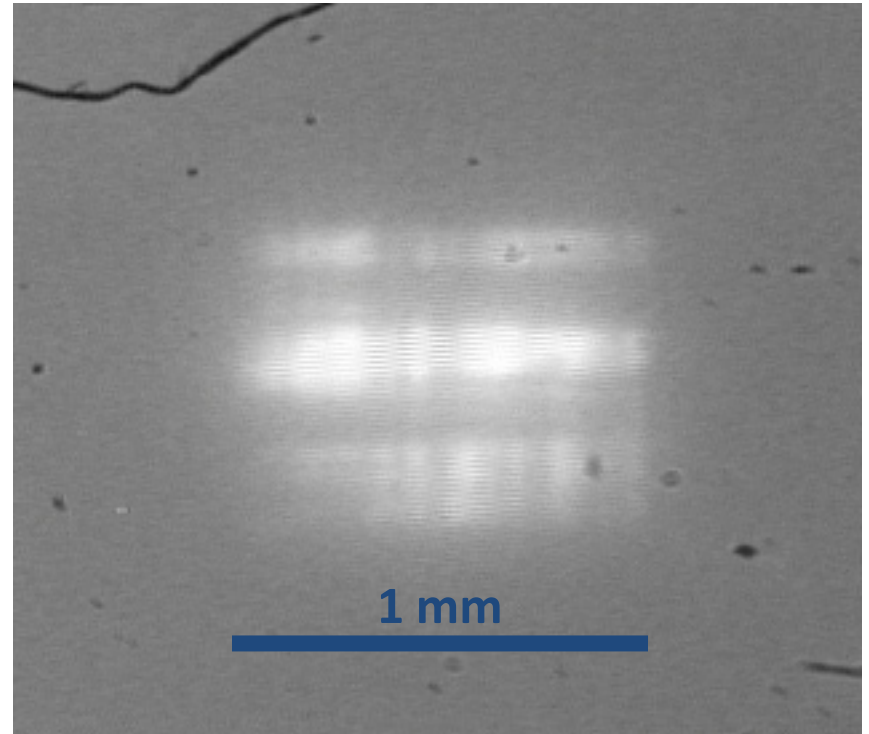


Mirrors are bent to neat their elastic limit  
Clean focus

# Visual image of beam off-focus



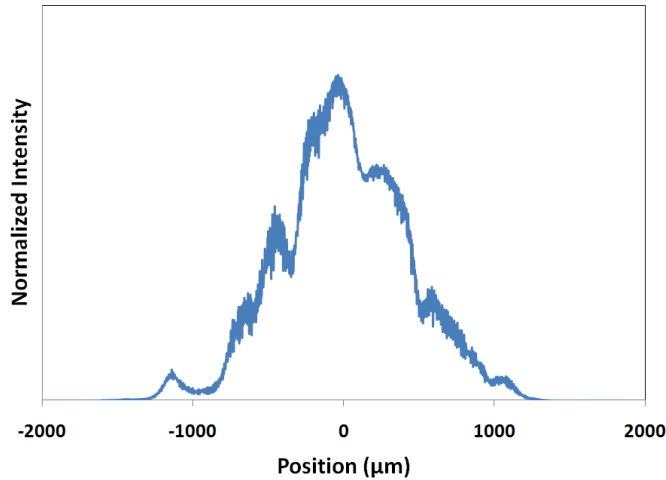
Without pinhole



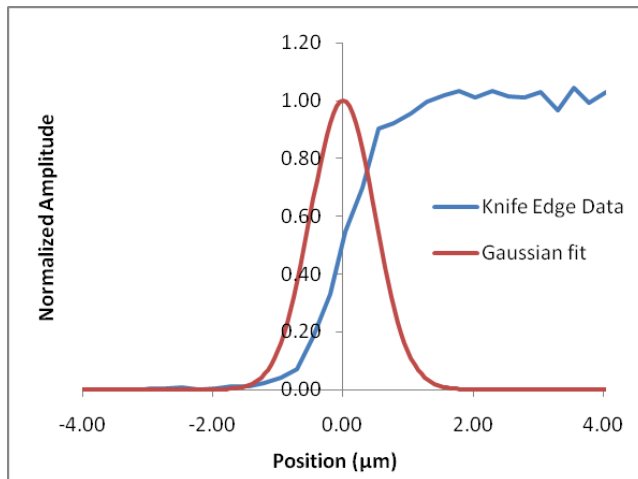
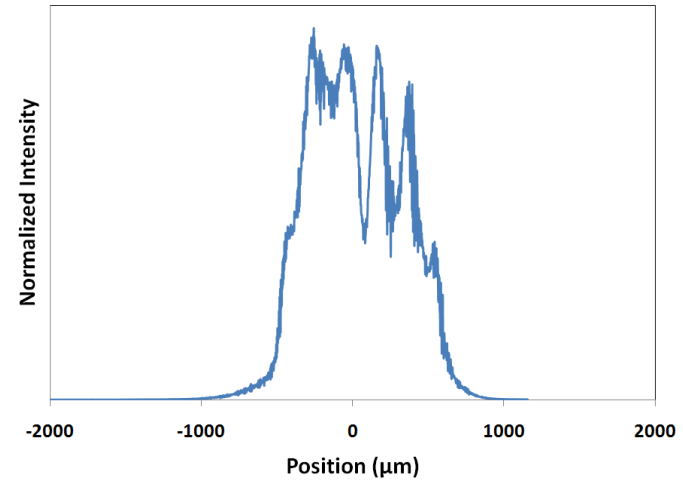
With pinhole

