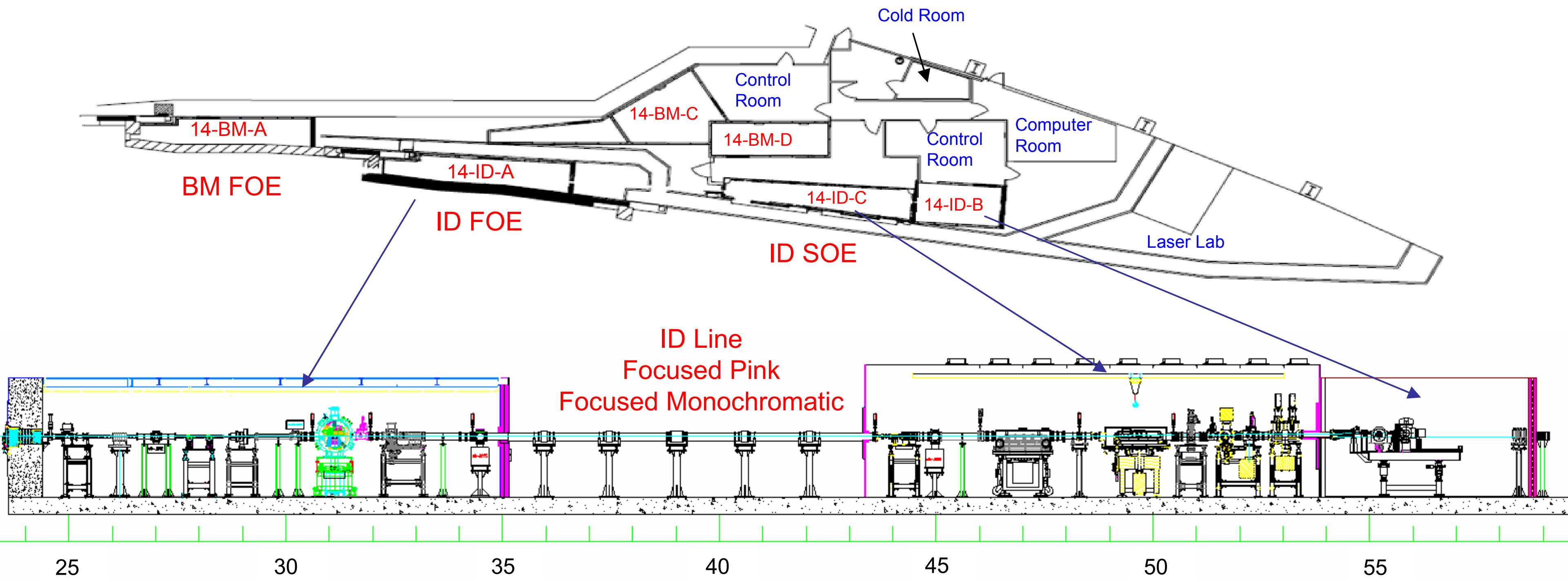
14-ID Beamline Upgrade

T. Graber
June 24, 2008



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April 18, 2008

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BioCARS Supported Techniques

- Time-resolved Laue Crystallography
- Time-resolved WAXS
- Monochromatic MAD Capabilities
- All of the above can be performed under Biosafety Level 3 (BSL3) conditions

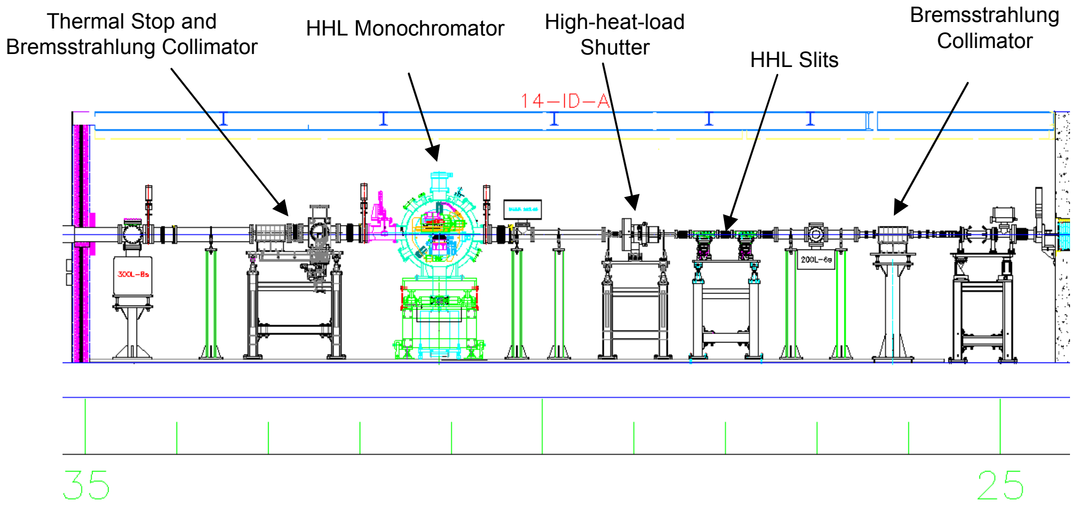
Upgrade Goals

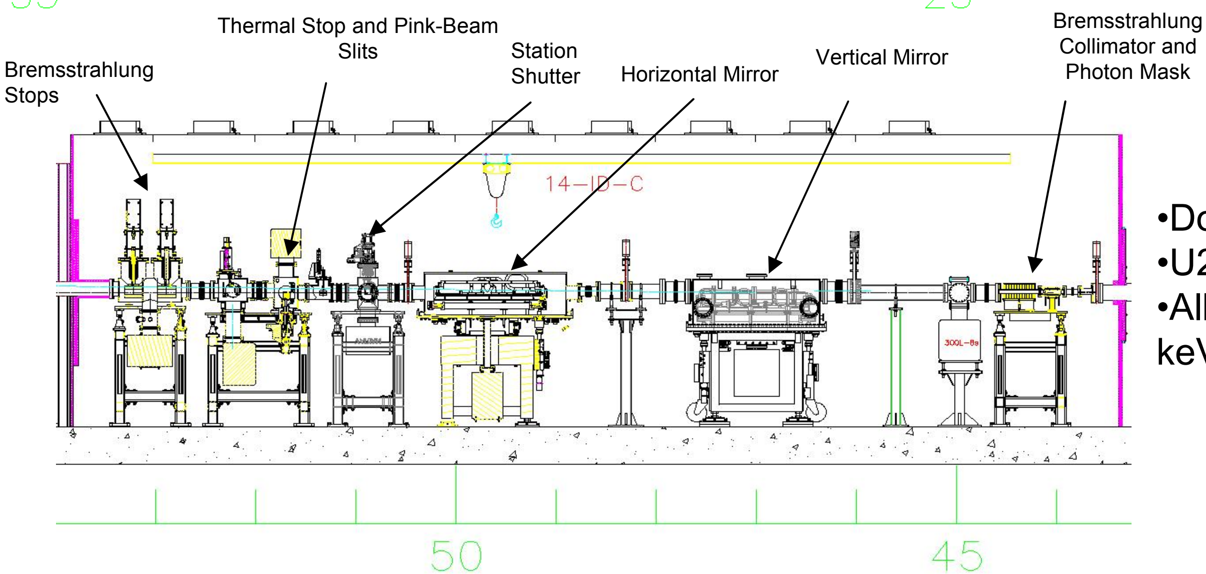
- Maximize x-ray flux density at sample (single shot pump-probe measurements?)
- Improve reliability and flexibility of the beamline
- Achieve ~100 picosecond time resolution
- Improve user throughput and grow community by operating in 24-bunch mode

New Capabilities Realized after the Upgrade

- Doubly focused pink and monochromatic beam with a demagnification of 5:1 vertical and 8:1 horizontal
- Flux density at sample increased by a factor of ~100 (number of pump-probe shots per image reduced from ~80 to ~4)
- Thermal management with a water-cooled white-beam chopper proves reliable
- Single-bunch isolation in 24-bunch mode increases the available beam time for time-resolved crystallography from 1 to 2 weeks per 12-week run to 9-10 weeks
- New broadly tunable picosecond laser system makes 100 picosecond time resolution possible

14-ID Optics Layout





- Double Undulator optimized at 12 keV
- U27 and U23
- Allows continuous coverage from 7-18 keV in the First Harmonic

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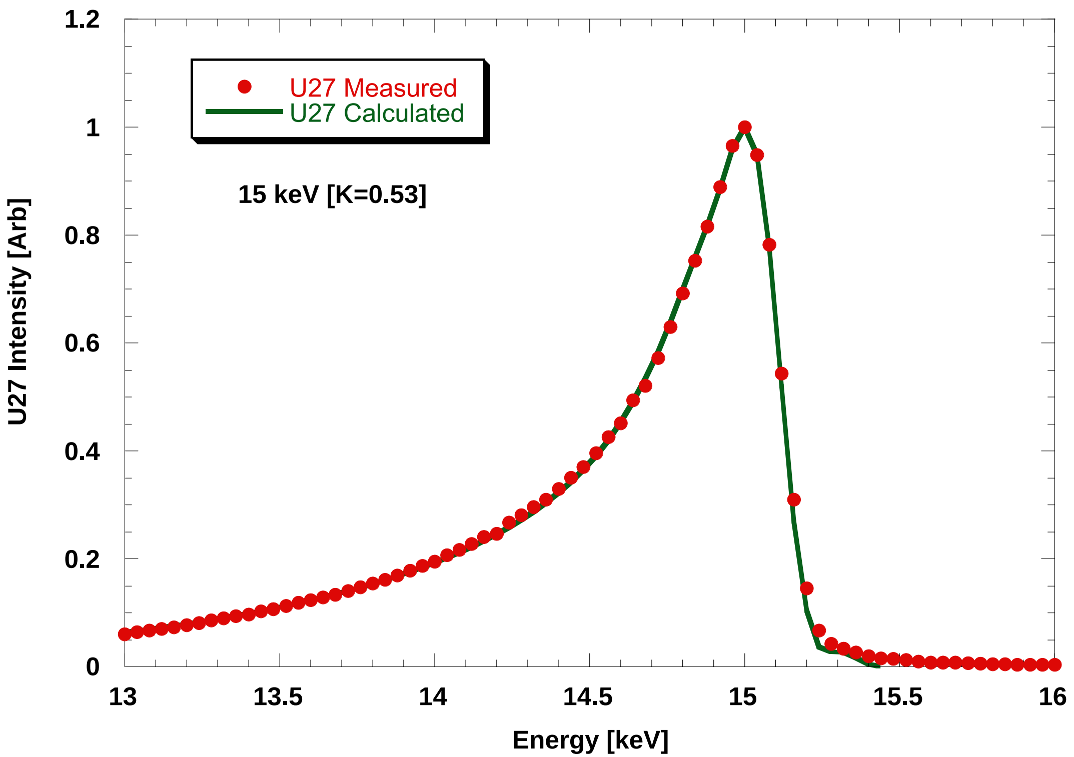
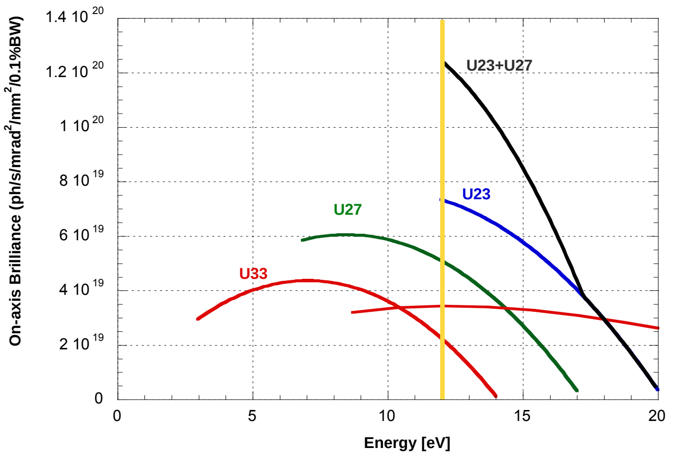
Undulators U27 & U23

U27 Measured and Calculated





Measured monochromator energy scan over first harmonic with the undulator tuned to 15 keV (red circles). XOP Calculation of first harmonic peak (solid green curve).

