MX Breakout Session

Bob Fischetti

Workshop on Science Opportunities with an MBA Lattice
Advanced Photon Source

October 21-22, 2013

MX Breakout Session Agenda

1:00 Overview of committee recommendations, and beamline performance - Bob Fischetti
1:20 Mitigating radiation damage - Gerd Rosenbaum
1:40 Sampling handling and alignment techniques - Malcolm Capel
2:00 Ultra-high multiplicity to detect a weak phasing signal - Janet Smith
2:30 How to think about diffraction data from tiny crystals - Steve Harrison
3:00 Town Hall Discussion
5:00 Adjourn

Locations of other sessions

Scanning Probe Imaging Location - 401/A5000
Coherent Diffraction and Phase Contrast Imaging, XPCS Location - 401/E1100
Timing and Dynamics Location - 402 Auditorium
Interface and Single Crystal Diffraction Location - 438/C010
Structural and High Energy Scattering, SAXS Location - 431/C010
Spectroscopy and Inelastic Scattering Location - 401/Lower Gallery
Macromolecular Crystallography Location - 401/A1100

Outline of this talk

- MX Working Group Members
- What does the Brightness increase mean for our beamlines
- Examples
  - Microcrystallogrphy
  - In situ data collection

MX Working Group for APS MBA-lattice Science Case

Internal
Robert Fischetti, APS and GM/CA (Team coleader)
Keith Brister, LS-CAT
Malcolm Capel, NE-CAT
Andrzej Joachimiak, Argonne, SBC-CAT
Kanagalaghatta Rajashankar (Raj), NE-CAT
Stephen Wasserman, LRL-CAT

External
Bill Weis, Stanford University (Team coleader)
Steve Harrison, Harvard University
Janet Smith, University of Michigan
Bi-Cheng Wang, University of Georgia

Goals for the Workshop

- *Inform the APS community* concerning the properties of an MBA low-emittance lattice being considered in the APS Upgrade.
- *Gather input on the new science opportunities* offered by such a source.
- Address how our current suite of beamlines map onto these envisioned science opportunities, and *what new capabilities are needed.*
- *Explore the technical advances in optics, detectors, and undulators* that are required to realize these science opportunities.
- *Identify areas that require R&D efforts* to achieve the ultimate performance from an MBA x-ray source.

Input from the user community and APS staff essential

Workshop Agenda - Day 2

Breakout Discussion and Report Preparation

9:00 - 11:00 *Macromolecular Crystallography (A1100)*

Workshop Reports and Plenary Discussion (402 Auditorium)

11:00 - 11:30 Scanning Probe Imaging - Stefan Vogt/Tonio Buonassisi and Rafael Jaramillo

11:30 - 12:00 Coherent Diffraction and Phase Contrast Imaging, XPCS
Jin Wang/Ian Robinson

12:00 - 13:30 Working Lunch: Q&A Discussions from Breakout Sessions (Lower Gallery)

Workshops Reports and Plenary Discussion (continued in 402 Auditorium)

13:30 - 14:00 Timing and Dynamics - David Keavney/Paul Evans

14:00 - 14:30 Interface and Single Crystal Diffraction - Jon Tischler/Paul Fuoss

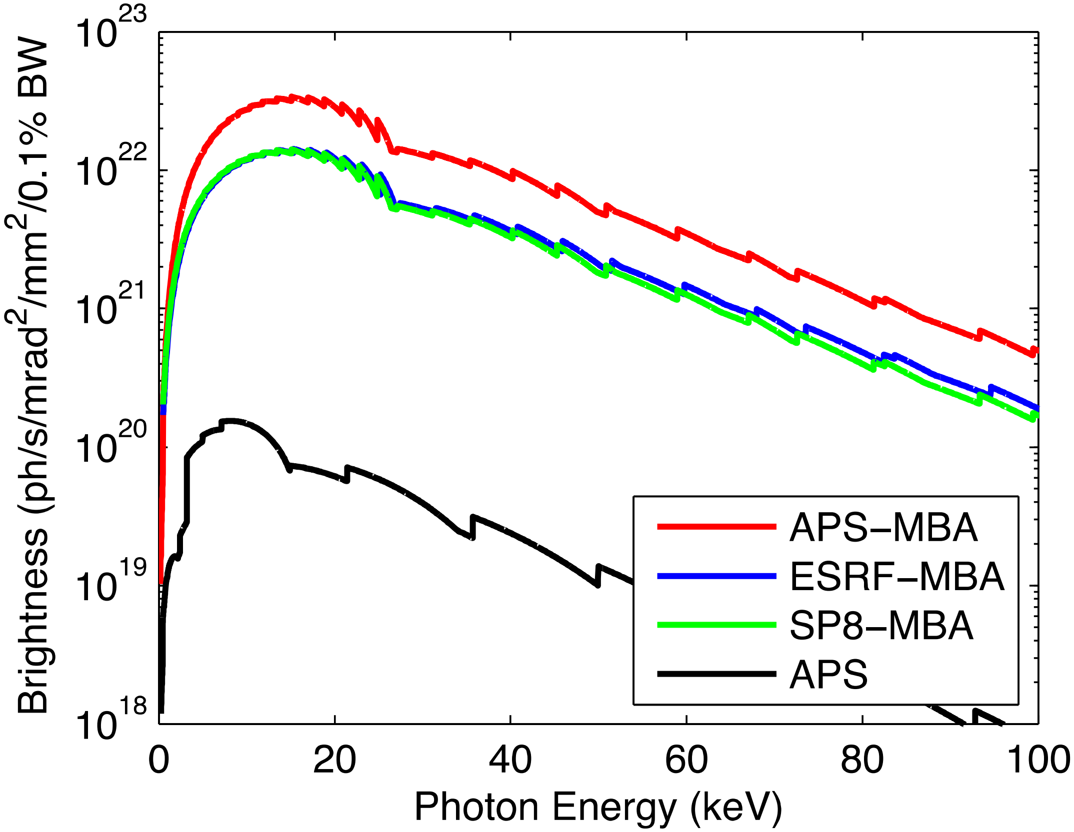
14:30 - 15:00 Structural and High Energy Scattering, SAXS - Jan Ilavsky/Lyle Levine

