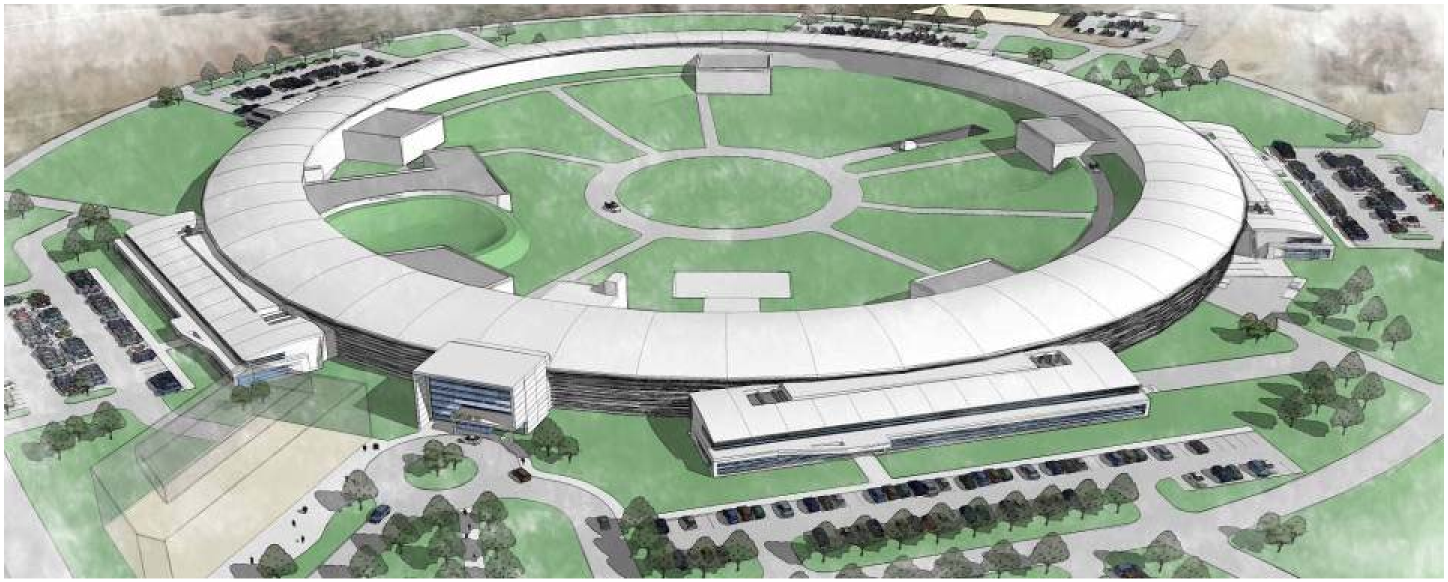
National Synchrotron Light Source II



Powder Diffraction Beamline

J.M. Ablett & D.P. Siddons
EFAC, October 4, 2007

Office of Science
U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

Scientific Mission of NSLS-II PD Beamline”

(user and workshop input http://www.bnl.gov/nsls2/workshops/UserWorkshop.asp)

The proposed powder diffractometer at NSLS-II will be the US' only high-resolution instrument capable of collecting data at high energies (20 keV to 100 keV). This will make it ideal for *in situ* and time resolved studies of samples held in environmental cells.

High energy for atomic pair distribution function method (PDF) and Environmental Cell (e.g. DAC) work.

Some scientific thrusts/importance for high energy X-ray elastic scattering

Water and ice *Structures and transformations (0.1 – 50 GPa)*
Novel clathrates *Energy resources, hydrogen/energetic gas storage*
Dense liquids, melts, glasses *Why is gallium a liquid at HP? Glass technology*
Pressure induced amorphization *New materials with new properties under pressure*
Strongly correlated systems *Structures, dynamics, and mechanisms. Novel behavior*
Structures of new ferroelectrics *New materials and pressure tuning behavior*
Nanomaterials *Differences between bulk and nano-materials*

Beamline Requirements and Specifications

- Several itterations of beamline requirements over the past 6 months - settled on high-energy high-resolution PD capabilities - in collaboration with discussion with potential BAT members.
- In early stages of BAT formation:- <u>workshop on November 30th, 2007.</u>

- <u>Major Specifications:</u>
  - Attenuators to remove significant power load from the low energy region.
  - Mirror: 1.5 m long Pt-coated. 20 - 40 keV range. 2 mrad incidence angle.
  - Monochromator. Sagitally Focusing Asymmetric Laue. (In use at NSLS X17 and tested at X7b)
  - Energy Range. 20 keV to 100 keV (High energy end determined by Damping Wiggler Output)
  - Energy Resolution. dE/E 1×10^{-4} to dE/E 1×10^{-3}
  - Vertical Divergence. Mirror acting as collimator ~ <5 ❍rad rms or ~ 100 ❍rad (mirror out - natural x-ray beam divergence)
  - Vertical Focusing: 1.5 m long mirror acting as ~ 1:1 focusing device. - 400 ❍m (fwhm) (assuming 3 ❍rad rms figure error).
  - Horizontal Focusing: Sagitally Focusing Laue (1:1) ~ 300 ❍m (fwhm). 0.5 m long Horizontally Focusing Graded-Multilayer (~28:1) - ~ 14 ❍m (fwhm) - (slope error included)
  - Flux on Sample - see later!

Beamline Requirements and Specifications

- Photon Beam Stability Requirements

(NSLS-II stability workshop page: www.bnl.gov/nsls2/workshops/Stability_Wshop_4-18-07.asp)

- Operational Modes: crystal analyzer (angular stability), area detector (position stability).

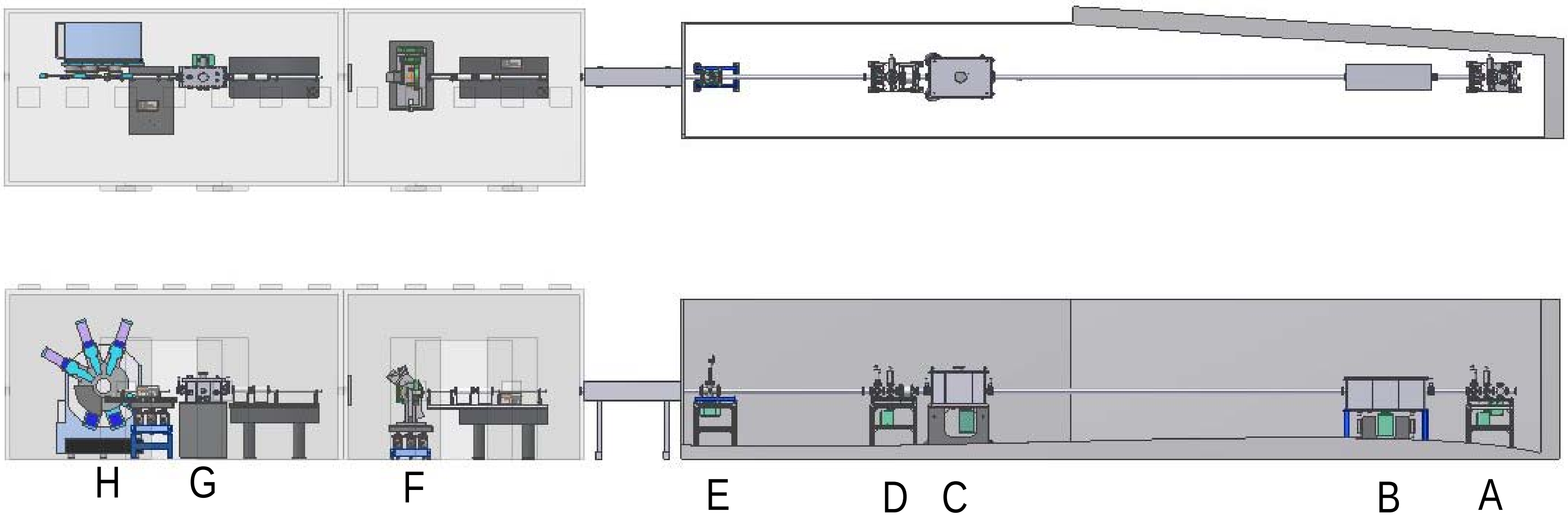
<u>10 μm spatial stability</u> in both x-and-y (area detector)