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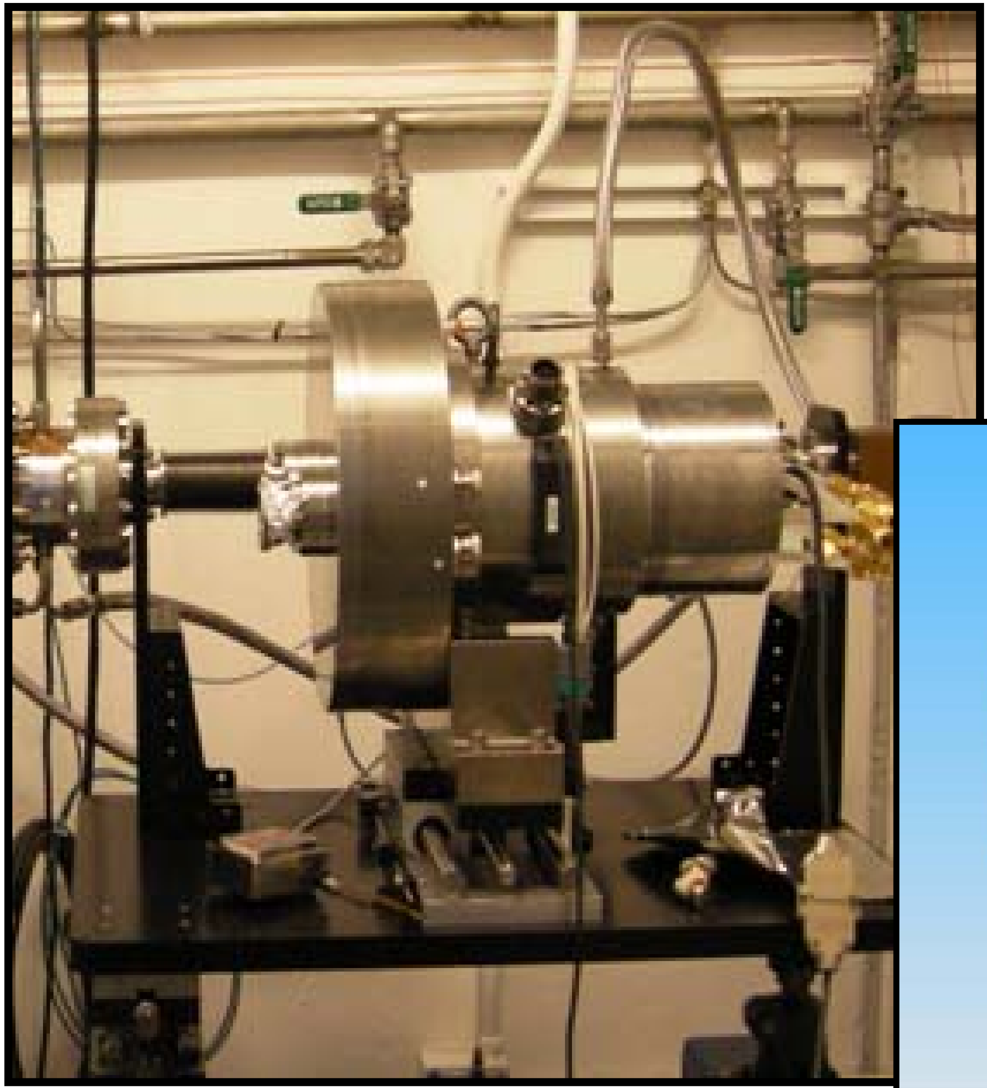
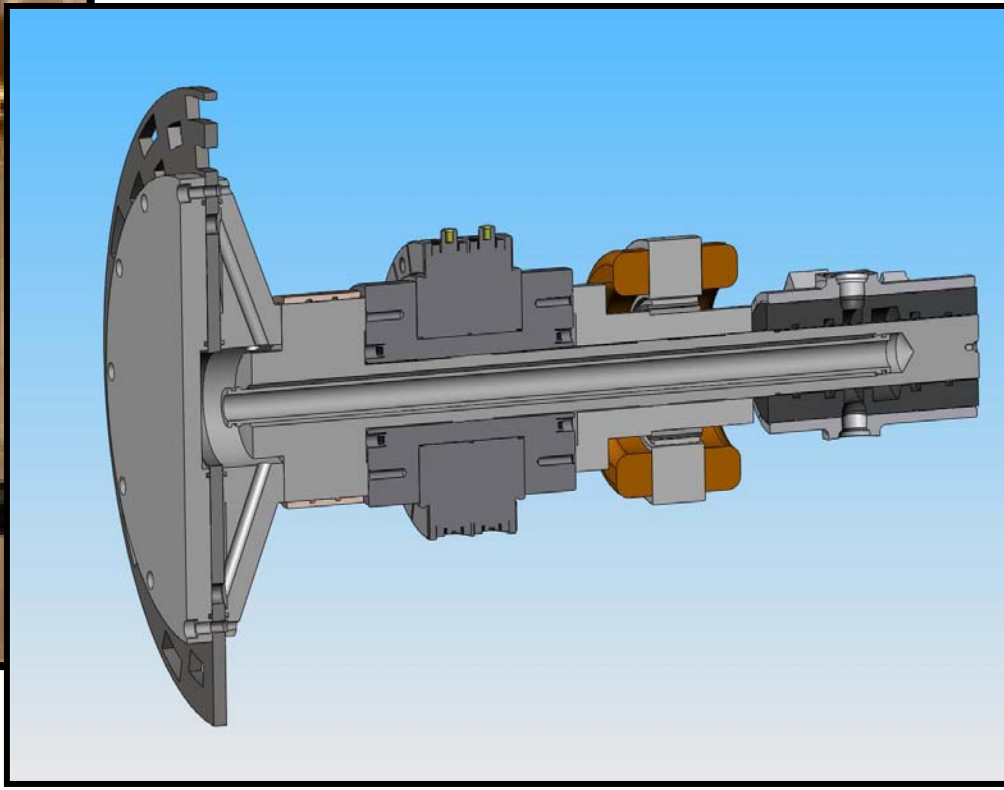
White-beam Chopper

- •Air Bearing
- •Water Cooled
- •Vacuum Isolated
- •Reduces downstream heat load





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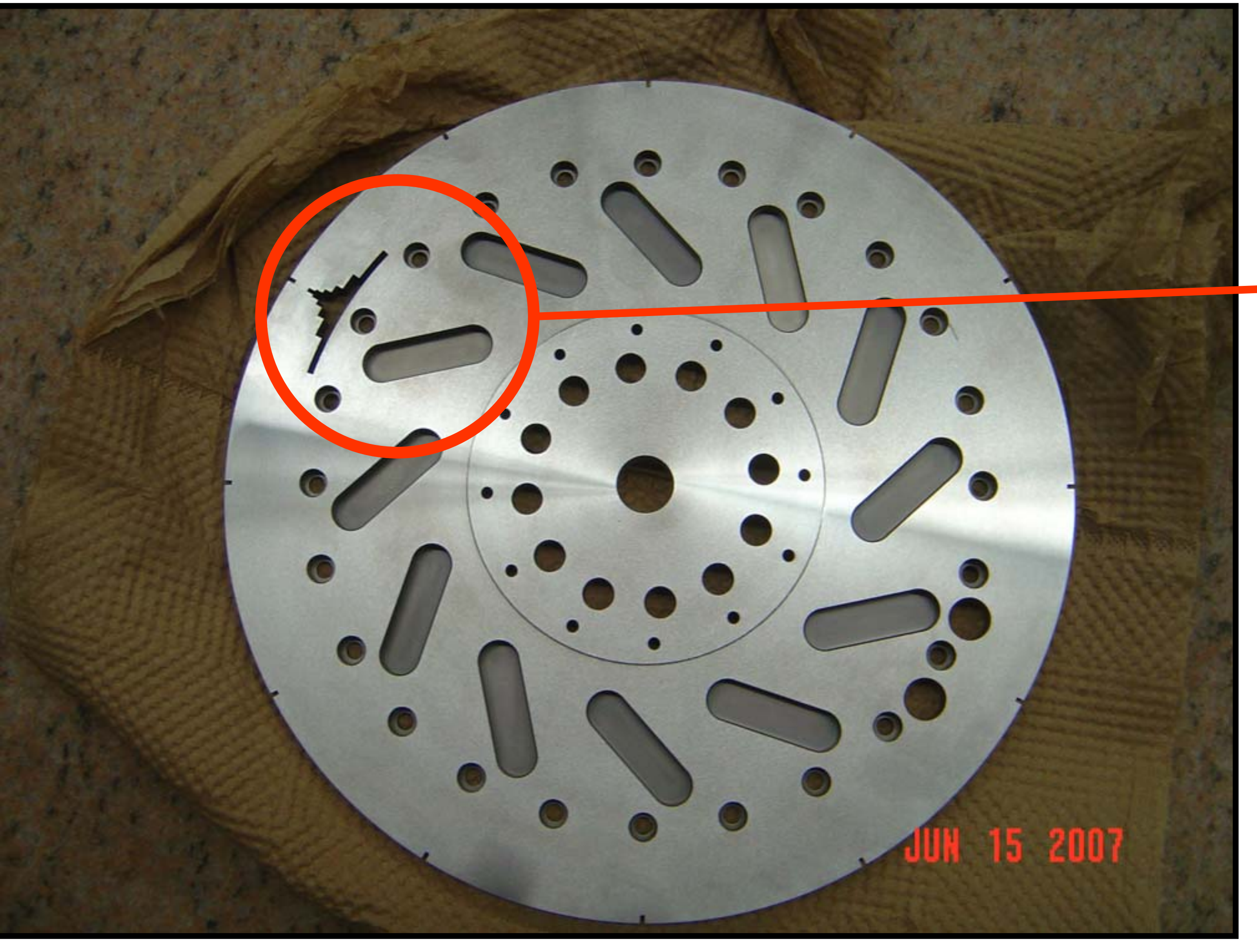
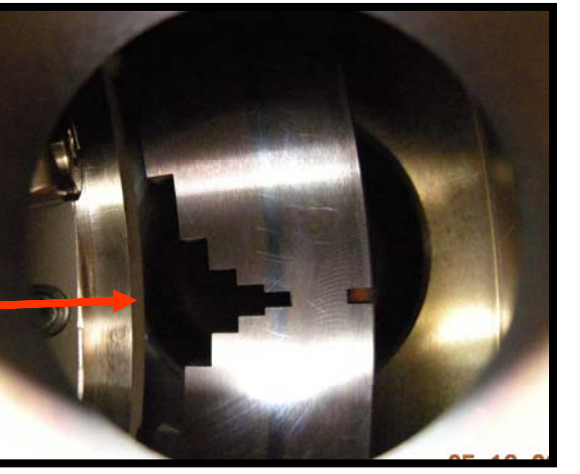
Professional Instruments Company
pico@airbearings.com