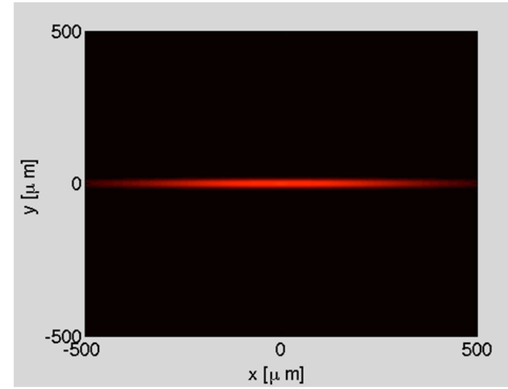
15:00 - 15:30 Spectroscopy and Inelastic Scattering - Steve Heald/Clem Burns

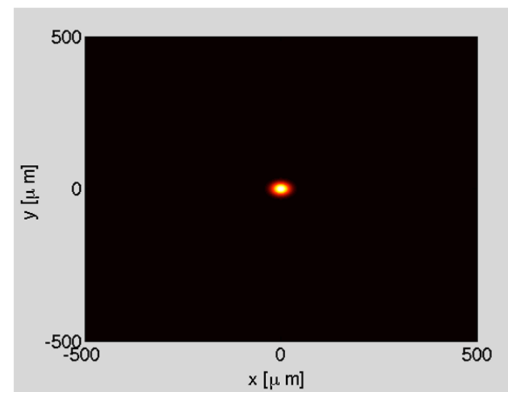
15:30 - 16:00 Macromolecular Crystallography - Robert Fischetti/Bill Weis

