Surface and Interface Scattering at NSLS-II

Karl Ludwig
Boston University
(*Thanks to C. Nelson, Q. Shen, S. Hulbert, A. Fluerasu, R. Headrick… for slides*)
XIS Workshop – January 10, 2012

U.S. DEPARTMENT OF ENERGY

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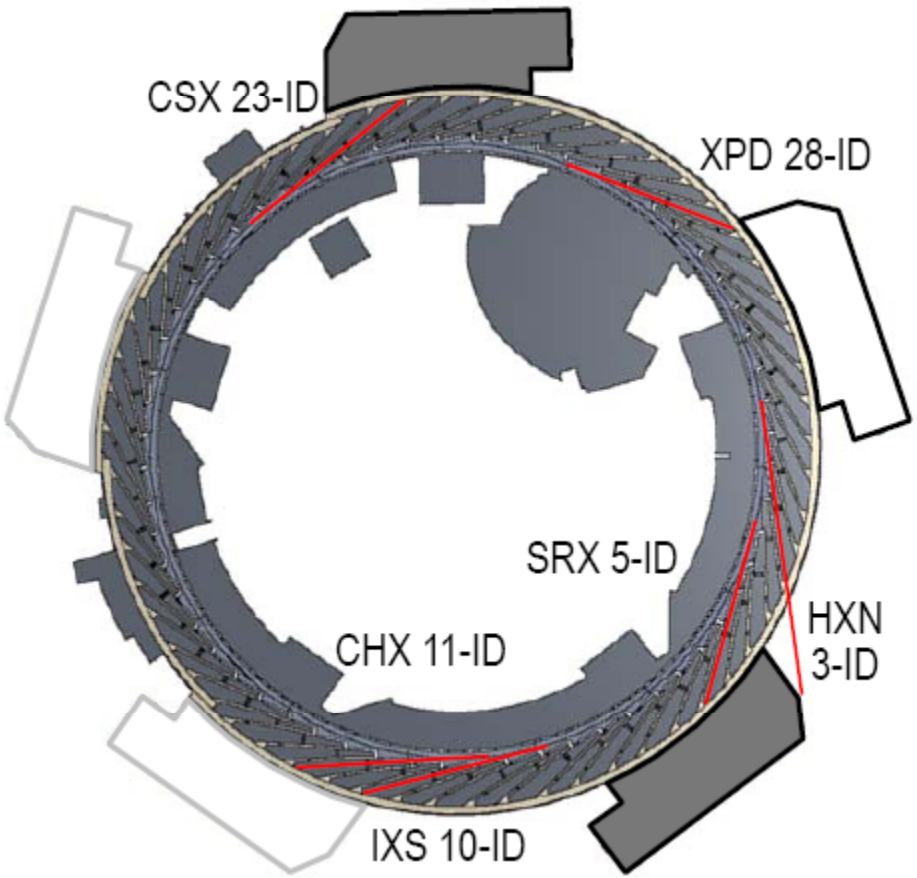
Surface and Interface Scattering at NSLS-II

- Basic NSLS-II Machine Parameters
- "Project" Beamlines at NSLS-II
- "NEXT" and "NxtGen" Beamlines
- Beamline for Integrated *In-Situ* and *Resonant* X-ray Studies (ISR)

Basic NSLS-II Machine Parameters

- Ring Energy: 3 GeV
- Current: 500 mA
- Ring Circumference: 792 m
- 30 straight sections (high-β 9.3m/ high-β 6.6m)
- Vertical Emittance: 8 pm-rad
- Horizontal Emittance: 550 pm-rad
- Average spectral brightness of 10^{21} ph/s/0.1%BW/mm^2/mrad^2 from 2-10 keV

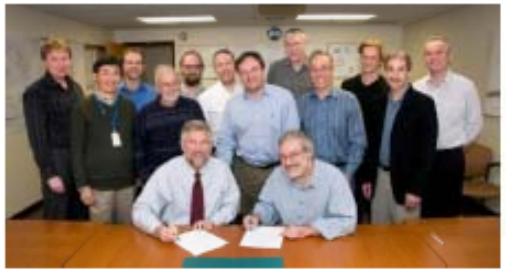
Six "Project" BL's Approved as Part of Original NSLS-II Project