Rotor and Slot Arrangement





- Nominal frequency 4980 rpm
- 83 Hz or 1kHz operation
- Temperature rise 16^{o}C

Slot Width [mm]	Open Time [us]
48	789 (6.5%)
24	386 (3.3%)
12	189 (1.6 %)
6	92 (0.8%)
3	45 (0.4 %)
1.5	22 (0.2 %)

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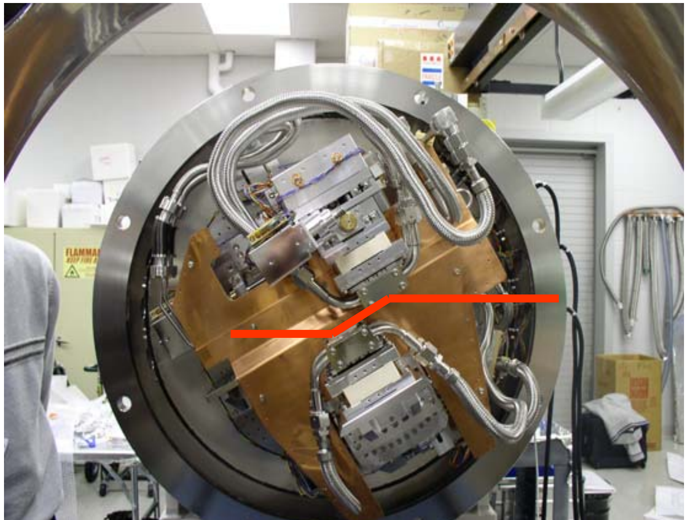
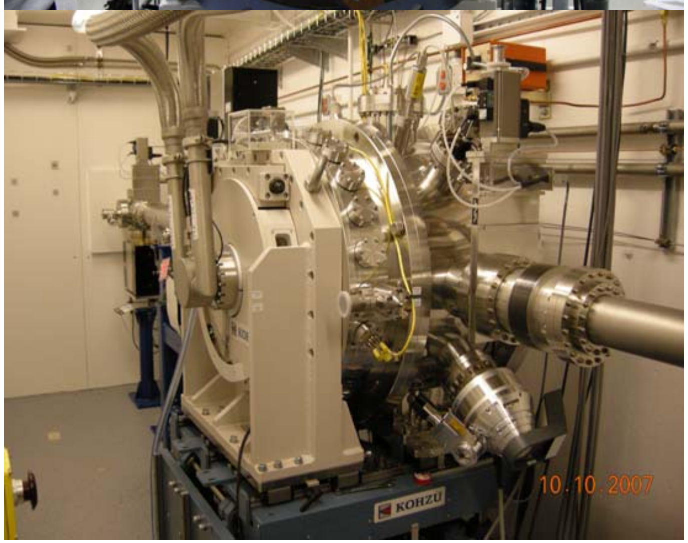
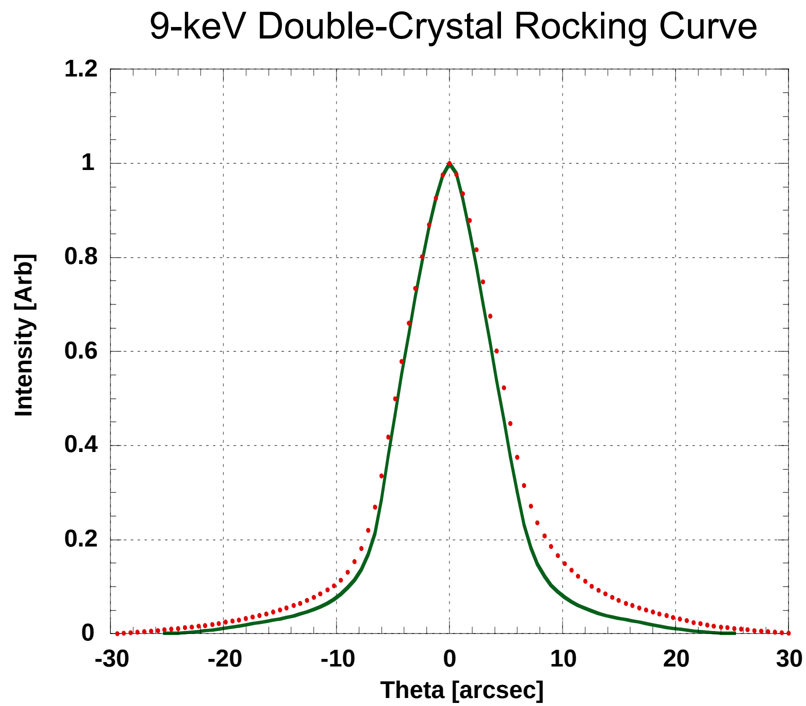
Kohzu High-Heat-Load Monochromator





- Cryogenically cooled Si [111]
- Energy range: 7-20 keV
- Energy Resolution: $\Delta E/E \sim 1.7 \times 10^{-4}$
- 9.8” Measured RC Width (9.0” theoretical)



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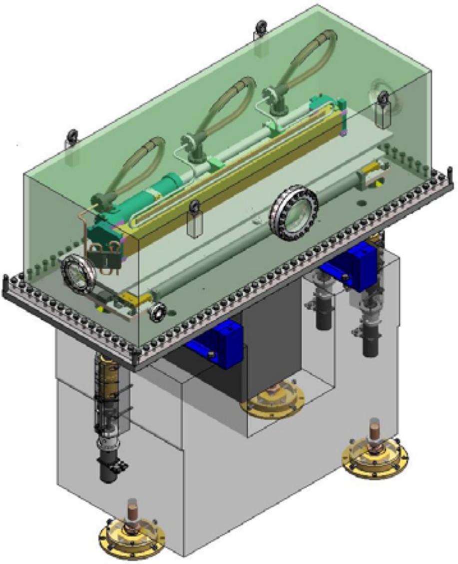
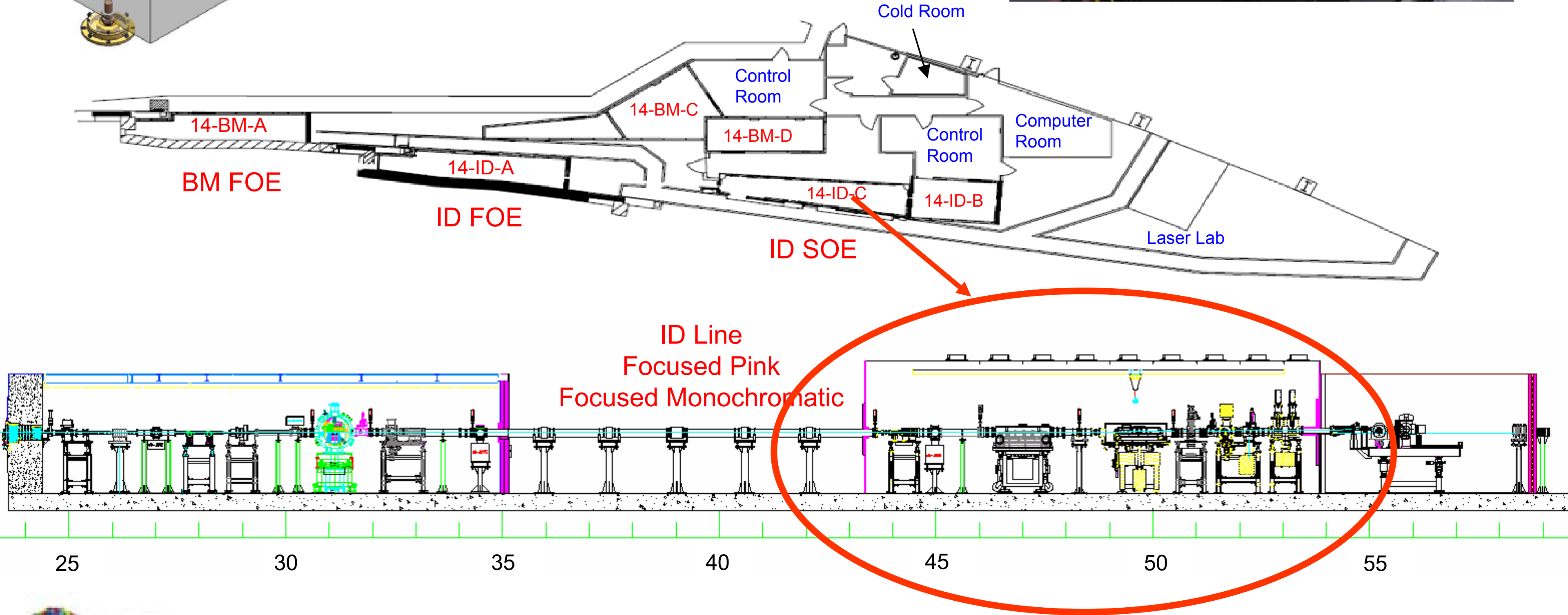