16:00 - 16:30 Workshop Closeout Workshop Committee

Potential: World-Leading Brightness



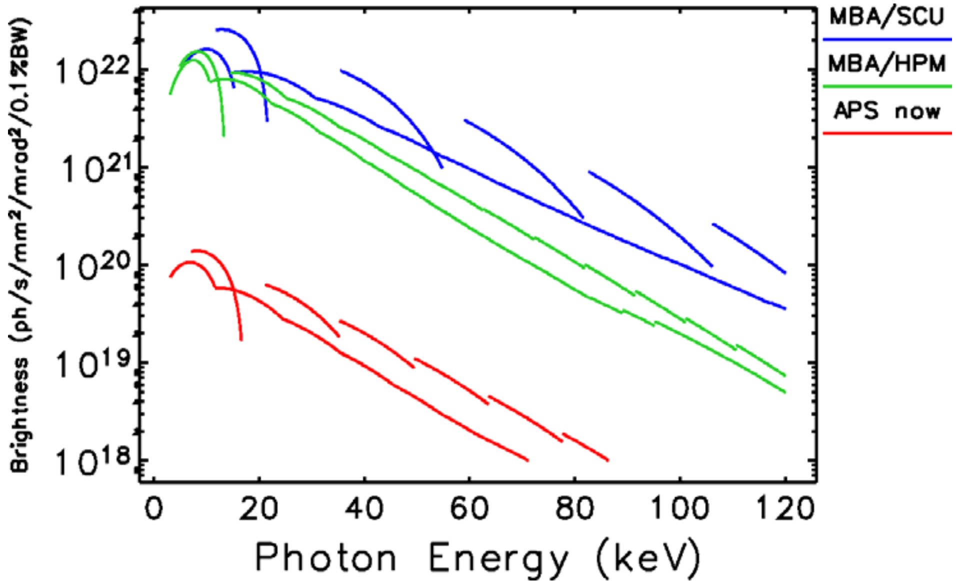


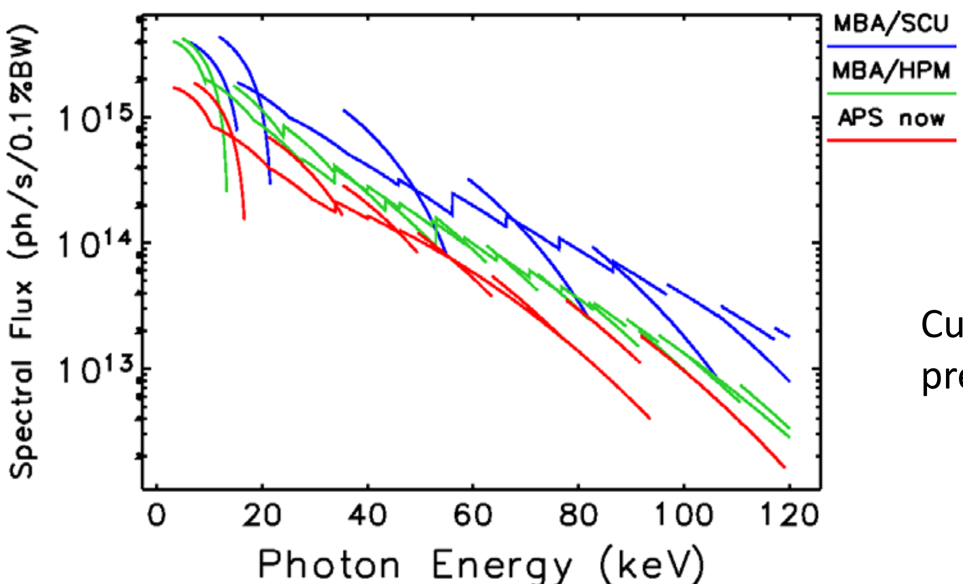


- 6 GeV multi-bend achromat (MBA) storage ring
- Natural emittance of 60-80 pm with 200 mA current
- Small-gap superconducting and conventional undulators
- Enhanced beam stability

Brian Stephenson

Examples of Preliminary Expected Performance





Smaller gaps, optimized periods, higher current, and lower emittance more than compensate for lower beam energy

Brightness increases of 100x or more compare to brightest devices in APS today

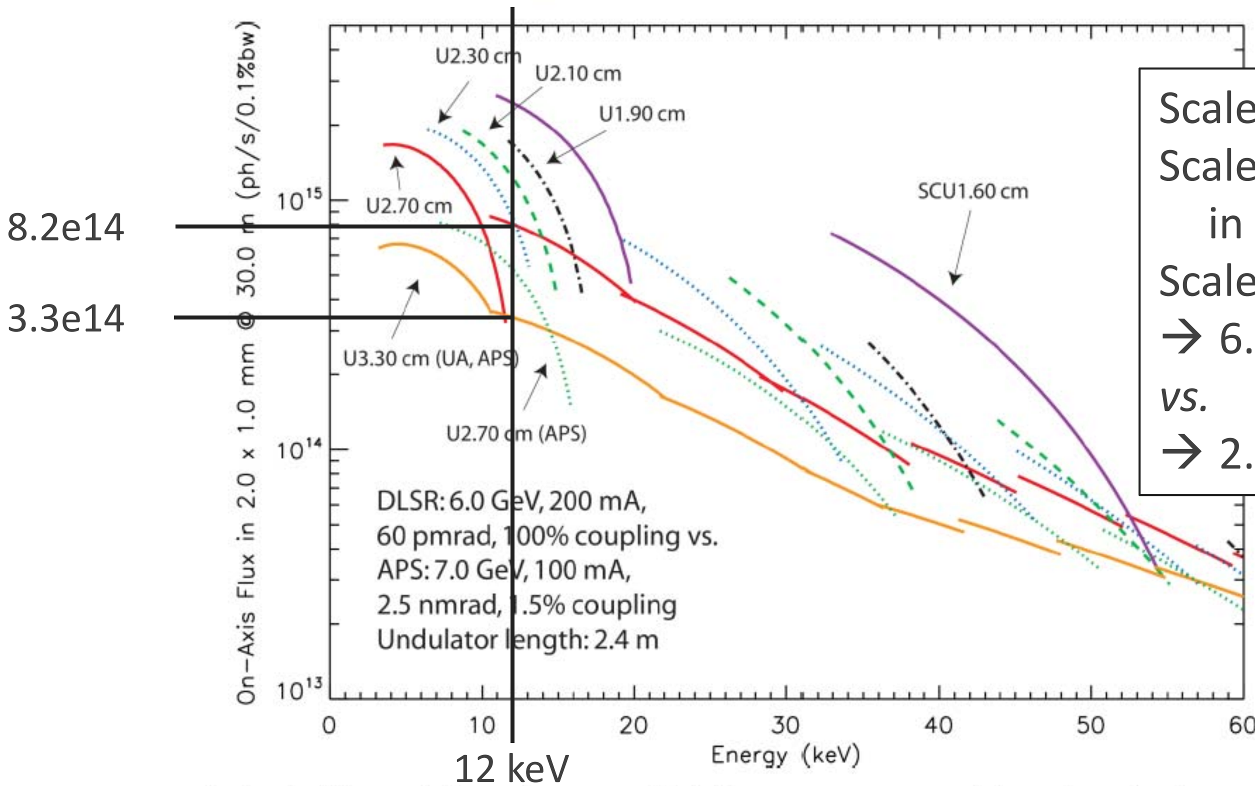
Flux increases a factor of two or more

Curves based on 80-pm lattice, present-day undulators

Brian Stephenson

Beamline Performance

On-Axis Flux Tuning Curves of HPMs and SCUs: 0 - 60 keV



Scale for Si(111) BW
Scale for number of poles in cant und
Scale for losses on BL
→ 6.1e13 ph/sec
vs.
→ 2.0e13 ph/sec