- Inelastic X-ray Scattering (IXS)
- Hard X-ray Nanoprobe (HXN)
- Coherent Hard X-ray Scattering (CHX)
- Coherent Soft X-ray Scattering & Polarization (CSX)
- Sub-micron Resolution X-ray Spectroscopy (SRX)
- X-ray Powder Diffraction (XPD)

Beamline Advisory Team (BAT) Concept

- All beamlines at NSLS-II are developed with participations of BATs
  - Representing a specific segment of scientific community
  - Advising NSLS-II on scientific mission & technical programs at beamline
  - Working closely with BL group during design, construction, commissioning, early operations
  - Participating in beamline design meetings, reviews, and commissioning experiments

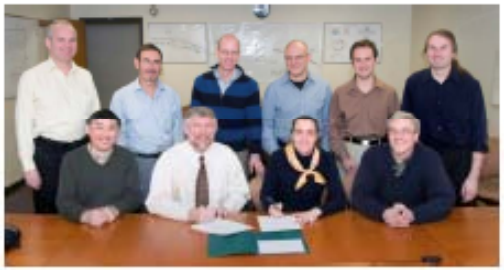


SRX BAT

XPD BAT

IXS BAT

CHX BAT



CSX BAT

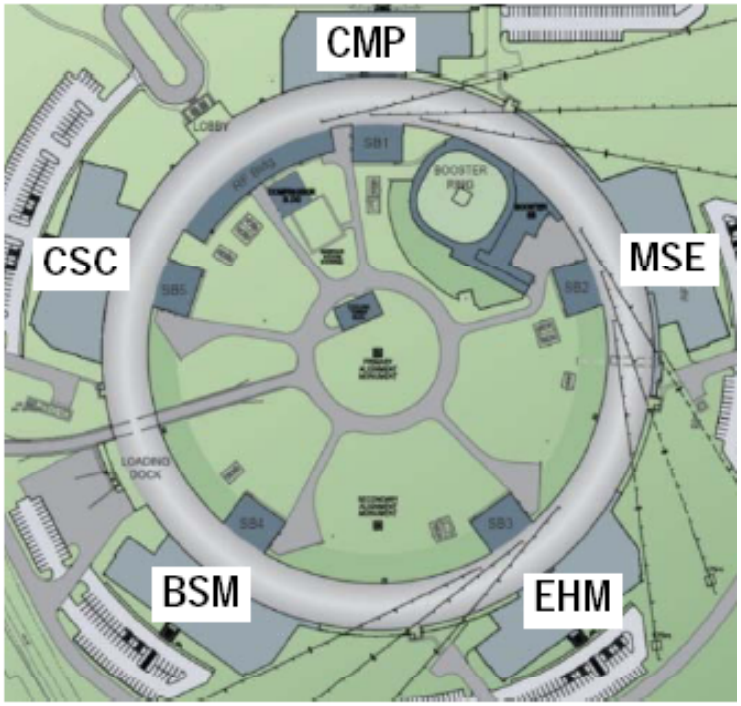
HXN BAT

Beamline Development Projects

- Beyond the 6 Project Beamlines
  - NSLS-II Experimental Tools (NEXT) Project
    - 6 insertion device beamlines
    - BES funded MIE project
    - Expand (double) BES beamline portfolio
    - Scientific program complementary to Project Beamline programs
  - NIH beamline projects
    - 3 insertion device beamlines
    - NIH funded
    - Initial biological sciences beamline portfolio
  - NxtGen Beamline Development
    - Up to 16 bend magnet and three-pole wiggler beamlines
    - To be funded by NSLS-II Project contingency, NSLS-II operations funding, and other sources to be identified
    - If fully built out, will double the number of operating beamlines at NSLS-II

Science Village Concept

- Science Village concept has been widely advocated by the scientific community. Photon Science Directorate strongly supports this concept and plans to co-locate beamlines likely to be utilized by a specific scientific community, after satisfying physical constraints
- These co-located beamlines, along with nearby Laboratory Office Buildings (LOB), serve as 'hubs' for Science Villages distributed around the ring
- LOB in a Village may house ancillary instruments to provide off-line experimental and analysis capabilities for the community, & serve as primary place to mingle with fellow colleagues & co-workers
- Staff and users in a Village may share beamline instrumentation and learn from each other's experience and expertise
- Possible five Science Villages based on 6 project and 34 approved beamlines:
  - Condensed matter & materials physics (CMP)
  - Materials science & engineering (MSE)
  - Environmental & heterogeneous materials science (EHM)
  - Biology and soft matter science (BSM)
  - Chemical science and catalysis (CSC)