# Beam profile at Zone Plate and final focus

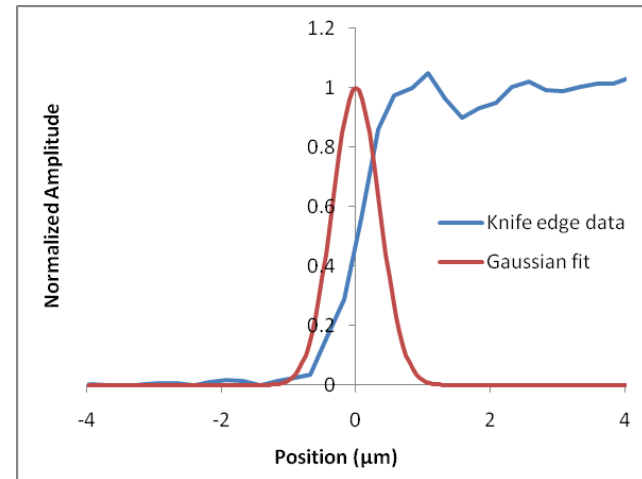
## Horizontal



## Vertical



FWHM = 1.16  $\mu\text{m}$

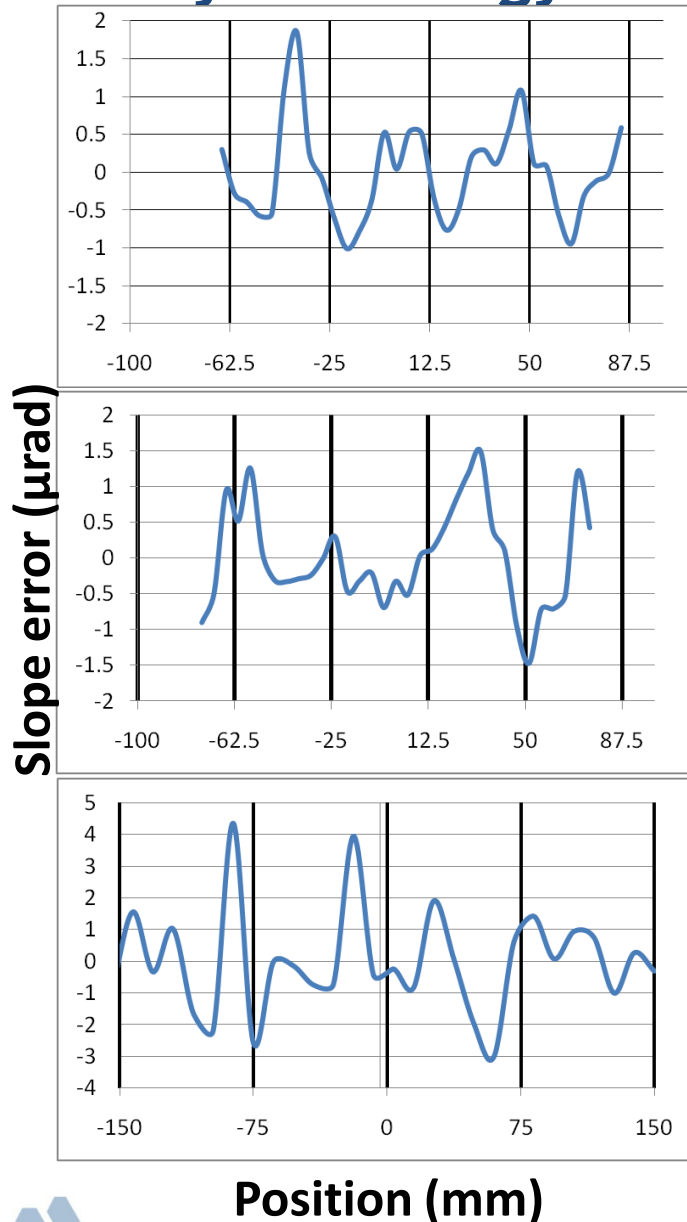


FWHM = 0.81  $\mu\text{m}$

Structure not observed at focus!