<u>1 μrad angular stability</u> in vertical diffraction plane (analyzer mode).

Monochromator temperature stability < 10^{o}C

.

Beamline Layout - Overview



A. 29.0 m. Be window & filters

B. 31.1 m 1.5 m long mirror (foc. & coll)

C. 39.95m Laue Mono - sagitally focusing

D. 41.36 m Beam Monitoring

E. 45.1 m Photon Shutter

F. 51.3 m Sample A Position

G. 55.592 m HFM Multilayer

H. 57.6578 m Sample B Pos.

Experimental Hutch 1: High-Energy, High-Resolution: Costed

Experimental Hutch 2: 'Routine' Powder Diffraction with Area Detector. Enclosure costed. Equipment to be used from NSLS-I stock. Possibly served by canted wiggler device or as an additional endstation.

Damping Wiggler Insertion Device

- Damping Wiggler (high- β straight).
- Damping Wiggler will provide high flux, high brightness hard x-rays ($E_{critical}$~10.8 keV).
- Current thinking: either one 7m long device or two 3.5 m devices - one canted.

machine and damping wiggler parameters

Eo	3 GeV
Io	500 mA
N periods	70
λ_u	10 cm
B	1.8 Tesla
k	16.81

rms electron beam values

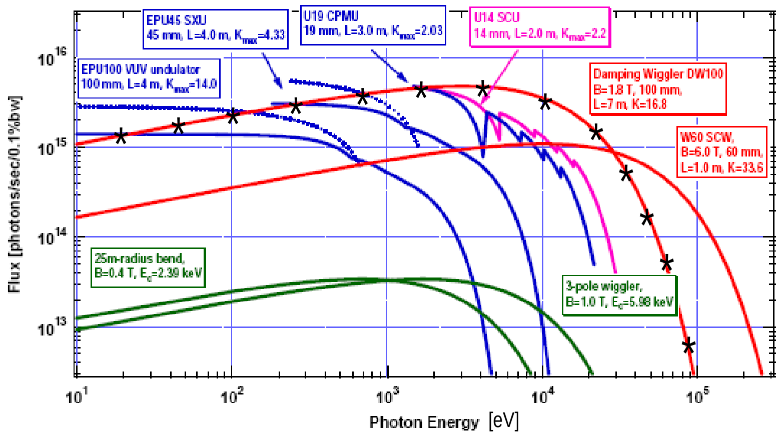
σ_x	99 μm
σ_z	5.5 μm
σ_x'	5.5 μrad
σ_z'	1.8 μrad