NEXT Beamlines

ESM -- Electron Spectro-Microscopy

FXI -- Full-field X-ray Imaging from µm to nm

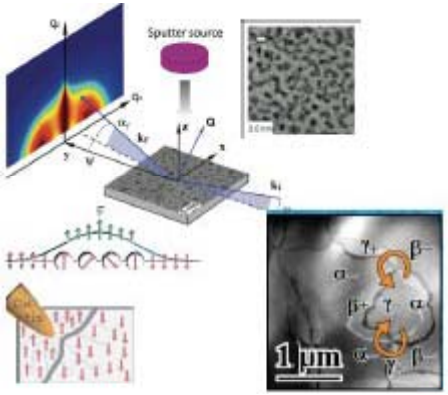
ISS -- Inner Shell Spectroscopy

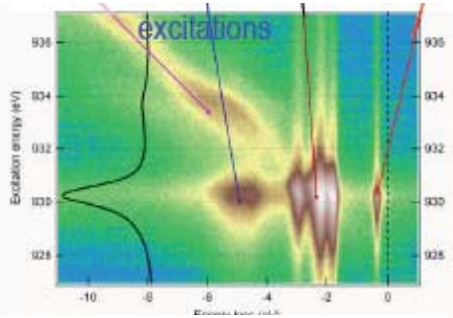
ISR -- Integrated In-Situ & Resonant X-Ray Studies

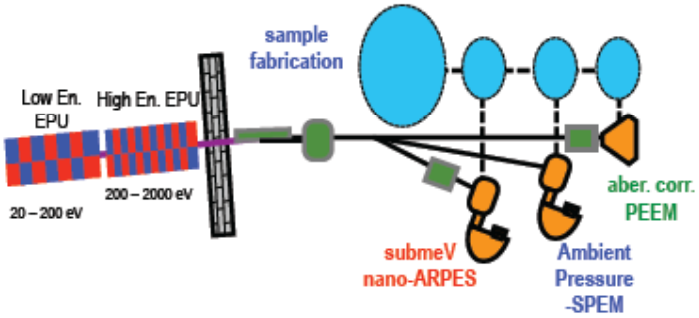
SIX -- Soft Inelastic X-ray Scattering

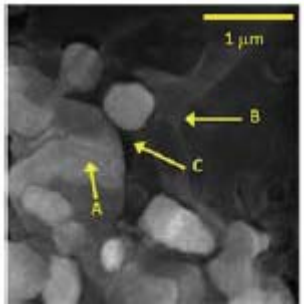
SMI -- Soft Matter Interfaces

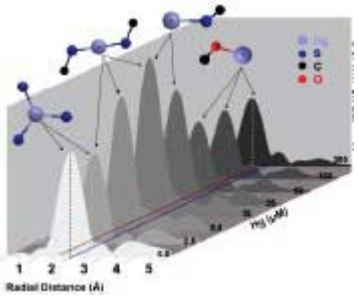
- Steve Hulbert named interim Beamline Project Manager

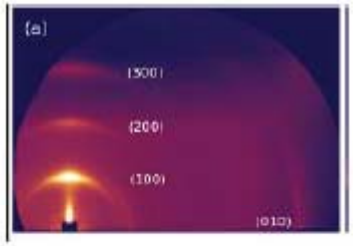












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NEXT Project (*Interim*) Management Team

WBS 2.0

NEXT Beamlines

S. Hulbert
Sources & Optics Interface manager -- R. Reininger
ES&H -- S. Hoey
QA -- C. Porretto
Project Controls – E. Abela, J. O'Connor