# X-ray Metrology when bent near the elastic limit



Focused to 9  $\mu\text{m}$

Slope error = 0.76  $\mu\text{rad}$

Corrected slope error = 0.64  $\mu\text{rad}$

Focused to 12  $\mu\text{m}$

Slope error = 1.21  $\mu\text{rad}$

Corrected slope error = 0.72  $\mu\text{rad}$

Focused to 52  $\mu\text{m}$

Slope error = 1.91  $\mu\text{rad}$

Corrected slope error = 1.68  $\mu\text{rad}$

Position (mm)



# Operational micro-diffraction capabilities

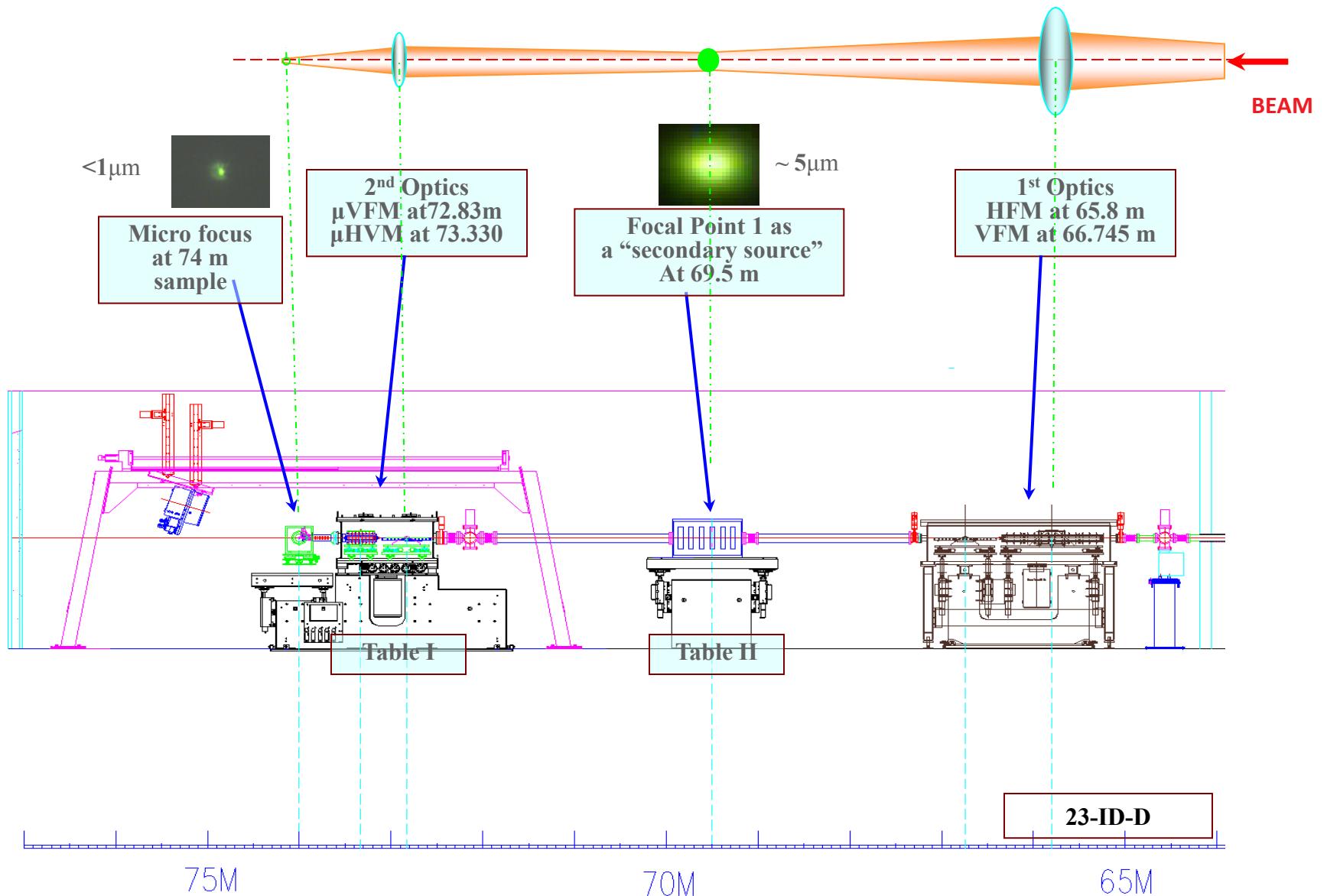
- **GM/CA CAT**
  - 23ID-B,D: 5, 10 , 20 microns
- **Diamond**
  - ID24: 5 microns
- **ESRF**
  - ID-13: 0.3 microns for crystallography (not dedicated)
  - ID23-2: micro-diffraction (7 microns)
- **SPring-8**
  - BL32XU: 1-micron
- **Swiss Light Source**
  - X06SA: micro-diffraction (5 x 25 microns)