KB Mirror System



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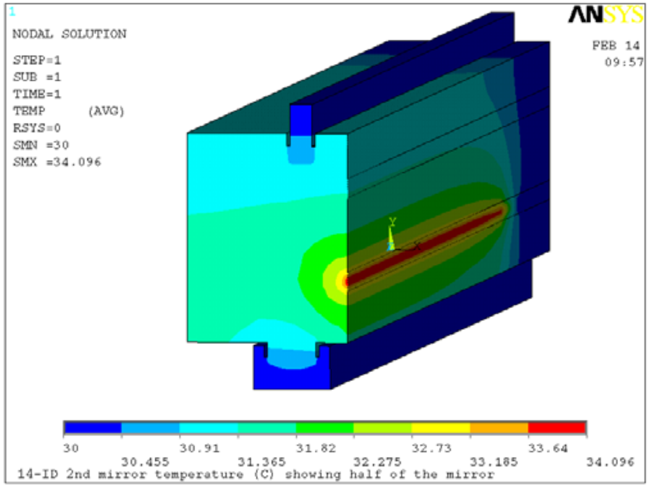
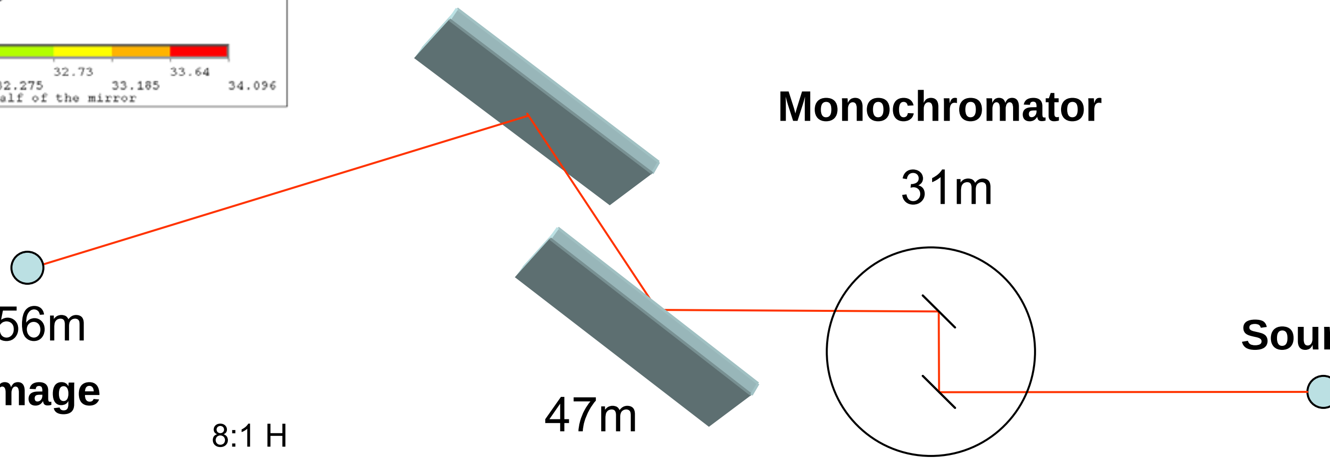
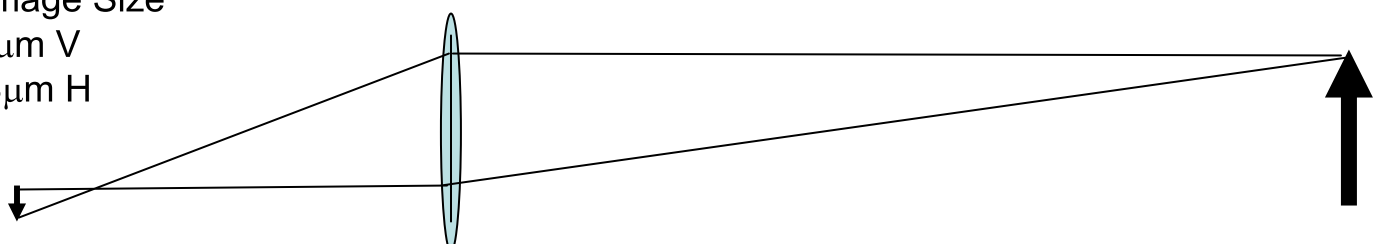
Oxford-Danfysik /SESO KB Mirror System



Stripes

- Rhodium
- Platinum
- Silicon





0.9 μrad RMS slope error blurs image to:

38.3 μm Vertical

82.2 μm Horizontal

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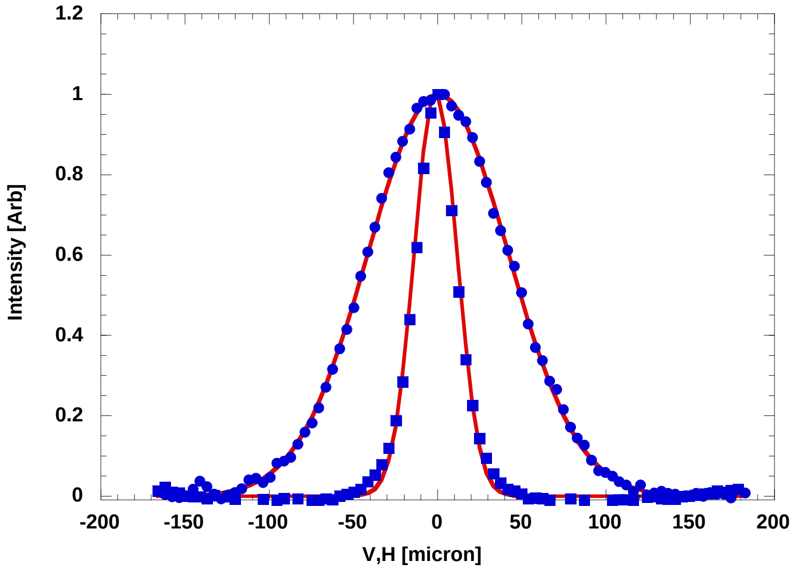
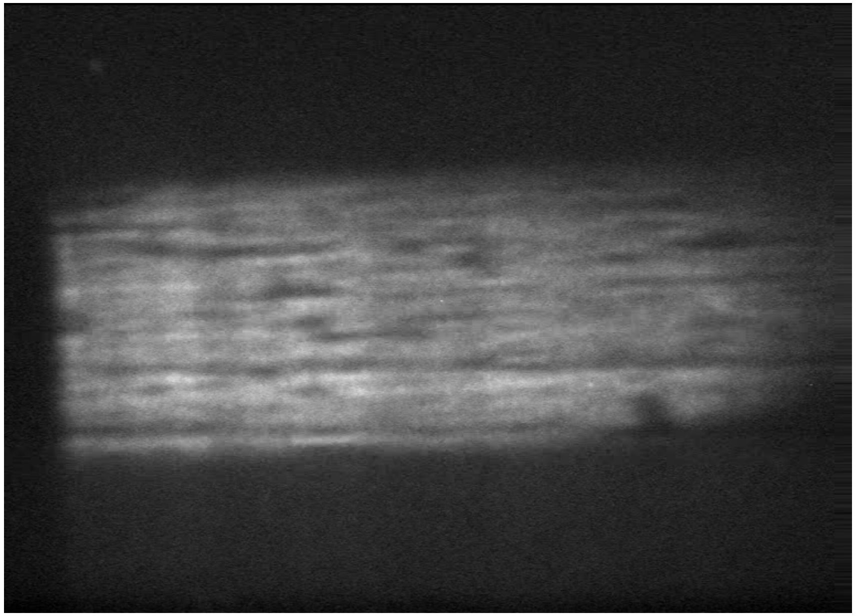
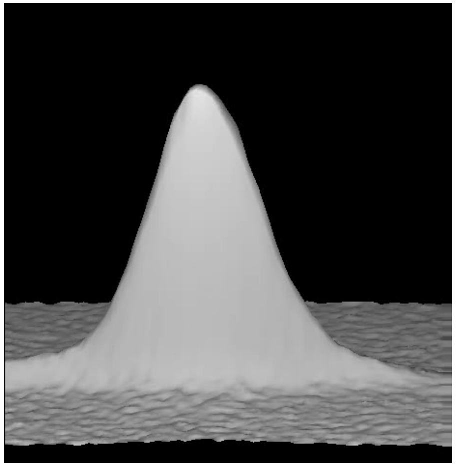
Monochromatic Mirror Performance







Horizontal Beam Size: 95μm FWHM

Vertical Beam Size: ~20μm FWHM

- 50% of total flux measured through a 65μm x 40 μm slit

- Theoretical value for a 2D Gaussian with this slit size is 51%

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Theoretical and Measured Beamline Parameters at 12 KeV

Parameter	Theoretical	Measured
White beam power U27 (K=0.92, measured at exit window)	150 W	142 W
White beam power U23 (K=1.16, measured at exit window)	329 W	329 W
Total power focused pink beam (U23+U27 measured at sample)	125 W	103 W
Number of photons in a single bunch in 24 bunch mode.	$1.0 \times 10^{10}\ \gamma$	$8.4 \times 10^{9}\ \gamma$
Number of photons in a single bunch in hybrid mode.	$3.9 \times 10^{10}\ \gamma$	$3.2 \times 10^{10}\ \gamma$
Pink beam mirror reflectivity	76.4%	74.3%
Monochromatic flux with U27 (both mirrors 2.5 mrad)	$5.5 \times 10^{13}\ \gamma$/sec	$2.8 \times 10^{13}\ \gamma$/sec
Monochromatic Flux with U23 (both mirrors 2.5 mrad)	$8.1 \times 10^{13}\ \gamma$/sec	$4.3 \times 10^{13}\ \gamma$/sec
Monochromatic mirror reflectivity (Rh Stripe)	95%	93%
Horizontal focus size	~82.2 μm	~95 μm
Vertical focus size	~38.3 μm	~20 μm

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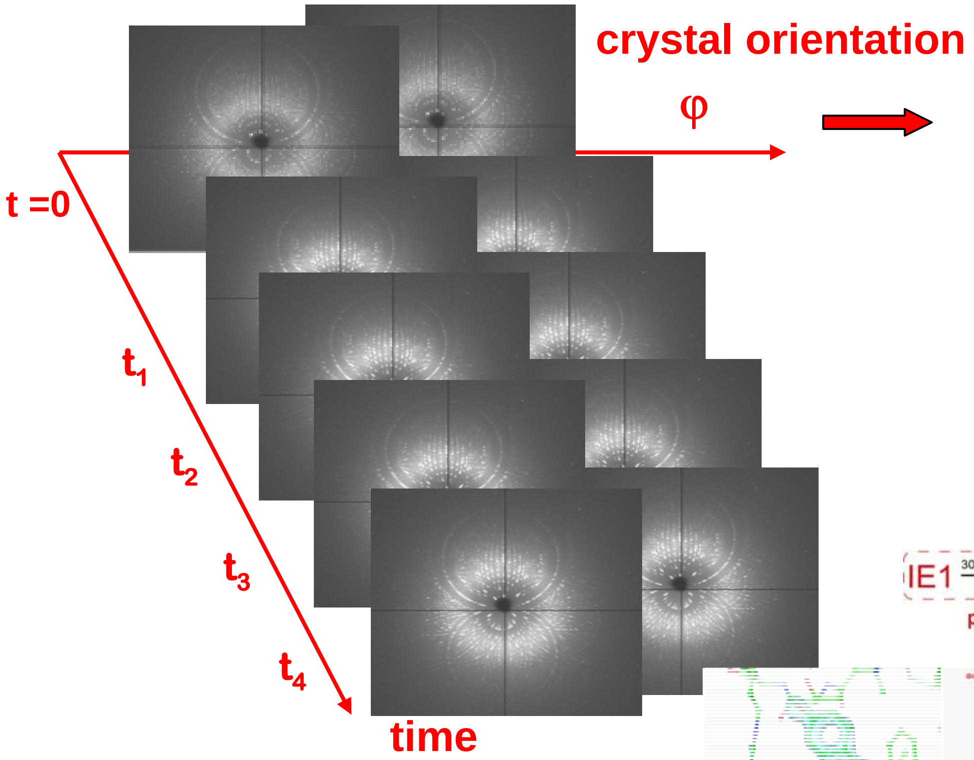
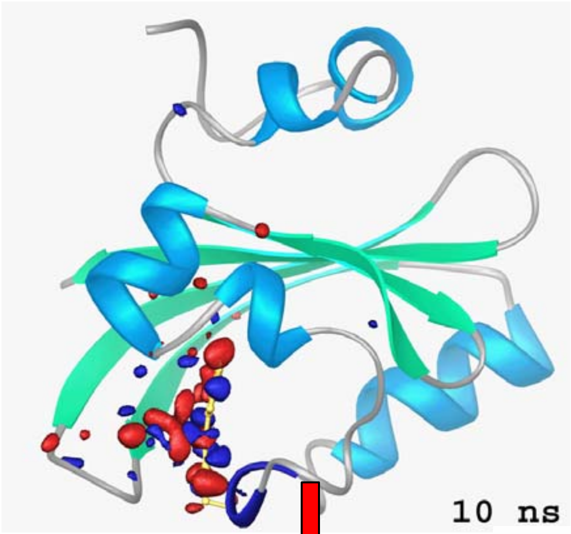
Time Resolved Data Collection