PSD Science Advisory Committee

NSLS-II Project Advisory Committee

2.01

NEXT Beamlines Project Management

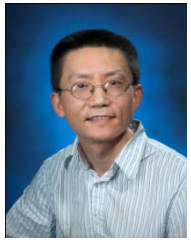
S. Hulbert

2.02 Conceptual Design and Analysis

R. Reininger

2.03 Common Beamline Systems

A. Broadbent



2.04.01

ESM: Electron Spectro-Microscopy

E. Vescovo

2.04.02

FXI: Full-field X-ray Imaging from Microns to Nanometers

Z. Zhong

2.04.03

ISR: Integrated In-Situ and Resonant X-ray Studies

C. Nelson

2.04.04

ISS: Inner Shell Spectroscopy

L. Berman

2.04.05

SIX: Soft Inelastic X-ray Scattering

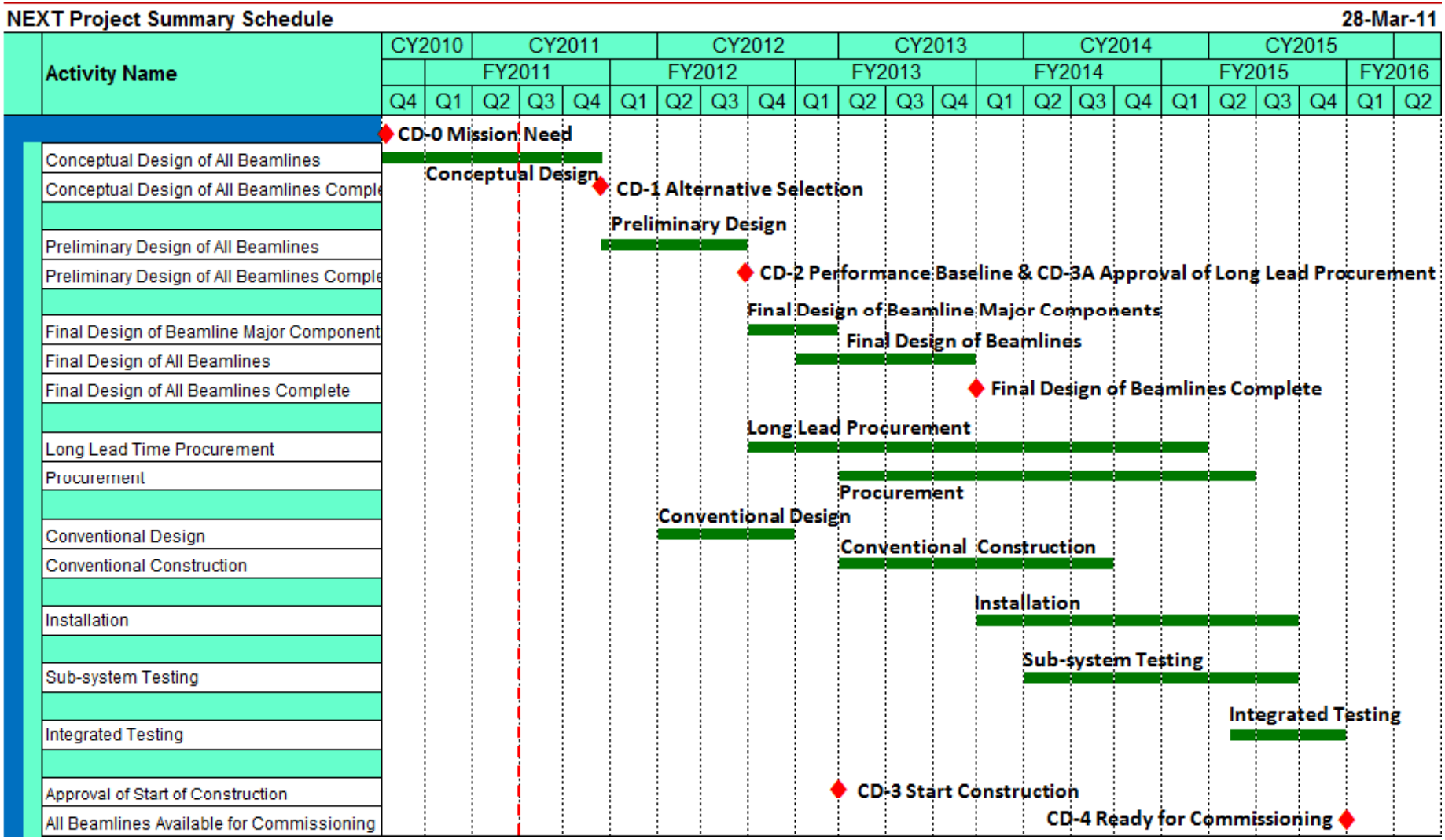
J. Dvorak

2.04.06

SMI: Soft Matter Interfaces

L. Wiegart

NEXT Beamline Project Proposed Timeline



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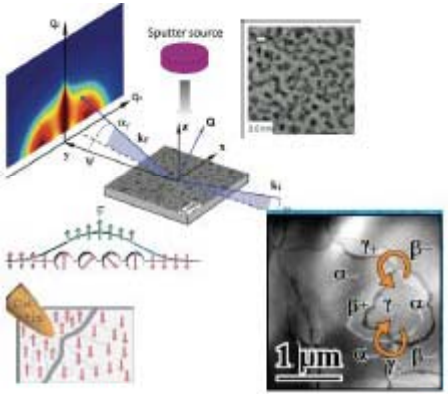

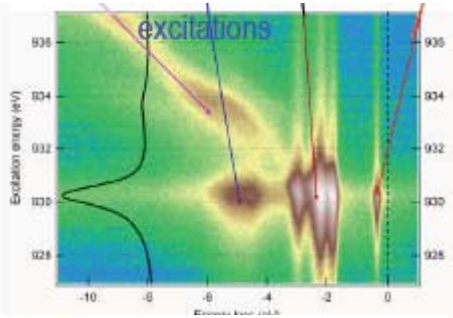

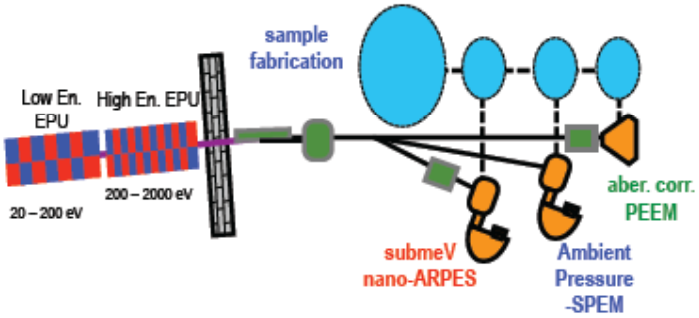

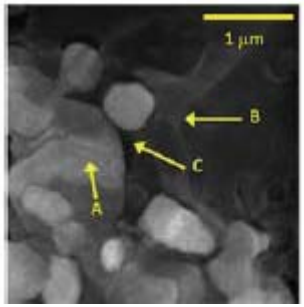

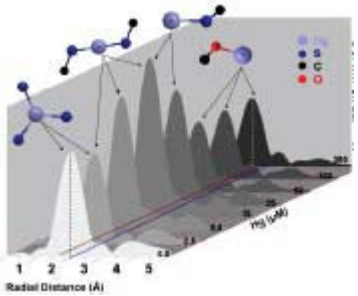

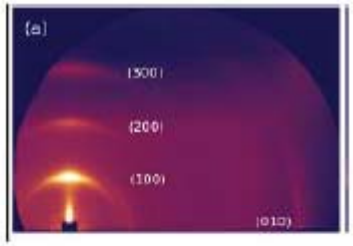

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BDP Team and Advisors