Total Power (integrated over all angles) ~ 65 kW

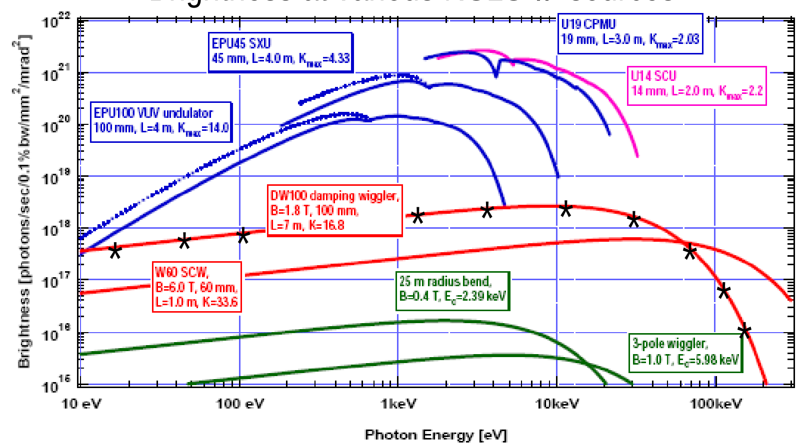
Power Density ~ 54.9 kW/ mrad^2

Damping Wiggler: Flux and Brightness

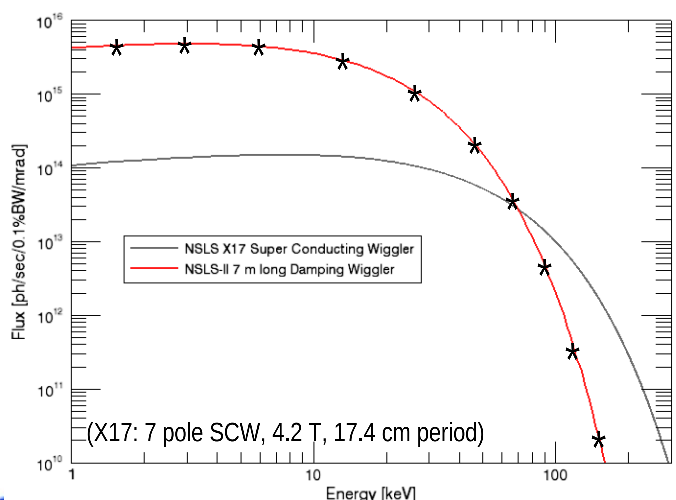
Flux at various NSLS-II sources



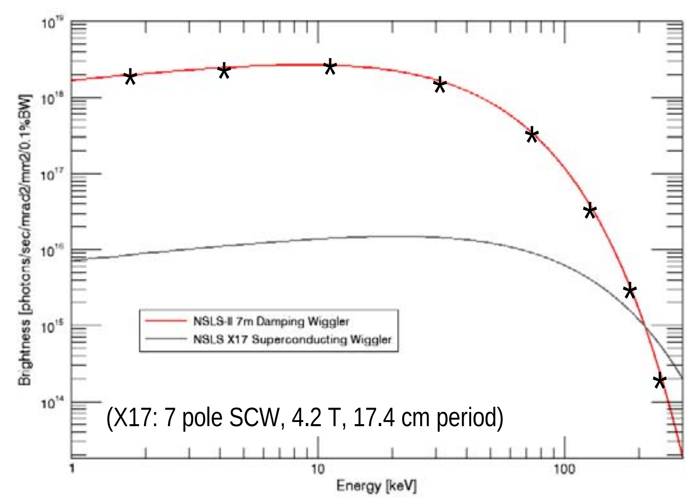
Brightness at various NSLS-II sources



Flux Comparison between NSLS-II DW and X17 SCW



Brightness Comparison between NSLS-II DW and X17 SCW



Managing the Power Load & Energy Range

Energy

110 keV

90 keV

70 keV

50 keV

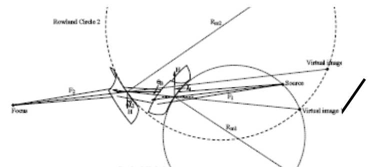
30 keV

10 keV

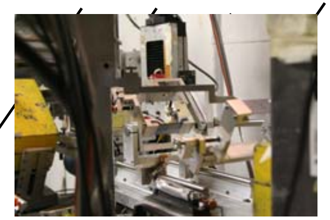
20 keV to 40 keV

filters, mirror
& Laue Mono

>40 keV



filters + Laue Mono



NSLS X7b Laue Mono (Z. Zhong)

Also used at X17 SCW beamline

Energy

110 keV

90 keV

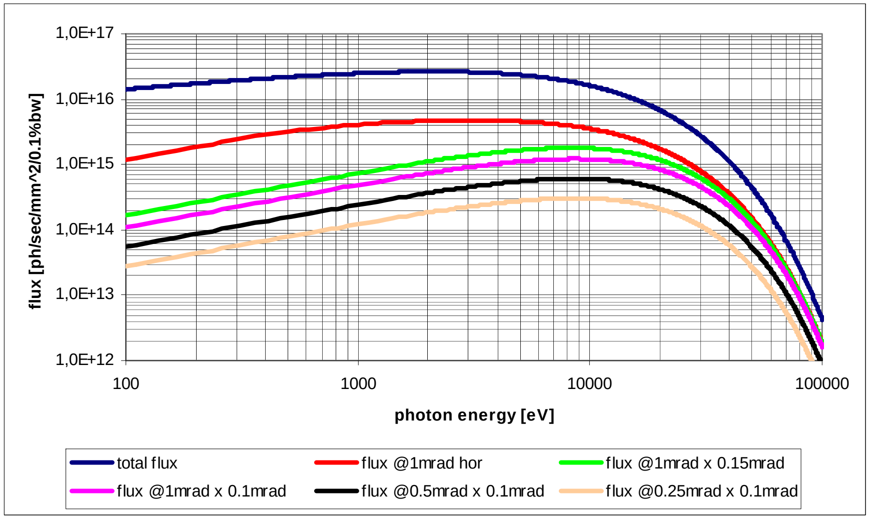
70 keV

50 keV

30 keV

10 keV

Power Load Considerations



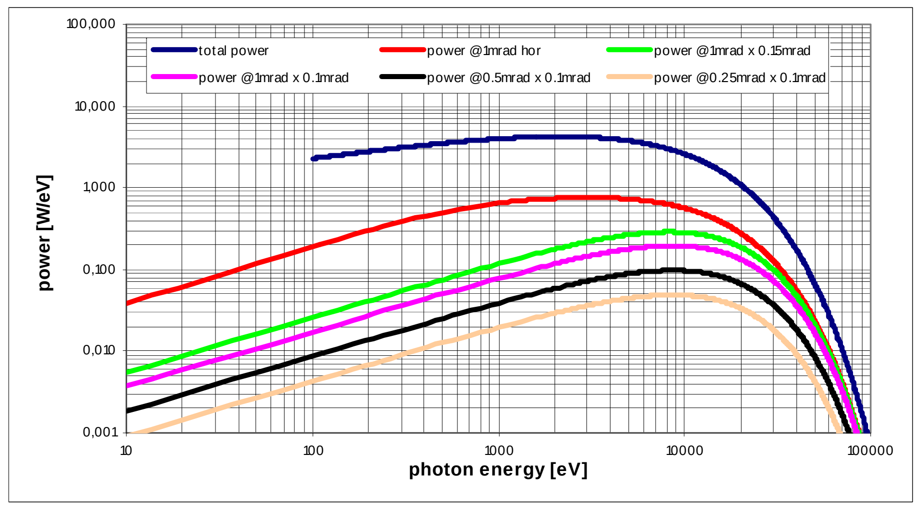
Aperturing

in Front End

Control acceptance by fixed aperture in front end and/or by adjustable 4 slit mechanism.

Total Power	64.5 kW
1 mrad [hor] x 0.1 mrad [ver]:	5.2 kW

corresponds to a 1.5m long mirror operating at a grazing incidence angle of 2 mrad in the front optical enclosure (~ 30 m from source point).