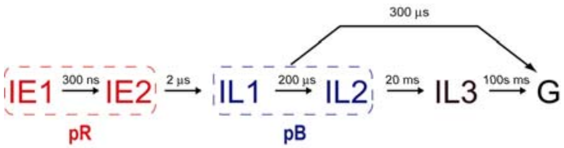
Timing scheme used at BioCARS in the standard 24-bunch APS operating mode. A single 100ps X-ray pulse is isolated by a recently upgraded ultra-fast X-ray chopper, rotating a ~1KHz, followed by a ms shutter.

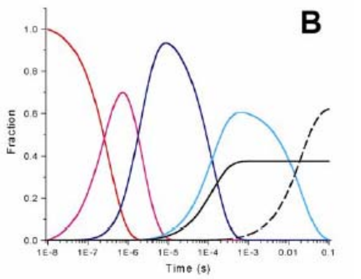


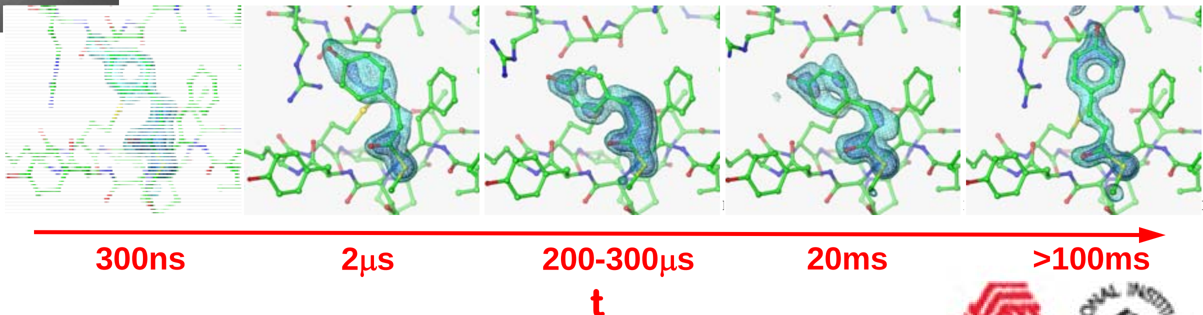
Time-dependent difference (light-dark) electron density map movie, $\Delta\rho$ (t)



10ns-100ms
E46Q PYP







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Experimental Infrastructure

Upgrades and Improvements

- Picosecond laser system installed and operational
- New timing electronics for storage-ring/laser-system synchronization
- Small vertical-beam focus and improved Jülich chopper jitter leads to 24-bunch single pulse isolation
- In-situ x-ray pulse monitoring

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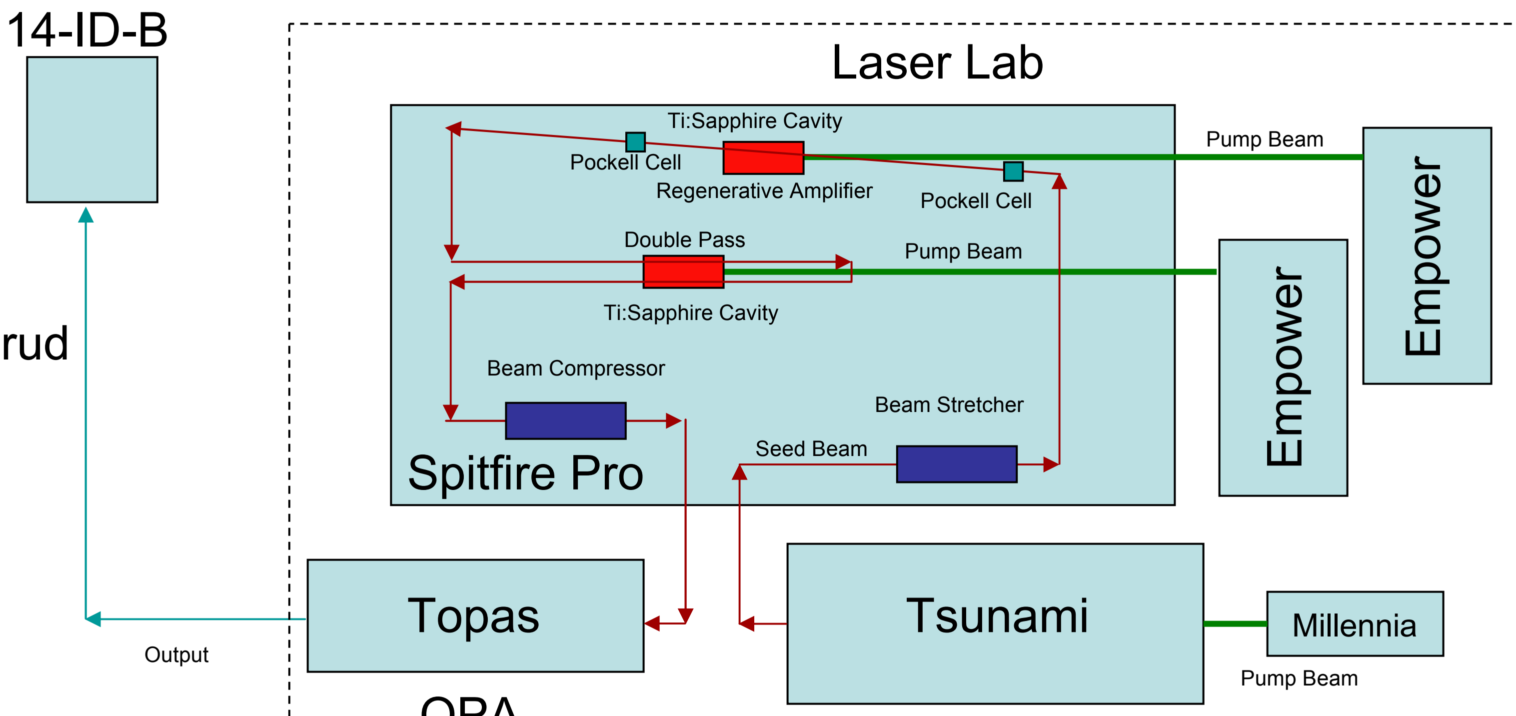
Picosecond Laser System

Modified Spectra Physics Spitfire Laser

P. Anfinrud

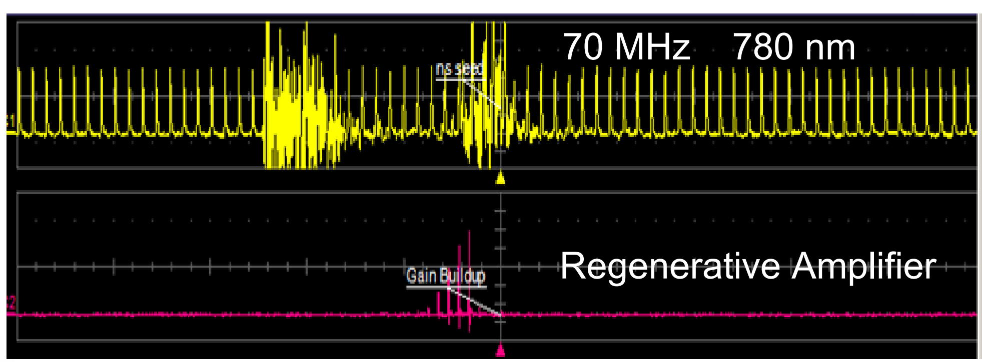


Schematic representation of the laser system

Optical Parametric Generation

$$\hbar\omega_p = \hbar\omega_s + \hbar\omega_i$$

$$\hbar k_p = \hbar k_s + \hbar k_i$$



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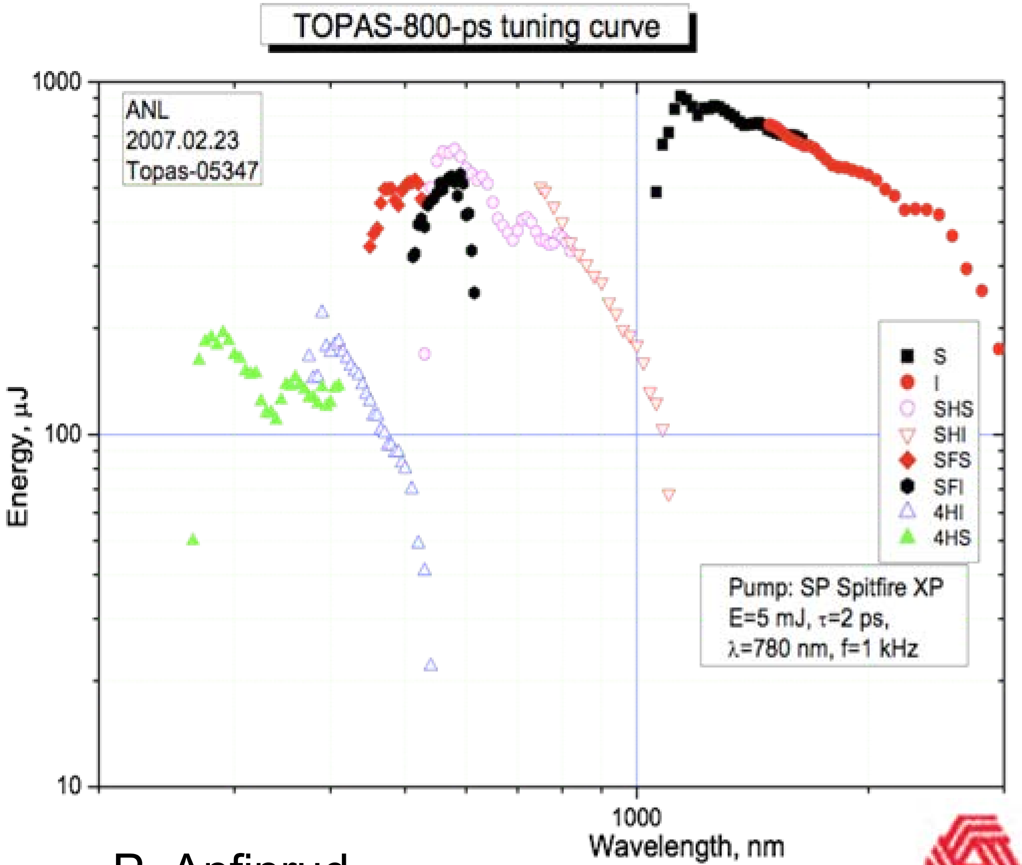
Picosecond Laser System Specifications