Team:

Karl Ludwig, Spokesperson (Prof. of Physics, Boston Univ.)— real-time studies, XPCS

Roy Clarke (Prof. of Physics, Univ. of MI)— time-resolved studies, direct method studies of interface structure

Andrei Fluerasu (Assoc. Physicist, PSD, BNL)— XPCS; group leader for NSLS-II CHX beamline

Randall Headrick (Assoc. Prof. of Physics, Univ. of VT)— real-time studies

John Hill (Physicist, CMPMSD, BNL)— RXS; group leader for BNL X-ray Scattering Group

Jean Jordan-Sweet (Research Staff Member, IBM)— microelectronics materials, diffraction techniques

Valery Kiryukhin (Assoc. Prof. of Physics, Rutgers Univ.)— RXS, scattering in high-**H**

Paul Lyman (Prof. of Physics, Univ. of WI-Milwaukee)— atomic structure of surfaces, CTR

Christie Nelson (Physicist, PSD, BNL)— RXS, scattering in high-**H**

Ron Pindak (Senior Scientist, PSD, BNL)— RXS, CTR phase retrieval; Chem. and Phys. Sciences Division Head

Advisors:

Joel Brock (Cornell University)

Jonathan Lang (APS, ANL)

Oleg Shpyrko (UCSD)

Phil Willmott (SLS, PSI)