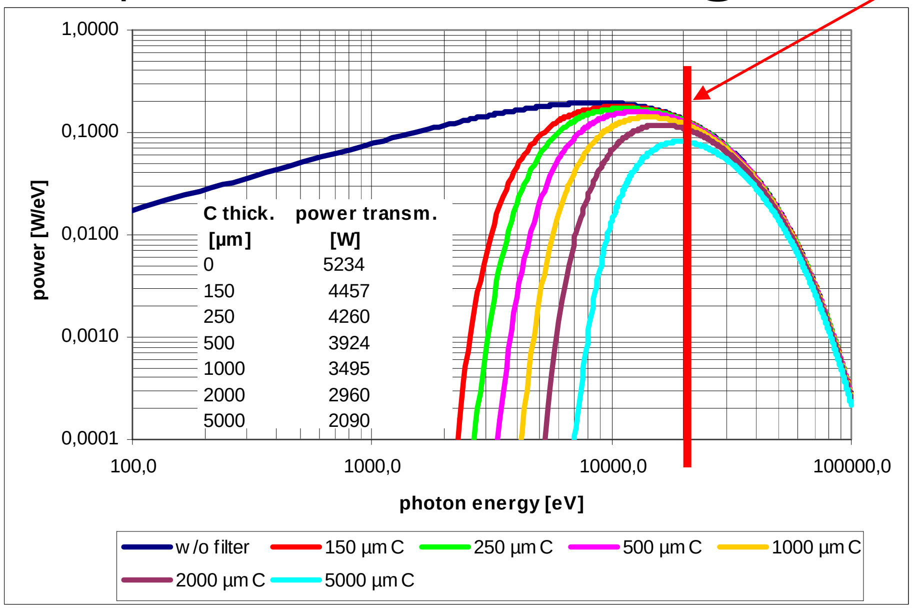


Office of Science
U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

Power Load Considerations - Filtering

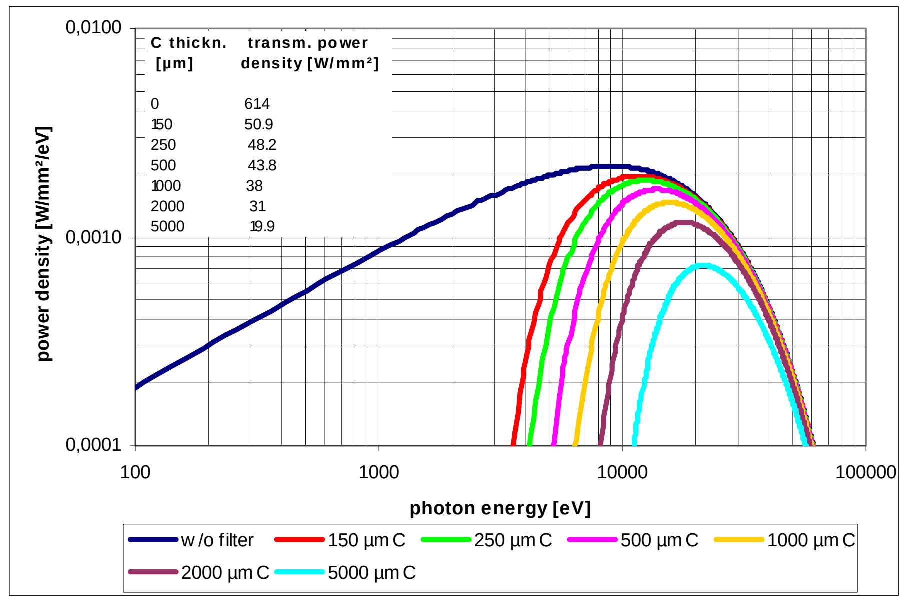
Acceptance: 1 mrad x 0.1 mrad @ 30 m



2 mm of C removes ~ 2 kW of power
5 mm of C removes ~ 3 kW of power

.....and beamline can operate effectively above 20 keV

Acceptance: 1 mrad x 0.1 mrad @ 30 m



Reduce power load on critical optical components by filtering out the low energy x-rays.

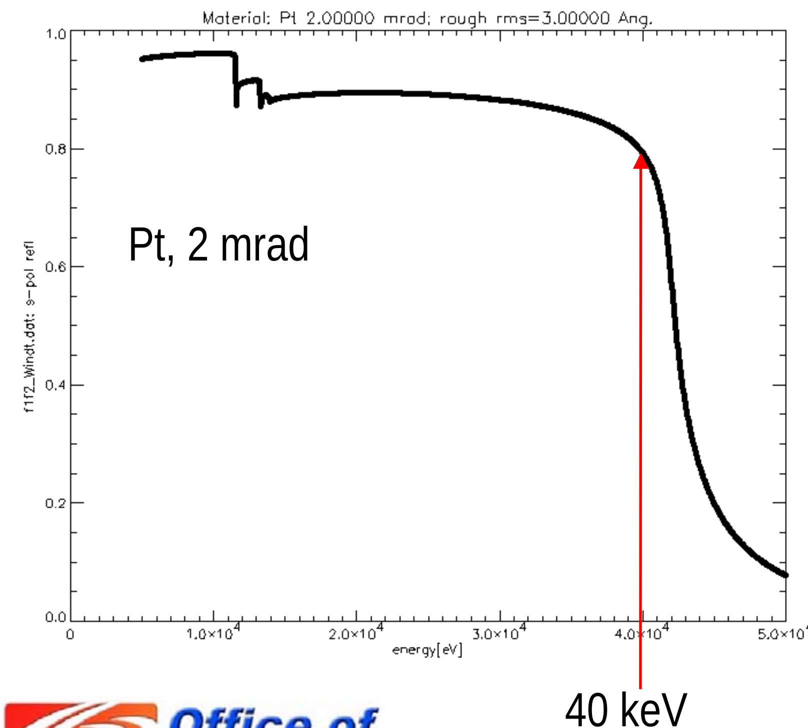
(Energy range of beamline > 20 keV)

Office of Science
U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

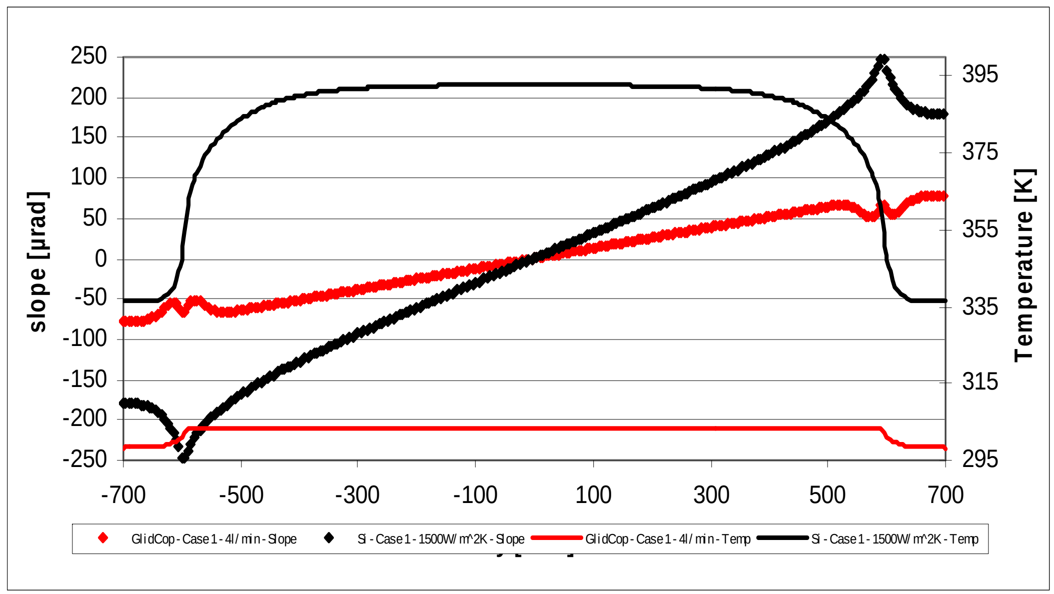
Vertically Focusing/Collimating Mirror

Length: 1.5 m, mirror bender
Pt Coated, 2 mrad grazing incidence
Vertically Focusing (~ 1:1) or collimating
Operating Energy Range (with Laue) 20 - 40 keV



Mirror FEA

'Worst Case' Scenario:
Total Power: 7 kW
Power Density: 0.18 W/mm^2
Black: Si mirror with side cooling
Red: Glidcop mirror directly cooled



Office of Science
U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

Double-Crystal Laue Monochromator

Sagittally Focusing Double-Crystal Laue Mono

In use at X17, (tested for X7B)

Dr. Zhong Zhong design

Can take a large horizontal fan (> 3 cm) and focus it down to a few hundred microns

Asymmetric Bent Si (111), Si (311)