- Calculated odd harmonic flux tuning curves of hybrid permanent magnet undulators (HPMs) and one superconducting undulator (SCU) for today's APS lattice and the proposed DLSR lattice. The magnetic length is 2.4 m for all devices. The minimum gap is 8.5 mm for the DLSR (11.0 mm APS).
- Reductions due to magnetic field error were applied to all undulators (estimated from one measured undulator A at the APS).
- The flux gain for the DLSR undulators is in range 2 – 3x. (A factor of 2 comes from the higher operating current of the DLSR).

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Time to the Garman Limit (cryo-cooled)

Garman limit[1] ~ 3.0 x 10^7 Gray (35% intensity loss)
Deposited energy in sample – not incident energy!

E ~ 12.68 keV

Beamline	Divergence (µrad, FWHM)	Smallest beam width (µm)	Smallest beam height (µm)	Flux (ph/sec)	Flux density (ph/s/µm2)	Dose rate (Gy/s)	Time to Garman limit (msec)
APS-U MBA 23-ID-D	3200 x 1200	0.40	0.50	5.3E+13	3.4E+14	1.0E+11	0.29
NSLS-II FMX‡	1700 x 700	1.00	0.50	5.0E+12	1.3E+13	3.9E+09	7.60
DLS VMX¥		0.50	0.50	1.0E+12	5.1E+12	1.0E+09	29.90
APS-U MBA 23-ID-D	270 x 180	6.10	5.20	6.1E+13	2.4E+12	7.6E+08	39.52
Petra3 MX2	500 x 300	4.00	1.00	5.0E+12	1.6E+12	4.9E+08	60.81
SPring8 BL32XU§	1520 x 980	0.90	0.90	6.2E+10	9.7E+10	3.0E+07	992.99
Petra3 MX1	200 x 150	28.00	13.00	1.0E+13	3.5E+10	1.1E+07	2766.63
APS 23-ID-D*	400 x 100	5.00	5.00	5.4E+11	2.8E+10	8.5E+06	3518.81
DLS I24	2000 x 50	8.00	8.00	1.0E+12	2.0E+10	6.2E+06	4864.40
ESRF ID23-2†	550 x 360	7.50	7.50	4.0E+11	9.1E+09	2.8E+06	10688.38
APS 23-ID-D*	400 x 100	70.00	25.00	2.00E+13	1.46E+10	4.50E+06	6650.55

*APS 23-ID intensities are for 12.0 keV except where noted
§SPring8 BL32XU intensities area at 12.398 keV
†ESRF Upgrade may have changed these numbers
‡NSLS-II AMX/FMX intensities are at 12.7 keV

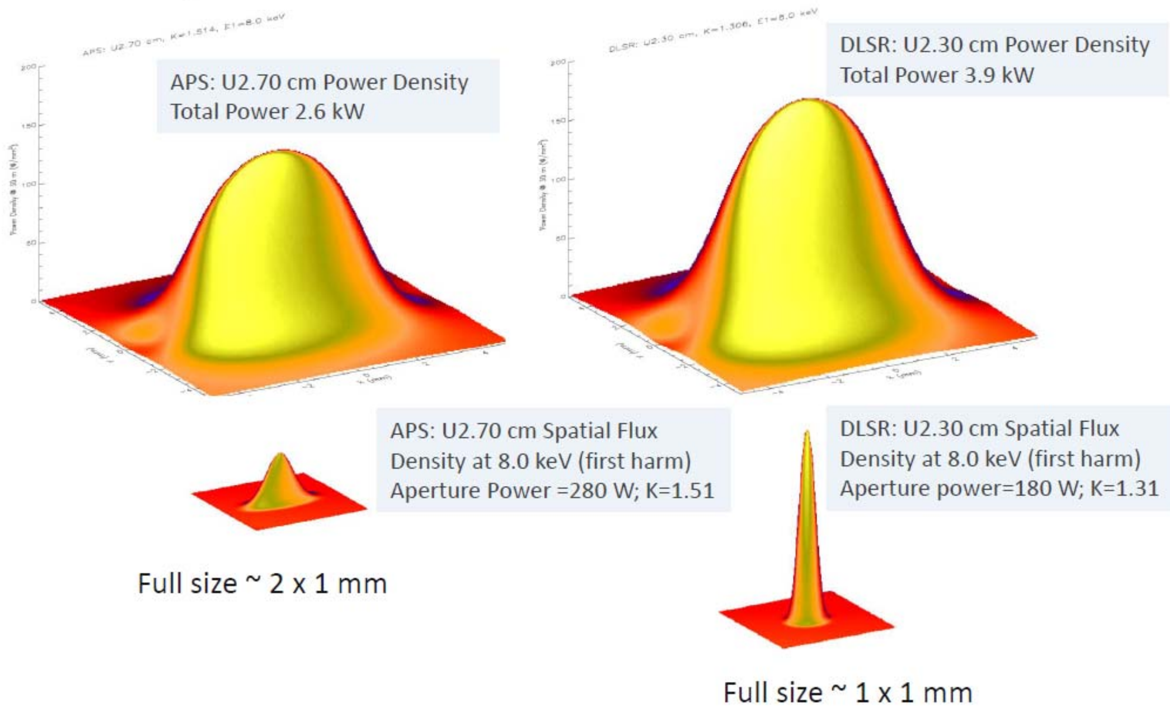
[1] Owen, R.L., Rudino-Pinera, E. & Garman, E.F. *Proc Natl Acad Sci U S A* **103**, 4912-7 (2006)
[2] RADDOSE http://biop.ox.ac.uk/www/garman/lab_tools.html