Planned/under development

- **NSLS-II: partial funded**
- **Petra-III : under construction**
- **Soleil: under construction**



# GMCA Micro-Focus endstation



# Conclusions

- Bimorph mirrors
  - provide state of the art focusing capabilities
  - clean off focus profiles
  - beware of bending to extreme **AND** looking far downstream of the focus
- Micro-focusing for MX
  - obtaining a 1  $\mu\text{m}$  beam is “easy”
  - beware of high convergence
  - designing the optics to provide 1- 50  $\mu\text{m}$  QUICKLY can be difficult

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## GM/CA Staff

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Richard Benn – mechanical engineering

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Mark Hilgart – software development

Sudhir Pothineni – software development

Michael Becker – mini-beam characterization

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Riccardo Signorato

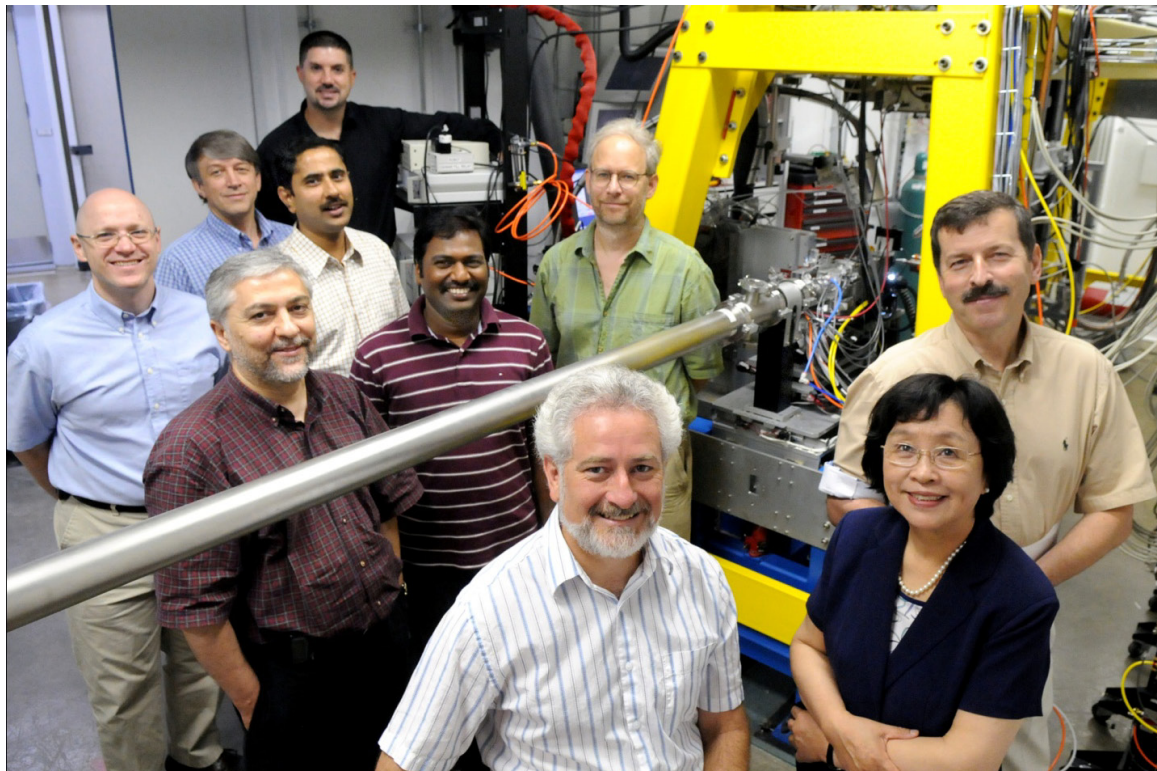
Wolfgang Diete



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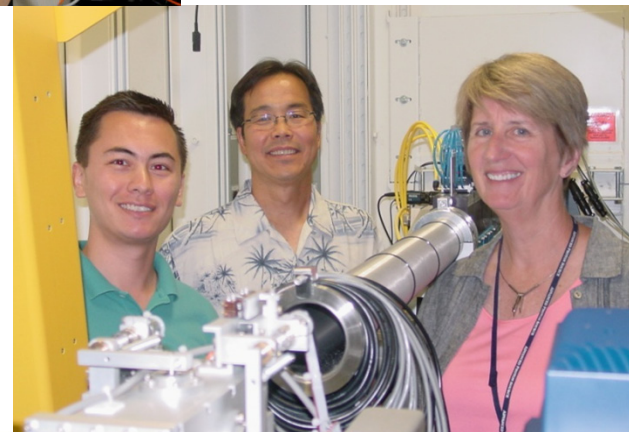


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# Thank you for your attention

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