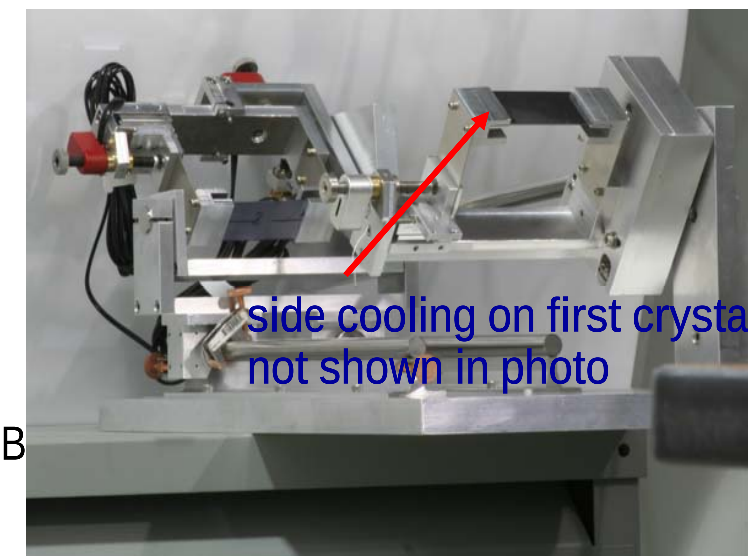
Band-Width (in focusing mode)
dE/E ~ few x 10^{-3} for Si(111) ~5×10^{-4} for Si(311) - great for area detector work. Need to unbend for high-resultion work- i.e. no focusing - acts like in normal Bragg case.

Energy Range: 20 to 100 keV

Side-water cooling works at X17, but more power on NSLS-II damping wiggler - needs R&D.

Fixed Exit - modified boomerang style design

2:1 Focusing in this design. 1:1 Focusing previously at X17/X7B

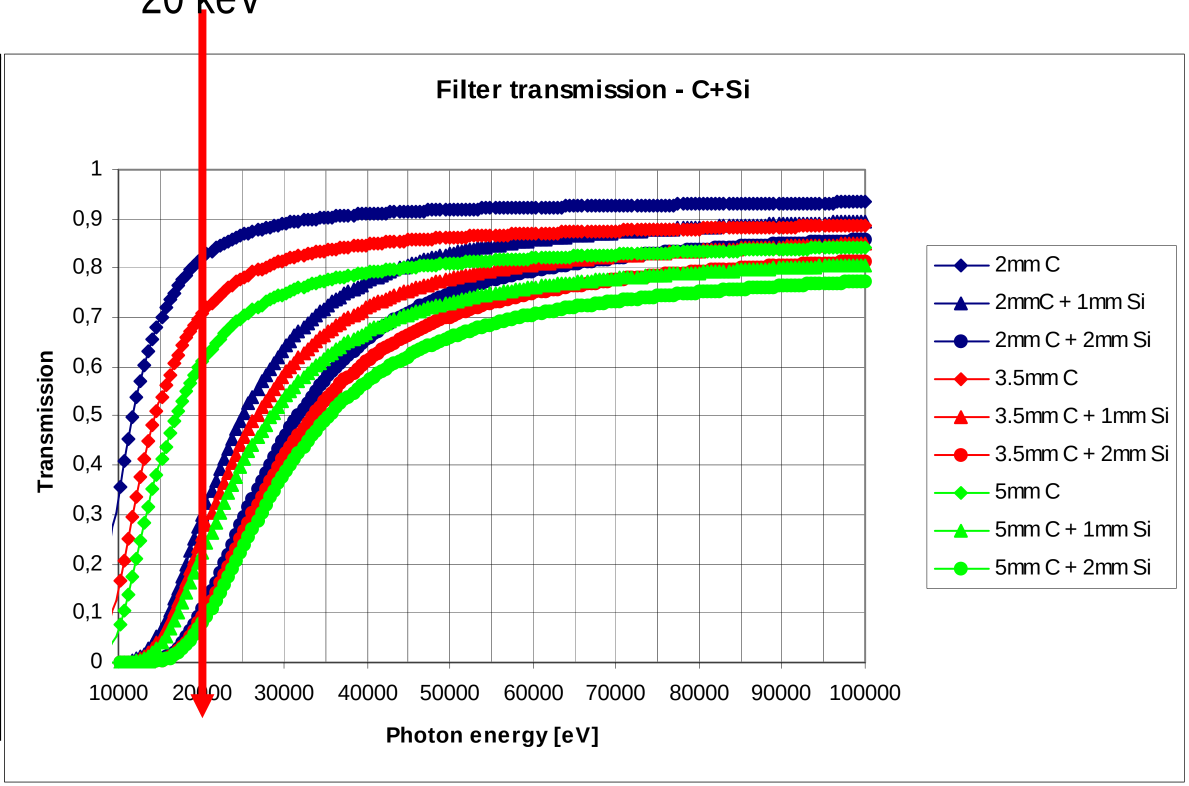


Double-Crystal Laue Monochromator

Power Loads

C - prefilter (mm)	Si filter (mm)	Power abs. in C [W]	Power abs. in Si [W]	Power abs. in 1st laue xtal [W]	Power dump in beam stop [W]
2	0	3331	0	1642	2435
2	1	3331	2246	354	1477
2	2	3331	2840	177	1060
5	0	4560	0	907	1941
5	1	4560	1341	271	1236
5	2	4560	1803	143	902

20 keV



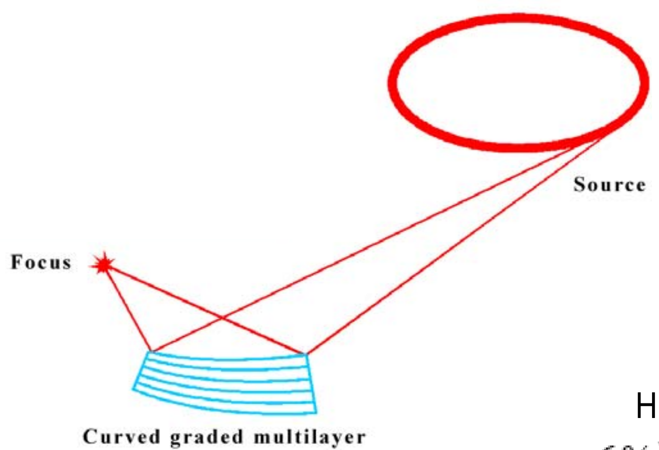
~ 150 W absorbed by first crystal. Still significant. R&D needed on cooling schemes. Maybe thinner crystals may help, but more difficult to control bending.

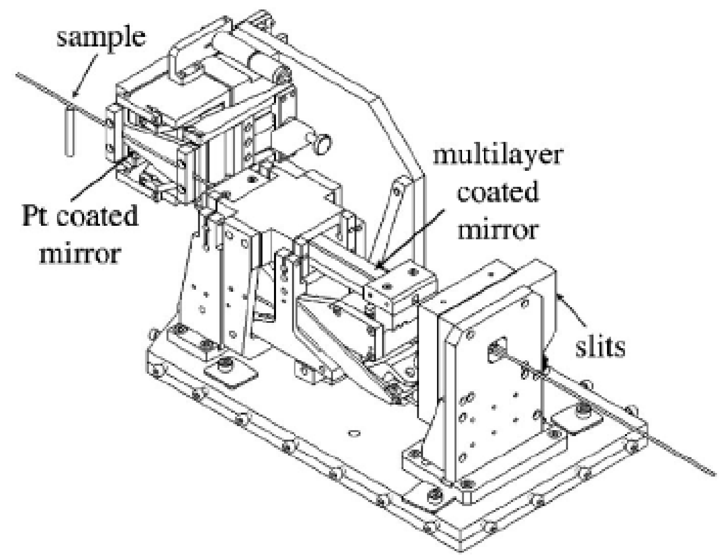
Horizontally Focusing Bent Graded-Multilayer

Current technology : 0.5 m. Possibly 1 m in the future.