Beamline Performance

Power α E^2

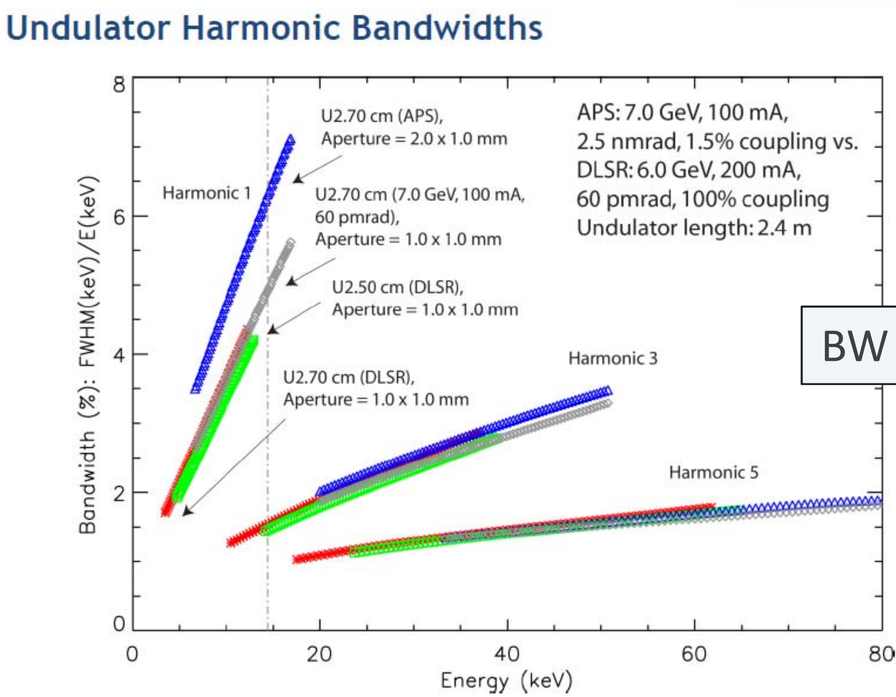
Power Desnity α E^4

Power Density and Flux in the Central Cone at 8.0 keV at 30 m



- Important: Notice that the size of the power distribution is essentially unchanged whereas the size of the central cone is reduced by a factor of ~ 2 in the horizontal plane (lower order odd harmonics).

Beamline Performance



- The first bandwidth becomes smaller. For example at 8 keV it is smaller by ~ 25% (red vs. blue markers)

BW of 1st harmonic decreases ~25%

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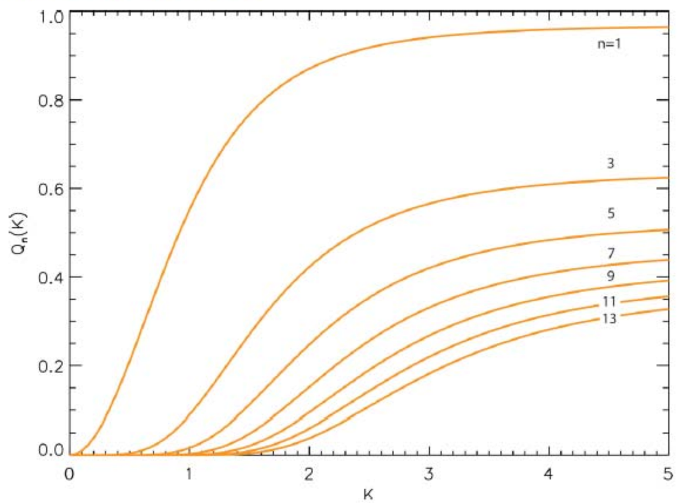
Beamline Performance

Flux in the Central Cone



The flux in the central cone with harmonic number n is

$$F^n \propto N Q_n(K) I$$

where *N* is the number of undulator periods and *I* is the ring current.
The coherent flux is directly related to the brightness

$$F^n_{coh} = F^n x\, fcoh = F^n x \frac{(\lambda/2)^2}{4\pi^2 \Sigma_x \Sigma_{x'} \Sigma_y \Sigma_{y'}} = B^n x (\lambda/2)^2$$

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Coherent fraction at 12 keV

Currently ~ 0.001

MBA ~ 0.1

Speckle???

MX Science Case for APS-U MBA Lattice

Higher flux density and microcrystallography

- More challenging samples – smaller, less homogenous
- How small can we go – 500 nm on edge?