Laser	Wavelength	Pulse Width	Max Rep Rate	Energy/Power
Millennia	532 nm	CW	CW	5W
Tsunami	780 nm	100 fs	70 MHz	850mW
Empower	527 nm	300 ns	1kHz	15mJ/15W
Spitfire	780 nm	1.2 ps	1kHz	4.8mJ/4.8W
Topas	See Plot	1.2 ps	1kHz	See Plot



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Fiber Coupled OPOTEK Laser

Optical Parametric Oscillator

Wavelength
410 – 630 nm
235 – 380 nm

Rep Rate
10Hz

Pulse Width
6 nsec

Pulse Energy
<35 mJ @ 410-630nm
<6 mJ @ 235-380nm

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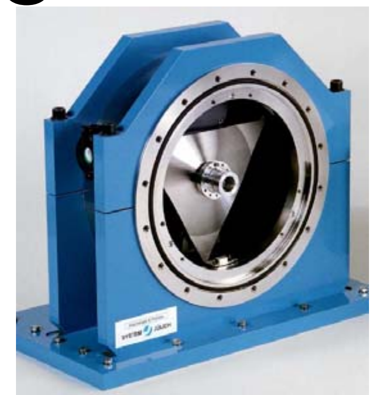
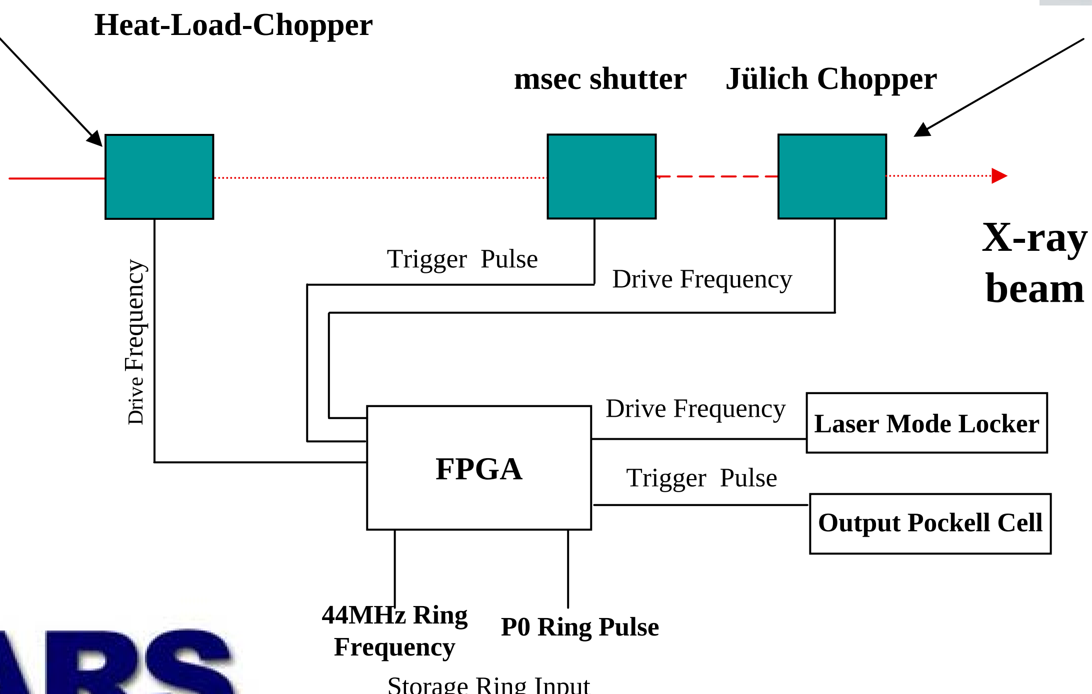
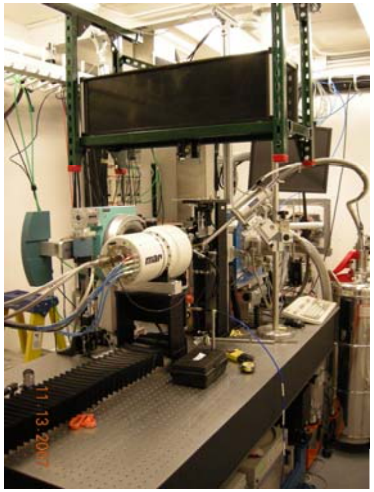
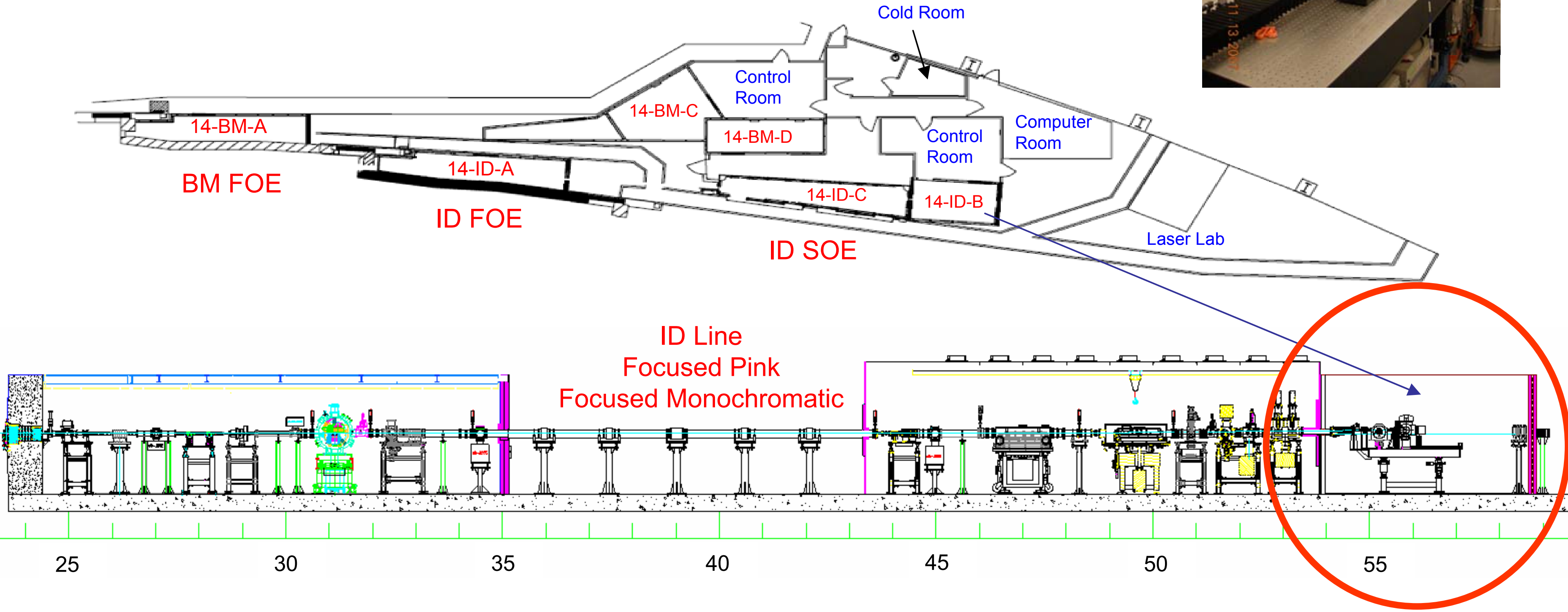
Shutter Train and Synchronization with the Storage Ring