Place as close to sample as possible for maximum focusing effect (applicable for Diamond Anvil Cell Work). 50:1 demag - 5 µm fwhm horizontal focus.

Example Layers: Ir (20Å)/B_4C(20Å) - 70 periods.





Hignette(ESRF), Rev. Sci. Instrum, 76(6), 063709, 2005

=6%). The multilayer consists of 30 W/B_4C layers with a period of 4.7 nm at the center. It has a 33% nonlinear gradient in period along the length of the mirror, designed so that the Bragg angle condition is kept despite the variation in the angle of incidence. This design is also necessary to achieve a

Flux and Beam Size at Sample Position (57.7 m)

Enegy [keV]	Flux at mirror wih 5mm Carbon [ph/sec/0.1%BW)	Flux after mirror (withdrawn above 40 kev) (ph/sec/0.1%BW)
20	4.6×10^{14}	3.7×10^{14}
40	1.7×10^{14}	1.2×10^{14}
60	3.7×10^{13}	
80	7.2×10^{12}	
100	1.3×10^{12}	

Bent Laue dE/E~1×10^{-3} Si(111). 0.5mm thick crystals + 2mm Si pre-filter. ~ 2:1 focusing

Enegy [keV]	Flux at Sample (ph/sec)	Beam Size
20	1.8×10^{13}	300 µm[h] x 400 µm [v] / 3mm - mirror in
40	1.1×10^{14}	300 µm[h] x 400 µm [v] / 3mm - mirror in
60	3.0×10^{13}	300 µm[h] x 5mm [v]
80	6.2×10^{12}	300 µm[h] x 5mm [v]
100	1.1×10^{12}	300 µm[h] x 5mm [v]

Bent Laue dE/E~4×10^{-4} Si(311). 0.5mm thick crystals + 2mm Si pre-filter. ~ 2:1 focusing

Enegy [keV]	Flux at Sample (ph/sec)	Beam Size
20	7.2×10^{12}	300 µm[h] x 400 µm [v] / 3mm - mirror in
40	4.4×10^{13}	300 µm[h] x 400 µm [v] / 3mm - mirror in
60	1.2×10^{13}	300 µm[h] x 5mm [v]
80	2.5×10^{12}	300 µm[h] x 5mm [v]
100	4.4×10^{11}	300 µm[h] x 5mm [v]

Flux and Beam Size at Sample Position (57.7 m)

Unbent Laue - for hig resolution work. Si(111) dE/E ~ $1x10^{-4}$

Enegy [keV]	Flux at Sample (ph/sec)	Beam Size
20	$1.8x10^{12}$	5.8 cm[h] x 400 µm [v] / 3mm [v] - mirror in
40	$1.1x10^{13}$	5.8 cm[h] x 400 µm [v] / 3mm [v] - mirror in
60	$3.0x10^{12}$	5.8 cm[h] x 5mm [v]
80	$6.2x10^{11}$	5.8 cm[h] x 5mm [v]
100	$1.1x10^{11}$	5.8 cm[h] x 5mm [v]

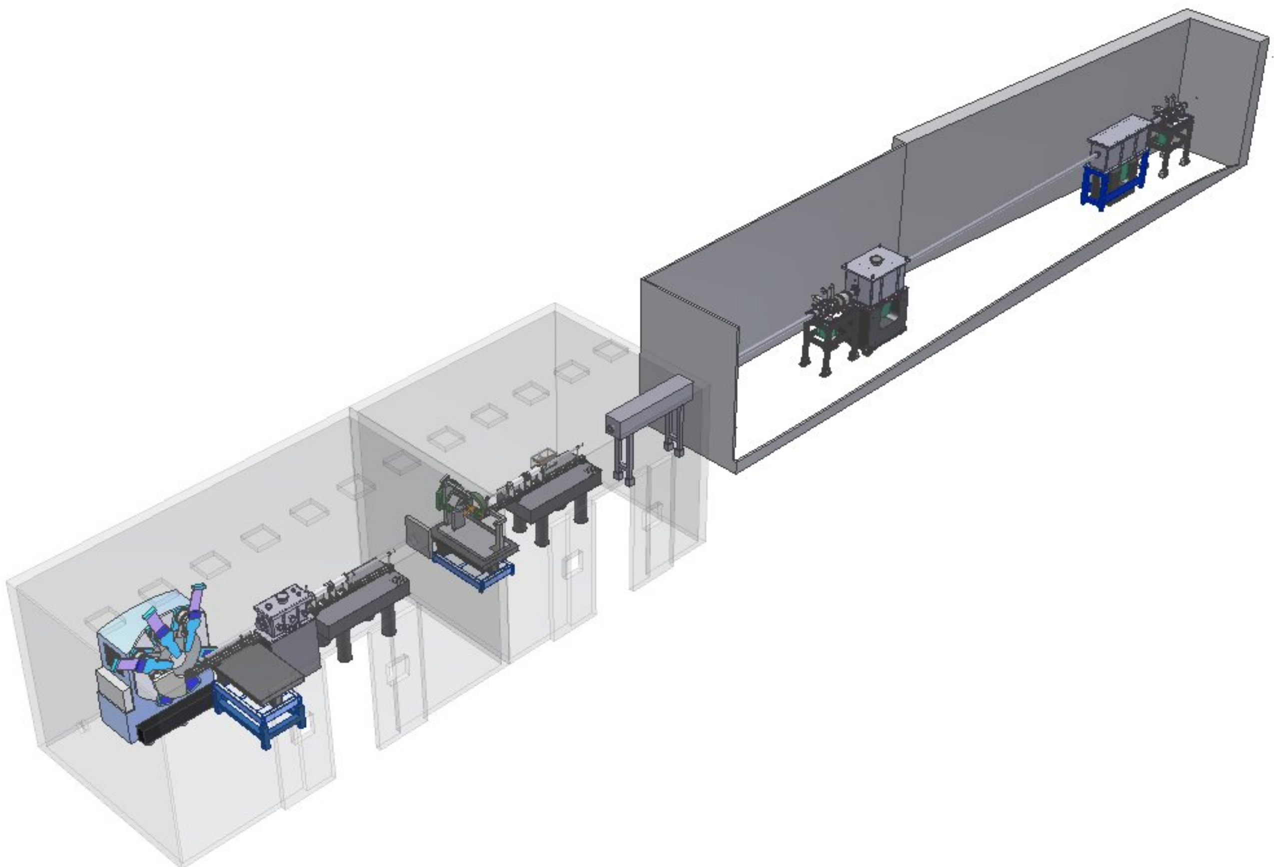
Now add the horizontally-focusing graded multilayer, 0.5 m long optic
Ir (20Å)/B_4C(20Å) - 70 periods.