Higher flux density → radiation damage occurs faster

- Reach Garman limit in 0.3-100 msec (cryo-cooled)
- Can one outrun 2^{nd} order radiation damage at RT?
- RT *in-situ* screening
- Multi-crystal data sets
- High multiplicity to overcome "noise" of multi-crystal

High flux density and X-ray Diffraction Near Edge Spectroscopy

- Improved S/N

Large crystals

- Utilize unfocused or partially focused beam for maximum stability

How to deal with partials

- Increased bandwidth (pink beam)
- Increased convergence

Need new sample handling/delivery methods

- Acoustic drop ejection – on grids, tape, capture with laser tweezers
- Slow LCLS type ejector

High speed (frame rate and "count rate"), high sensitivity detectors

- Photon counters vs. charge integrators?

Complementarity of Synchrotron vs. XFEL MX in the future

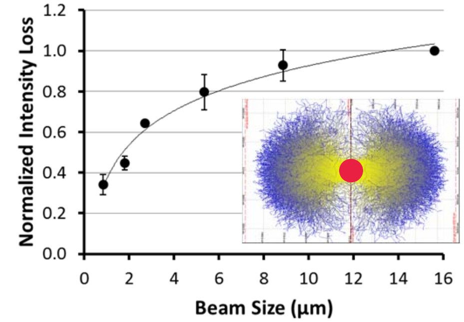
Microcrystallography of Biological Macromolecules

Microcrystallography has enabled the determination of high impact, 3D structures of proteins & protein complexes

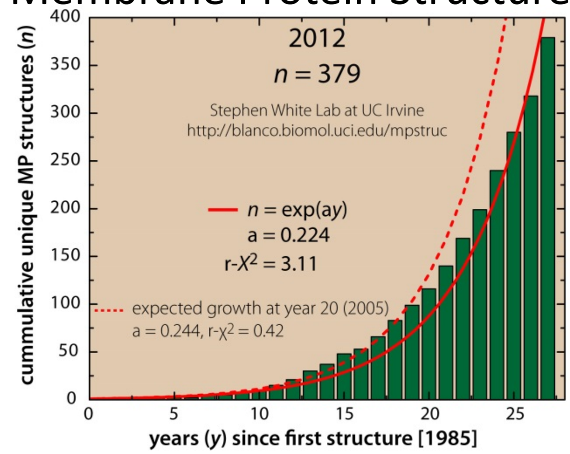
APS-U & MBA-lattice will significantly expand the MX horizon

- Exploit the high Brightness
  - Nano/Microcrystals – currently inaccessible
  - Improved S/N and resolution for small (0.5 – 5 µm), inhomogenous and/or weakly scattering crystals
- Exploit APS high energy source
  - Reduced radiation damage – photoelectron escape
- Enhanced modes of data collection
  - *In situ*, Rastering, Helical and Shutterless data collection