14IDB Station





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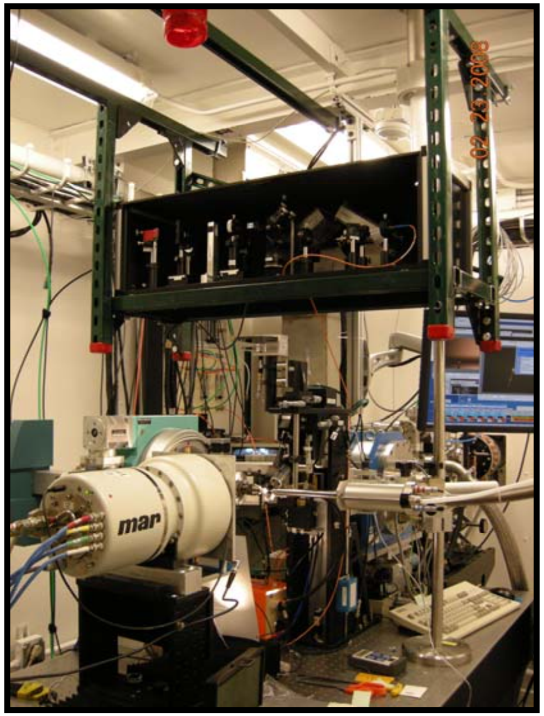
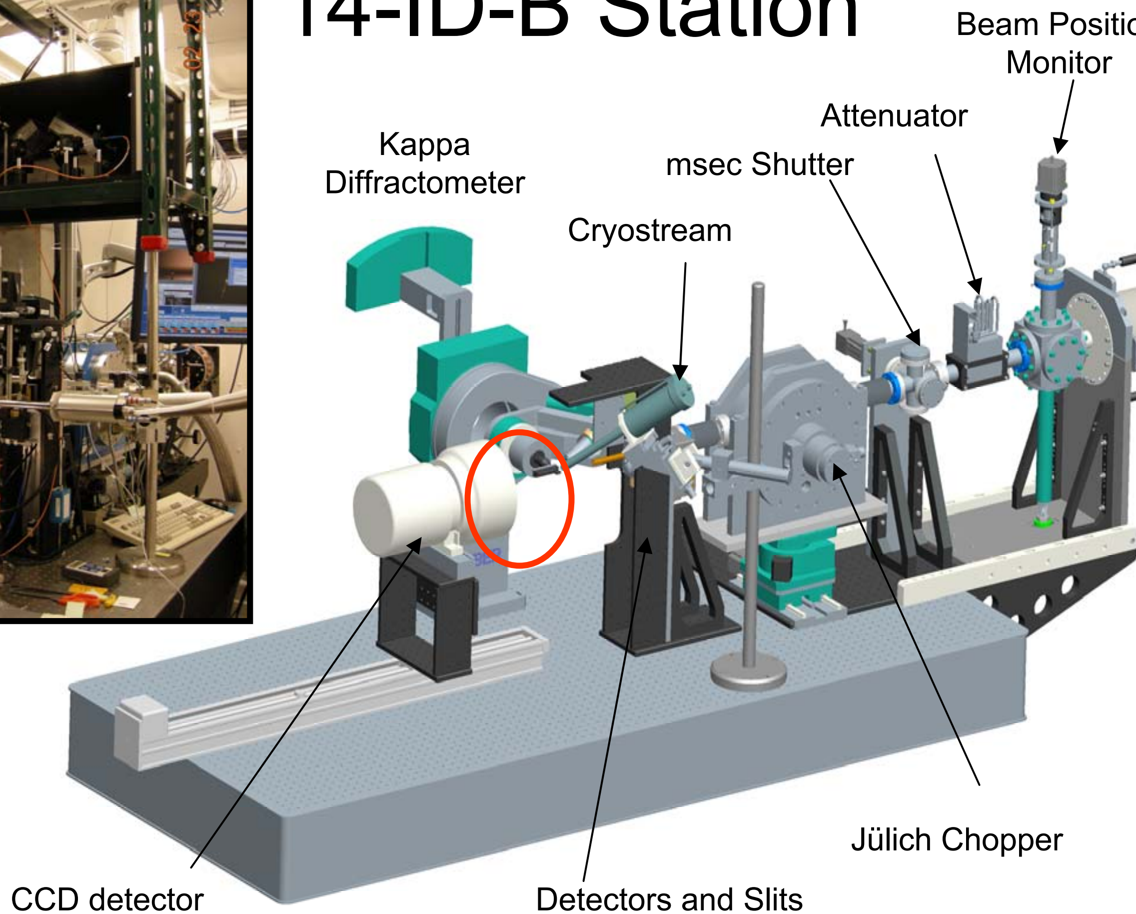
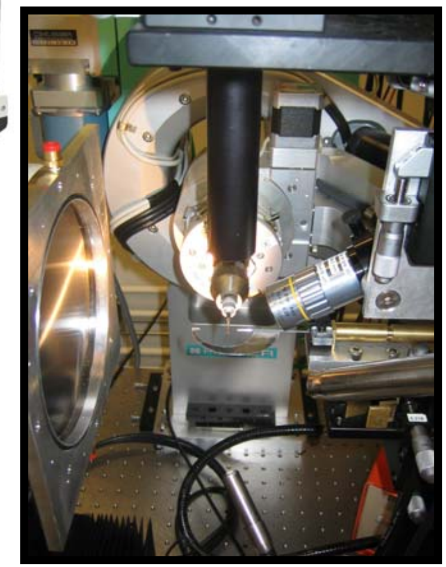
14-ID-B Station







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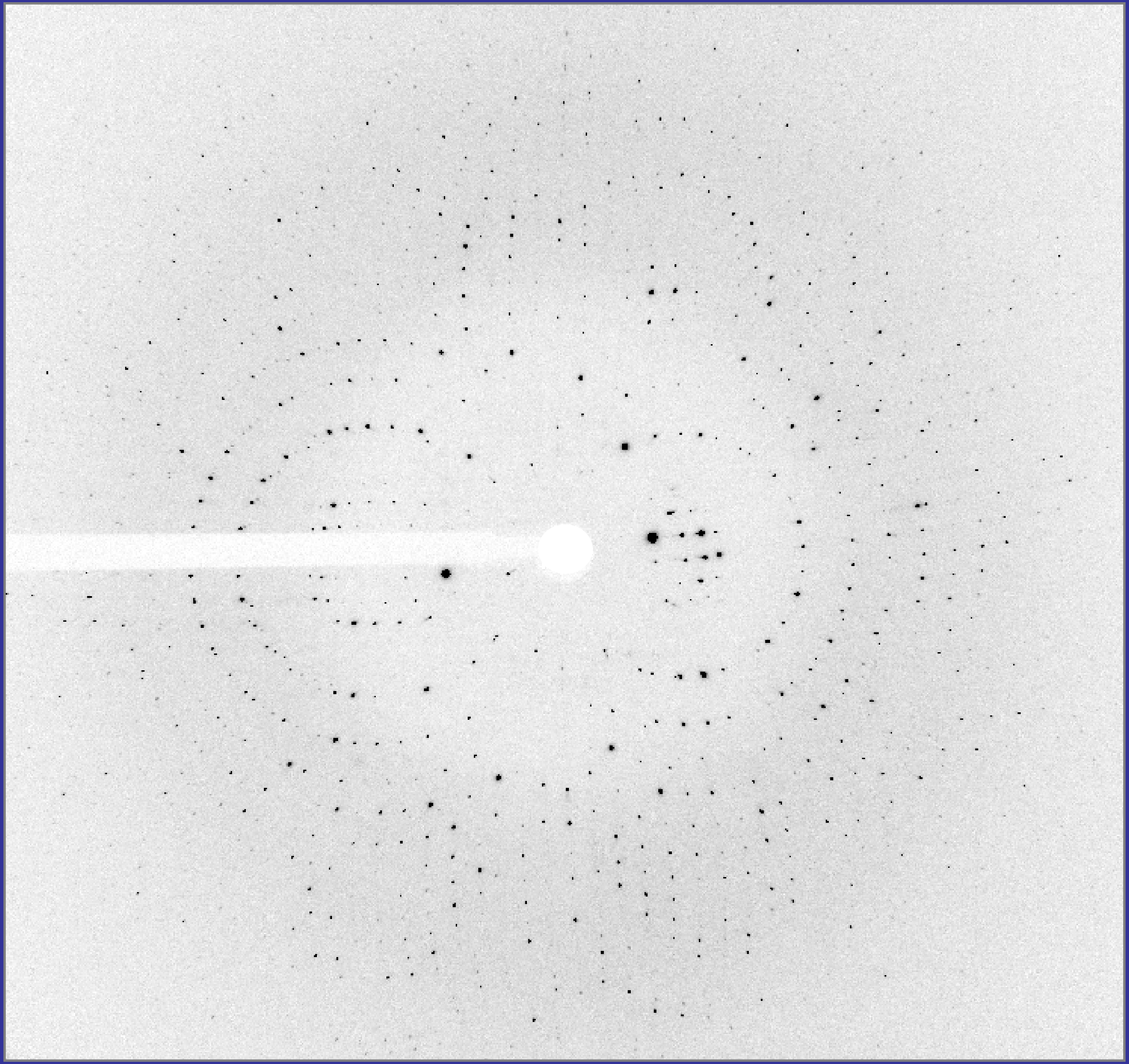
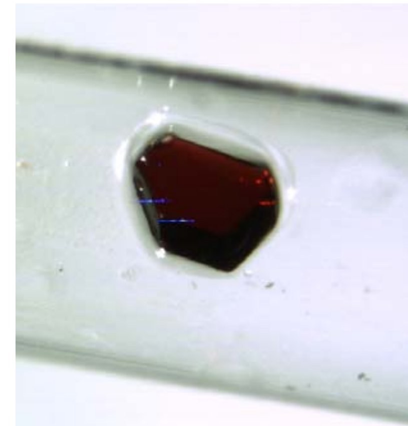
First Laue Image from the 14-ID Beamline December 2007
Time-resolved macromolecular crystallography with single X-ray pulses

- Single 160ps X-ray pulse (APS hybrid mode)
- Dual in-line undulators (U23 & U27 at 12keV)
- $35(v) \times 100(h)$ μm^2 beamsize (KB mirror pair)
- $\sim 2 \times 10^{10}$ photons per 160ps X-ray pulse
- Hemoglobin crystal, HbCO ($35 \times 100 \times 200$ μm^3 diffracting volume)



Latest time-resolved X-ray diffraction data
August 2008

Tetrameric hemoglobin HbII from clam *Scapharca Inaequivalvis*
Collaboration with William E. Royer, Jr.
Univ. of Mass. Medical School, Worcester, MA



- Double-focused X-ray beam size:

  ~70 (v) X 120 (h) μm^2
- Laser: 480nm, ~35ps laser pulse duration,

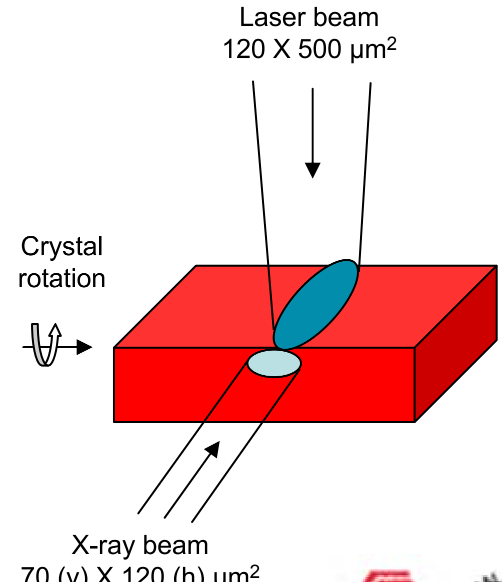
  ~140μJ, 120 X 500 μm^2 beamsize

  ~25% reaction initiation (ligand photodissociation)
- Collected a comprehensive time series on a single crystal:
  laser off, 100ps, 1ns, 10ns, 100ns, 1μs, 10μs,
  100μs, 1ms, 10ms

  (600 frames total; exposure per frame:

  8X1 single 150ps X-ray pulses; hybrid mode)
- Very strong signal difference map signal: +/- 10σ

Crystal and X-ray/laser beam geometry.