Unbent Laue - for hig resolution work. Si(111) dE/E ~ $1x10^{-4}$

Enegy [keV]	Inc. Ang. (mrad)	Hor. Accep (mm)	Flux (ph/sec)	Beam Size
20	7.7	3.15	$9.8x10^{10}$	14 µm [h] x 400 µm [v] / 3mm [v] - mirror in
40	3.9	1.95	$3.7x10^{11}$	14 µm [h] x 400 µm [v] / 3mm [v] - mirror in
60	2.6	1.3	$6.7x10^{10}$	14 µm [h] x 5mm [v]
80	1.9	0.95	$1.0x10^{10}$	14 µm [h] x 5mm [v]
100	1.5	0.75	$1.3x10^{9}$	14 µm [h] x 5mm [v]

Beamline Layout

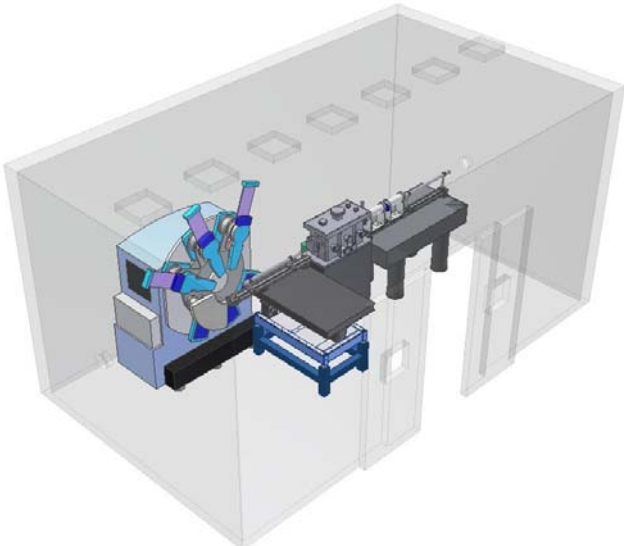
<u>Key Points:</u> Power loads are critical!!!
Attenutor assembly: absorb as much power as reasonable possible to guarantee a high performance stable beamline.

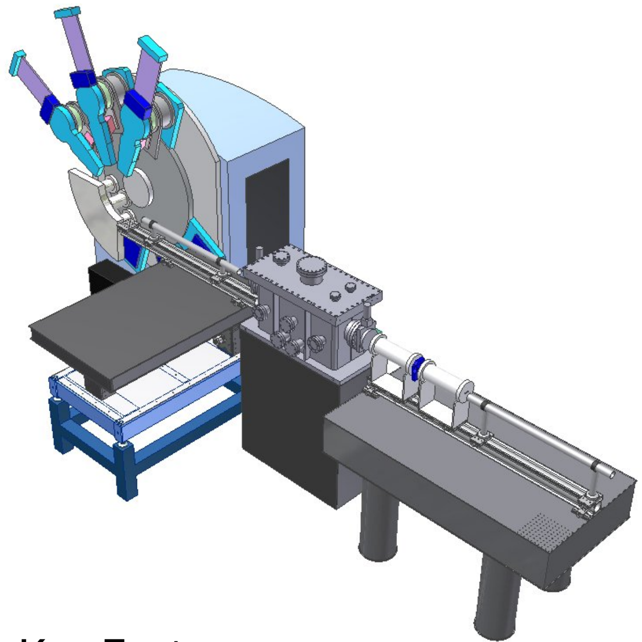


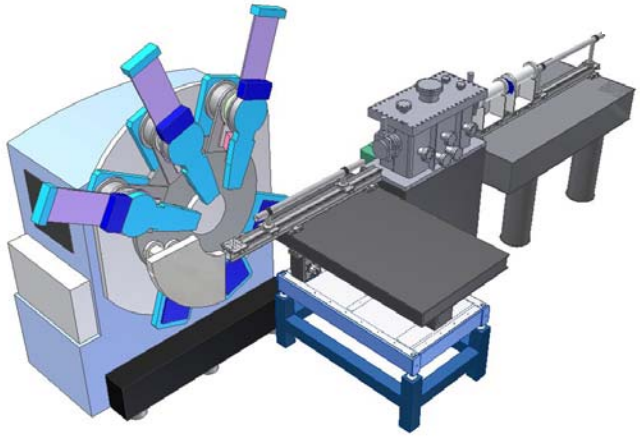
Office of Science
U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

Experimental Endstation 1 - Back Hutch







Key Features

High resolution diffractometer (in use in Swiss LS and Australian Light Source)

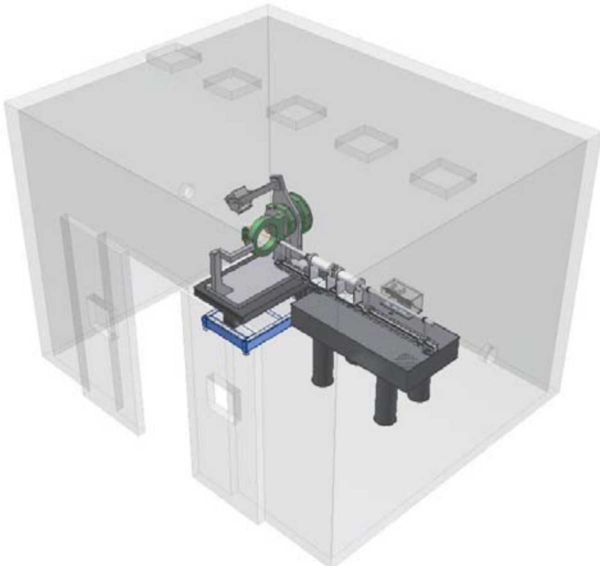
Graded Multilayer Focusing

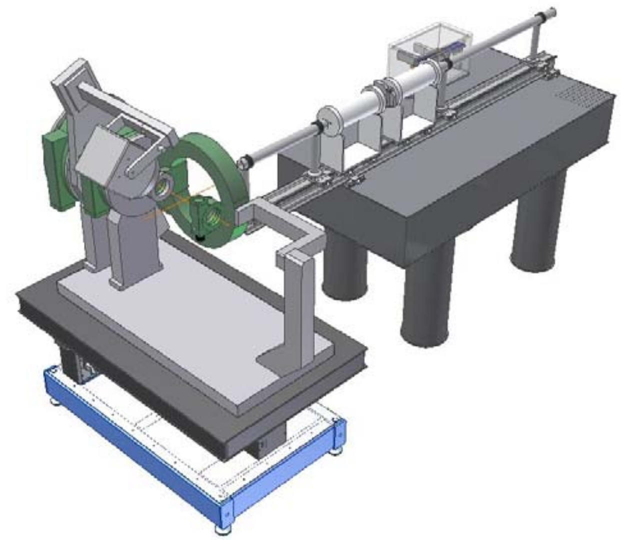
Robot Sample Changer * not shown

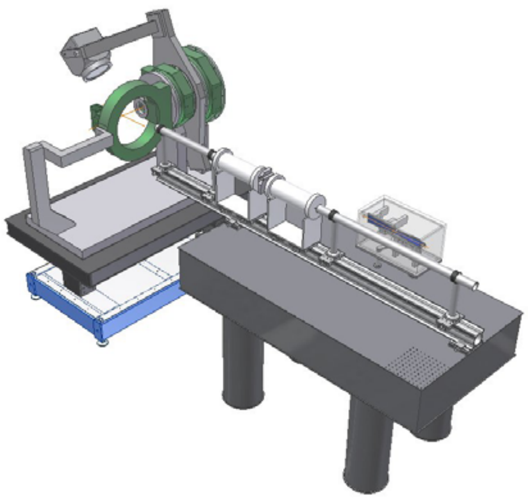
Laue Analyszer Crystals

7000 element Silicon Strip Detector, fast read out (msec)