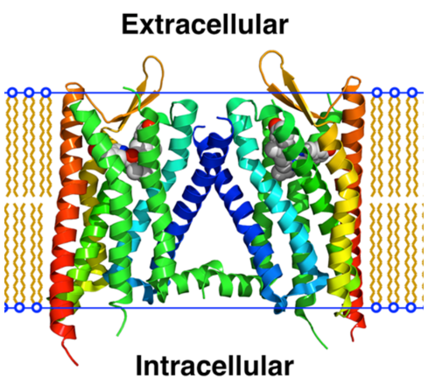
Reduced Radiation Damage

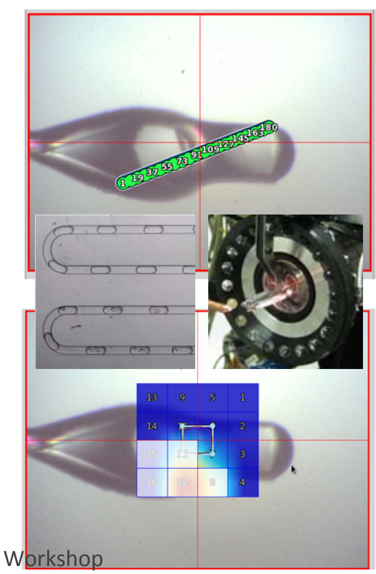


Membrane Protein Structures

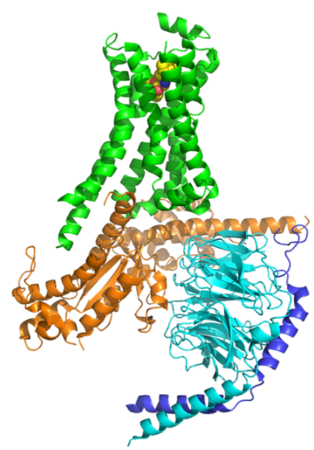


k-opioid GPCR
Ray Steven's lab

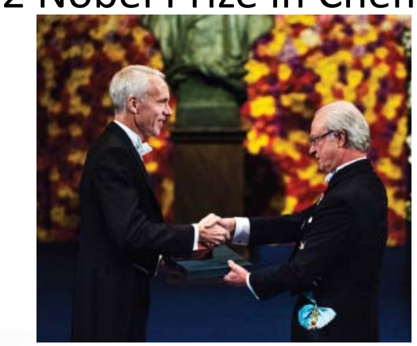




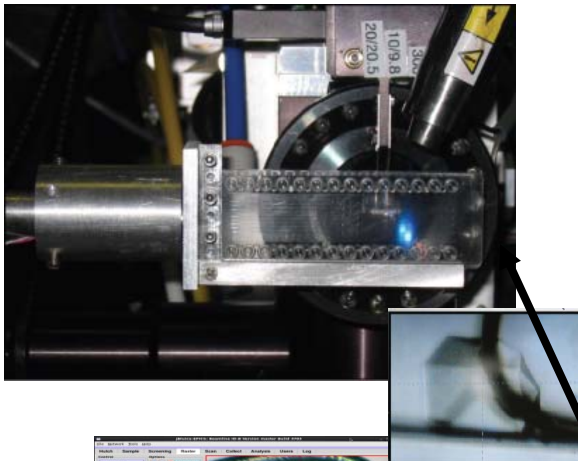
β_2 adrenergic receptor-Gs protein complex
Kobilka & Weis labs



2012 Nobel Prize in Chemistry



In Situ Room Temperature Data Collection



Blurring the line between synchrotron data collection and serial femtosecond crystallography. Higher brightness and faster detectors employed in the search for every shrinking crystals of increasing complexity and biological importance.

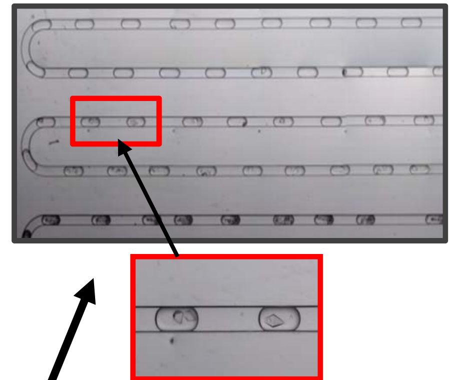
- In Situ screening will provide important diffraction feedback on limited quantities of biological material at an early stage in crystallization trials.
- Samples introduced by novel delivery systems (e.g. acoustic drop)
- Data collection on samples with large amounts of small crystals complexed to a variety of compounds.
- Data collection on high symmetry space groups (e.g. viruses)
- Development of data collection tools extending to the µm level