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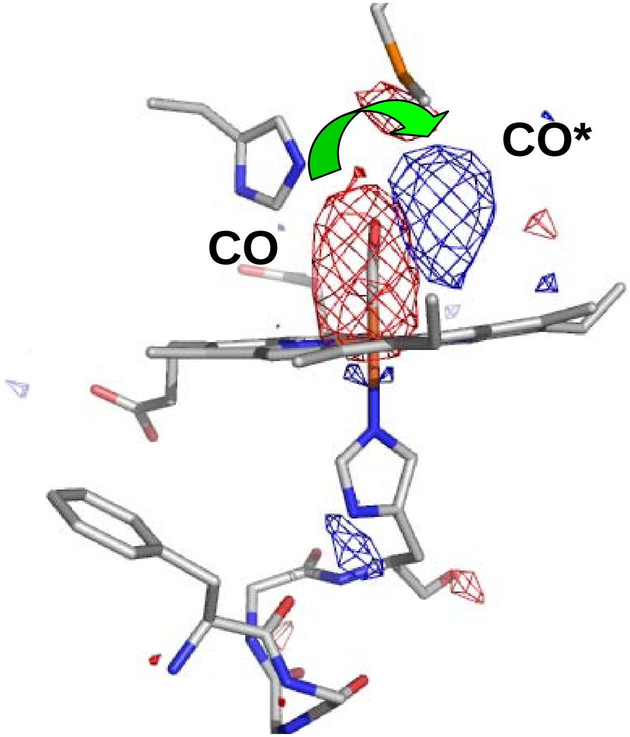
Vukica Srajer

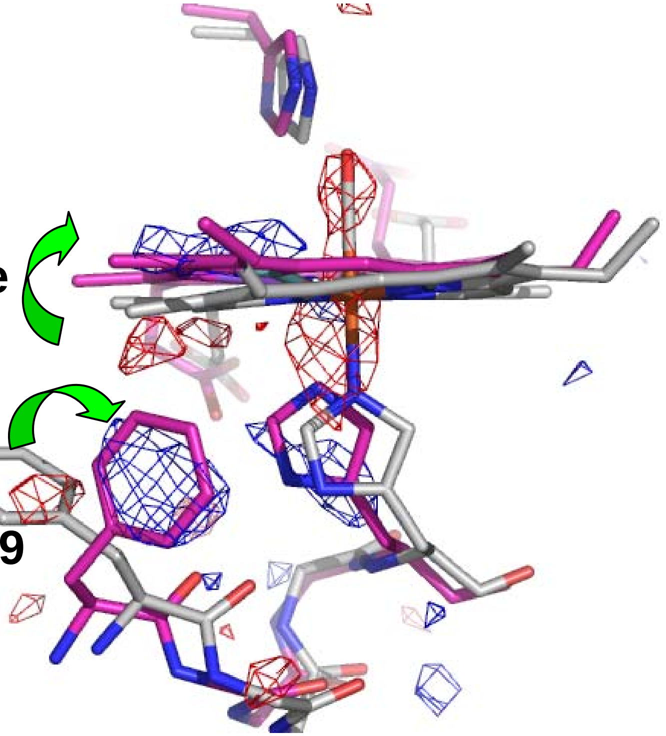
HbII: 2.2Å difference Fourier maps from April 2008

Subunit B

100 ps



1 μs
HbII-CO and deoxy models
(from static crystallography)



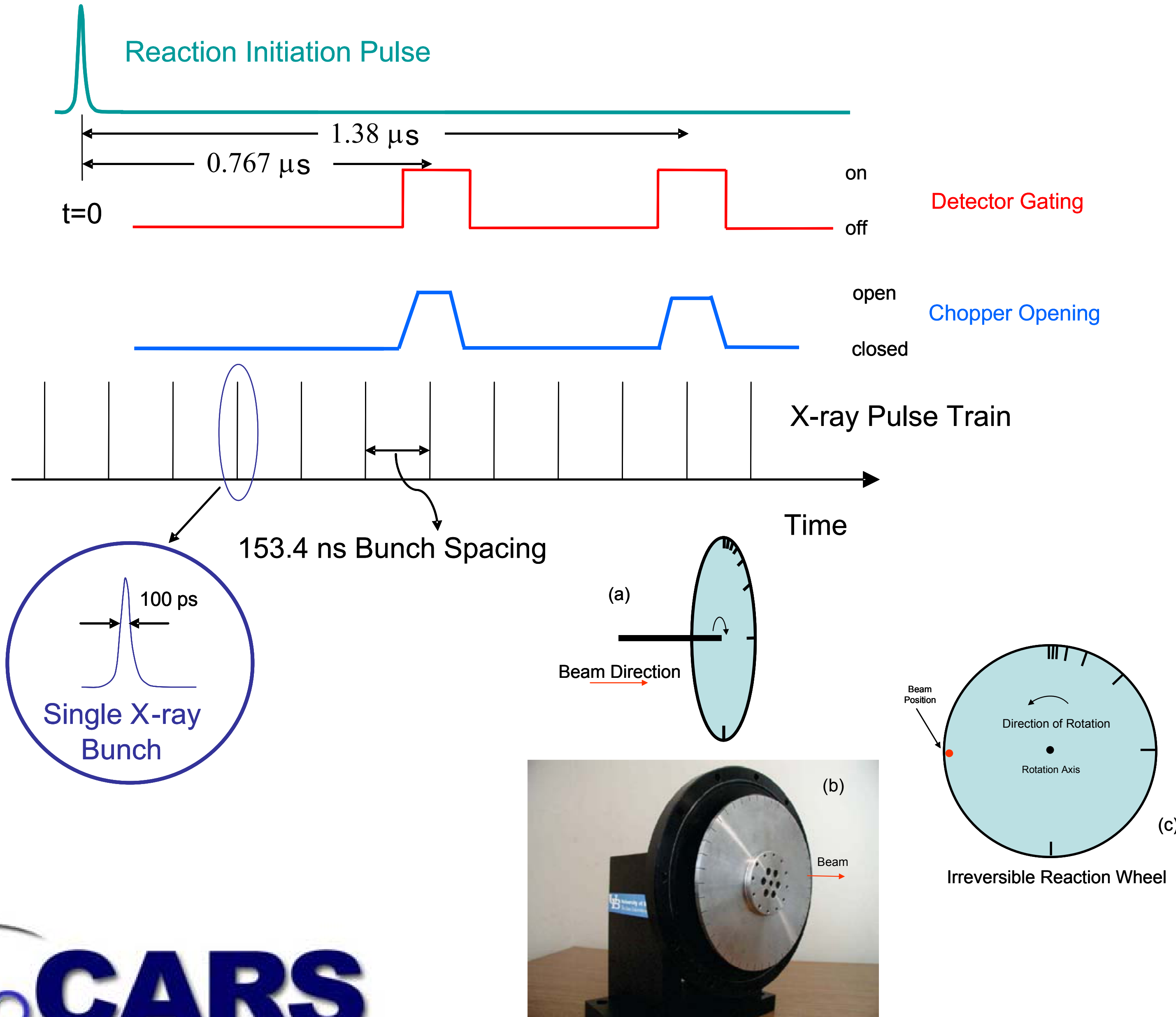
Red:
negative density
Blue:
positive density

Vukica Srajer

Irreversible Reactions

- Difficulty: It requires diffraction patterns from many crystals (of differing quality) to get the necessary number of pump-probe time points
- Solution: Fast time-slicing pixel array detector used in conjunction with a logarithmic x-ray chopper allows a single reaction-initiation pump pulse to be followed by many probe pulses

Timing diagram for detector-chopper x-ray pulse gating



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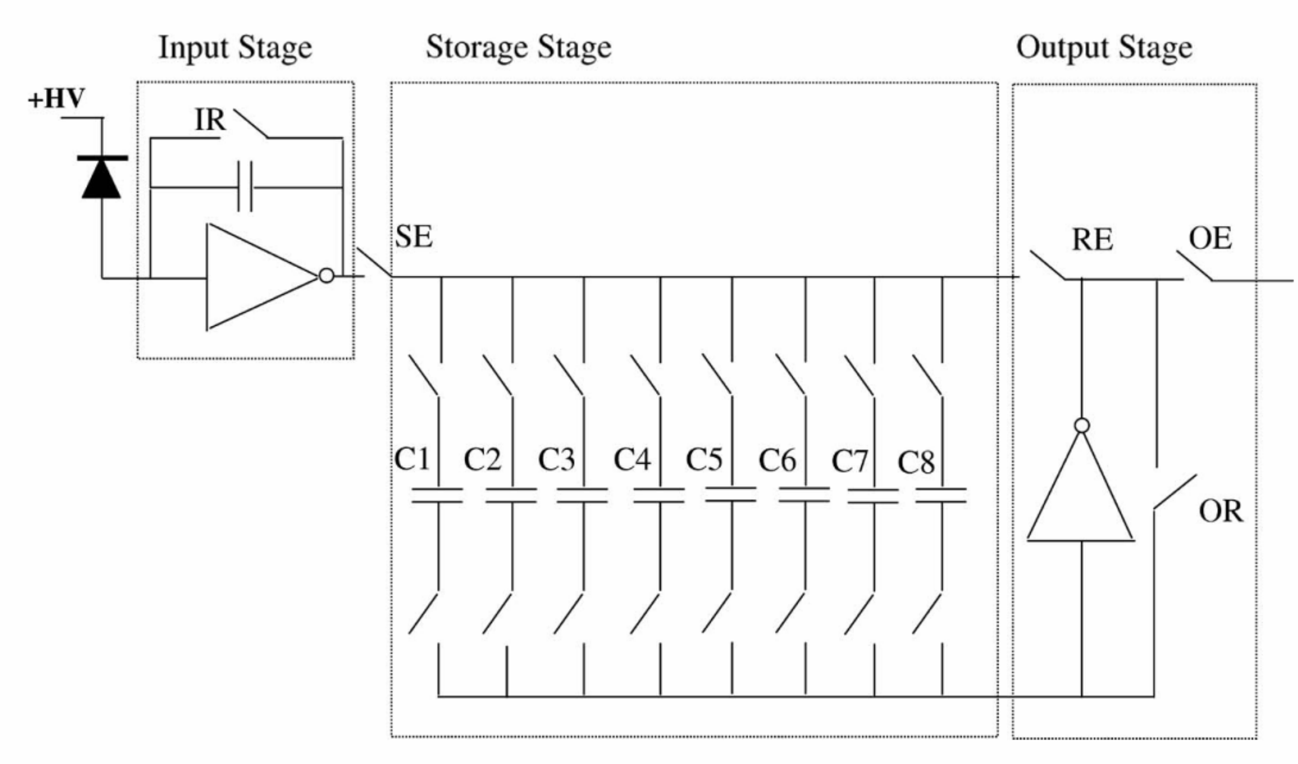
On-board storage for each pixel

Gruner's group at Cornell University is developing an analog-integrating PAD or APAD



- Allows multiple frames to be taken in quick succession
- The detector must be capable of measuring very high instantaneous count rates
- Typical count rate ~5000 photons per ~100 ps X-ray pulse per pixel, equivalent to 50 THz/pixel.

Conclusions

- Following the completion of the comprehensive 14-ID upgrade, users successfully conducted first 100ps time-resolved experiments.
- Excellent quality of data collected with 100ps time-resolution on ~10X smaller crystal volumes and with ~10X shorter X-ray exposures than in the past experiments at 14-ID.
- Experiments involving much smaller crystals and irreversible reactions are feasible at BioCARS.
- Major goal for BioCARS and users: to address by time-resolved crystallography more complex molecules and biological processes such as cooperativity, signal transduction and catalysis.
- BioCARS: premier facility for time-resolved macromolecular crystallography world-wide.

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Workshop on Time-resolved Macromolecular Crystallography 2008
November 20-22, 2008

Hosted by BioCARS

Held at Advanced Photon Source, Argonne National Laboratory

http://cars9.uchicago.edu/biocars/