Experimental Endstation 2 - Front Hutch







Enclosure costed, equipment to be used from NSLS-I

Key Features

CCD area detector

Graded Multilayer Focusing * - not shown

Robot Sample Changer * not shown

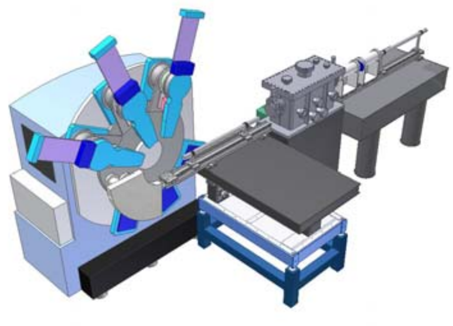
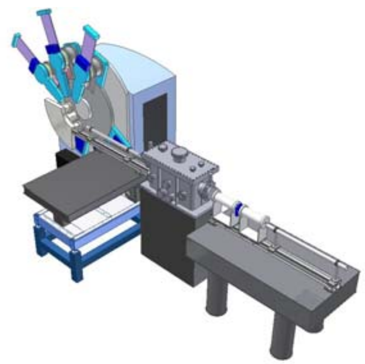
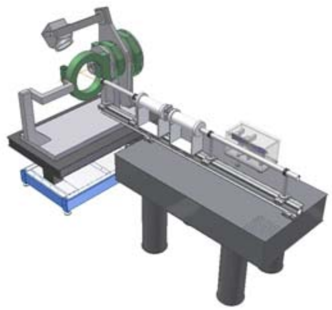
Outstanding Issues

- Canting will open up more endstations from 2 Damping Wigglers in the high-ß straight section. Would be advantageous to increasing number of users. Also, power load issues would reduce by a factor of ~ 2.
- Thermal heat loads seem tollerable but R&D effort needed -Filters, Mirrors and Laue Mono.
- R&D required on long graded bent multilayers.

Cost Estimate

Component	1.04.05.01 Undulator Beamline 1 Inelastic X-ray Scattering	1.04.05.02 Undulator Beamline 2 Hard X-ray Nanoprobe	1.04.05.03 Undulator Beamline 3 - Hard X-ray Coherent Scattering	1.04.05.04 Undulator Beamline 4 - Soft X-ray Coherent Scattering	1.04.05.05 Damping Wiggler Beamline 1 - XAS	1.04.05.06 Damping Wiggler Beamline 2 - Powder Diffraction	**Burdened Grand Total**
Enclosures	$ 1,150,960.00	$ 1,489,445.00	$ 1,583,317.00	$ 174,598.00	$ 1,212,499.00	$ 1,472,787.00	**$ 7,083,606.00**
Beam Transport	$ 685,166.00	$ 153,670.00	$ 1,565,088.00	$ 206,807.00	$ 409,495.00	$ 375,756.00	**$ 3,395,982.00**
Utilities	$ 335,616.00	$ 505,074.00	$ 316,364.00	$ 227,774.00	$ 227,668.00	$ 227,668.00	**$ 1,840,164.00**
High Heatload Optics	$ 976,463.00	$ 1,536,974.00			$ 2,127,744.00	$ 1,188,811.00	**$ 5,829,992.00**
Beam Conditioning Optics	$ 1,921,140.00	$ 2,290,227.00	$ 1,989,884.00		$ 1,583,678.00	$ 871,612.00	**$ 8,656,541.00**
Personnel Safety System	$ 183,352.00	$ 187,537.00	$ 278,181.00	$ 96,894.00	$ 183,352.00	$ 183,352.00	**$ 1,112,668.00**
Equipment Protection System	$ 86,262.00	$ 86,262.00	$ 86,038.00	$ 140,144.00	$ 86,038.00	$ 86,545.00	**$ 571,289.00**
White Beam Apertures				$ 262,817.00			**$ 262,817.00**
White Beam Components		$ 302,131.00	$ 1,278,815.00				**$ 1,580,946.00**
End Station 1	$ 2,522,167.00	$ 4,241,913.00	$ 3,509,501.00	$ 1,869,898.00	$ 752,988.00	$ 3,018,162.00	**$ 15,914,629.00**
End Station 2			$ 3,328,590.00		$ 1,967,186.00		**$ 5,295,776.00**
Beamline Controls	$ 674,461.00	$ 504,235.00	$ 386,610.00	$ 120,756.00	$ 147,727.00	$ 100,351.00	**$ 1,934,140.00**
Beamline Control Station	$ 35,686.00	$ 35,686.00	$ 35,686.00	$ 35,686.00	$ 38,902.00	$ 35,686.00	**$ 217,332.00**
Satellite Building		$ 1,025,465.00	$ 1,392,908.00				**$ 2,418,373.00**
Beamline Management	$ 1,368,930.00	$ 1,574,258.00	$ 1,529,692.00	$ 1,261,295.00	$ 1,462,293.00	$ 1,624,346.00	**$ 8,820,814.00**
Branching Mirror				$ 598,408.00			**$ 598,408.00**
Exit Slits				$ 1,102,562.00			**$ 1,102,562.00**
First Mirrors (m0 and M1)				$ 1,111,344.00			**$ 1,111,344.00**
Monochromator (m2 + gratings)				$ 1,565,745.00			**$ 1,565,745.00**
Polarization Selection Components				$ 243,890.00			**$ 243,890.00**
Refocusing Mirror				$ 2,730,113.00			**$ 2,730,113.00**
Specialized White Beam Comp					$ 472,353.00	$ 351,800.00	**$ 824,153.00**
Grand Total	**$ 9,940,203.00**	**$ 13,932,877.00**	**$ 17,280,674.00**	**$ 11,748,731.00**	**$ 10,671,923.00**	**$ 9,536,876.00**	**$ 73,111,284.00**

Summary

The proposed powder diffractometer at NSLS-II will be the US' only high-resolution instrument capable of collecting data at high energies (20 keV to 100 keV). This will make it ideal for *in situ* and time resolved studies of samples held in environmental cells.