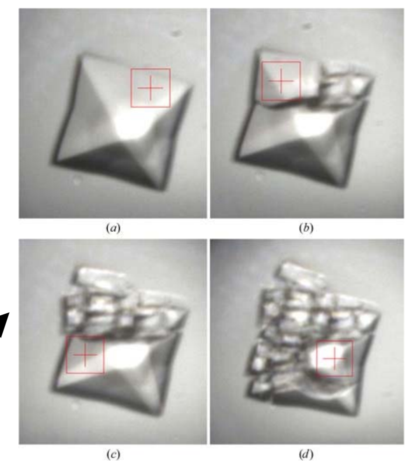
Sample

Raster

Vector

Mini- to Micro-beam tools

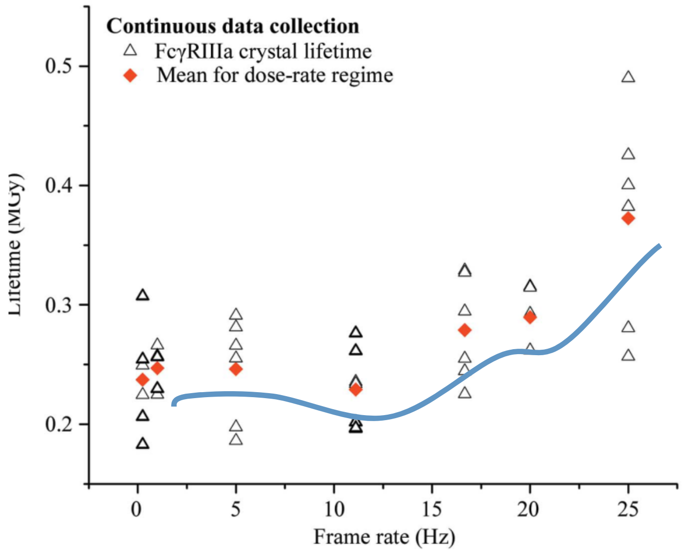


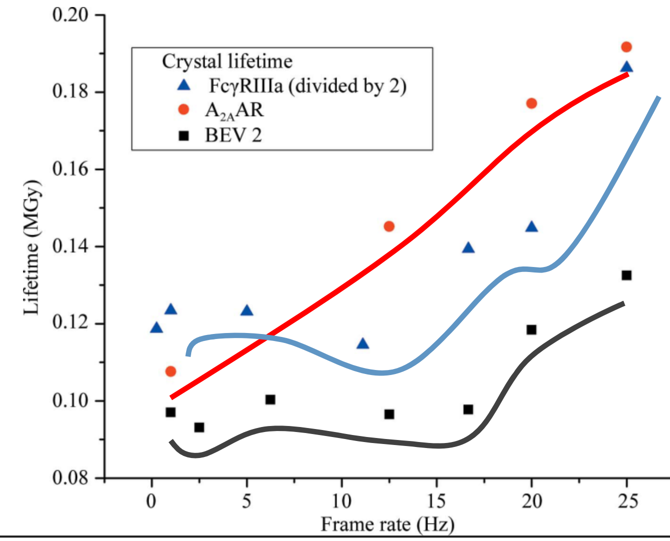


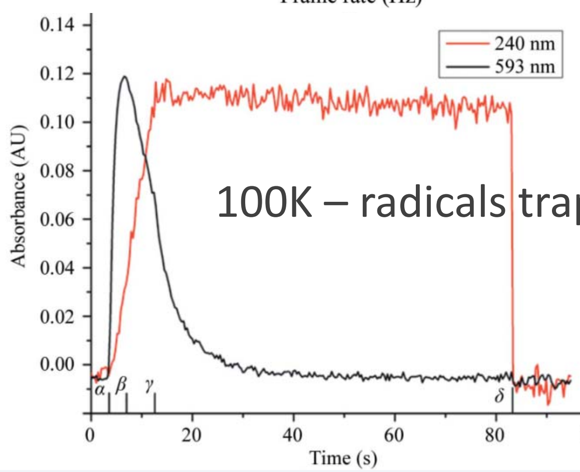
In situ data collection from a virus crystal at 3 different Positions.
D. Axford et. al., Acta Cryst. (2012) D68, 592-600

Craig Ogata

Outrunning 2nd Rad Dam at Room Temp – Beware!







100K – radicals trapped

Crystal lifetime
Start-stop
40 msec exp; 4 sec dead time → 0.186 Mgy
Shutterless
>90 msec exp → 0.257 MGy
40 msec exp → 0.373 Mgy

Compare cryo-cooled → 30 MGy

Black – generation of aqueous or solvated e-, followed by 1st order decay
Red – postulated to be hydroxyl radical

Owen *et. al.* Acta Cryst. (2012) D**68**, 810-818

Challenges to fully realize the benefits

1) Installation of **focusing mirrors** with the requisite surface smoothness (sub Angstrom) and surface slope errors (sub-microradian).
2) Development of **advanced sample handling** and visualization techniques for micron sized crystals.
3) Installation of low noise, high sensitivity 2D detectors with millisecond read out times (likely pixel array **detectors**).
4) Improvements in beam line beam position sensing and **beam steering** required to insure uniform illumination of microscopic samples throughout data collection.
5) Improved precision and reproducibility of **sample goniometry**, including sample alignment stages with sub-micron accuracy.
6) Efforts to **reduce noise** in diffraction data in general (e.g. He flight paths for low energy and/or long distance data collection);.
7) Development of automated methods for **multi-crystal merging** of partial data sets.
8) **Sample positional stability** under interactions with **cold streams** will be an issue. Current nitrogen cryo-gas streams have the potential for degrading positional stability of small crystals, through aerodynamic interactions. New methods for supporting and positioning samples may be required by the extremely small x-ray beams made possible by the MBA lattice upgrade.

MX Workshop Report

New scientific opportunities with MBA Lattice

- Micro-beam & Micro-crystal Crystallography
- *In situ* room-temperature diffraction
- Pink-beam crystallography
- Out-running secondary radiation damage
- Multiple Crystal data collection
- Large Homogenous Crystal
- X-ay Diffraction Near Edge Spectroscopy

Enabling Technologies and R&D

- New sample delivery/Handling Visualization methods
- Better integration of crystal growth with diffraction experiments
- Improved methods for processing multi-crystal data sets
- Methods for processing pink-beam data
- Improved stability of sample and endstation components
- Detectors
- Complementarity of XFEL and 4th Gen Synchrotron

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Beamline Performance

Discussions/Summary

- The issue of undulator phasing becomes important for the DLSR lattice. Remedies:
  - make one 4.8-m-long undulator instead of 2 x 2.4 m, however,
  - long undulators and a smaller minimum gap (8.5 mm instead of 10.5 mm) increase the attractive magnetic forces, but
  - we may make the magnets narrower in the horizontal direction (good field region 3 mm instead of 5 mm), and
  - use undulator with shorter periods (< 2.8 cm), so forces are expected to be less than for the Undulator A.
- Undulator gap precision is typically about 10 μm for the Undulator A and better for shorter period lengths (~ 5 μm). It may need to be set tighter for certain applications for the DLSR due to a smaller bandwidth.
- The power and on-axis power density for the DLSR undulators are typically higher by 30 – 50%. The power scales with E^2 and the power density with E^4 but we are operating at smaller gap with larger K value. For example, the Undulator A (3.3 cm) at 10.5 mm gap generates 6 kW power and 170 kW/mrad^2 power density and the U2.7 cm at 8.5 mm gap on the DLSR generates 9 kW power and 225 kW/mrad^2.
- Radiation damage may become increasingly important because of the smaller minimum gap. Need tighter beam-loss control and to consider SmCo magnet material, which has higher radiation resistance than NdFeB. (The tuning ranges would become somewhat smaller). All results presented here used NdFeB magnets.