Ken Evans-Lutterodt (PSD, BNL)

Vishy Ravindranath (PSD, BNL)

Jörg Strempfer (PETRA III, DESY)

Arthur Woll (CHESS)

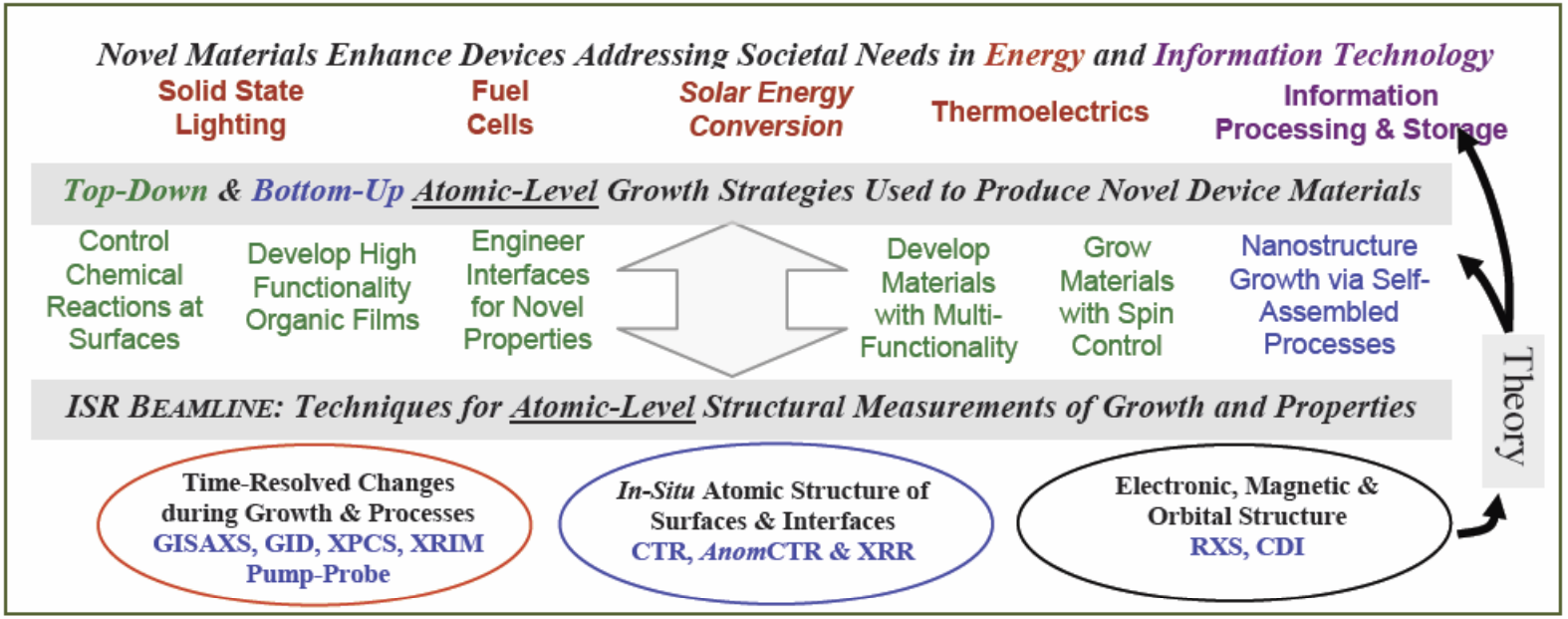
Mourad Idir (PSD, BNL)

Ruben Reininger (PSD, BNL)

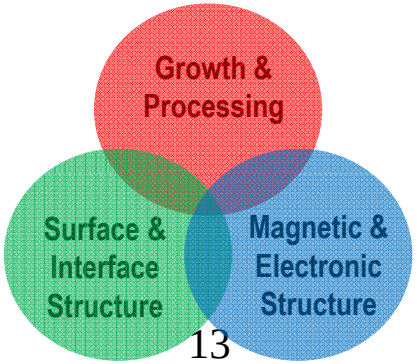
Oleg Tchoubar (PSD, BNL)

Paul Zschack (APS, ANL)

ISR Scientific Scope Overview



Addressing these complex issues and materials requires sophisticated investigations with **not just a single approach**, but rather with a *flexible* and *broad range of capabilities* that can be utilized *synergistically*.



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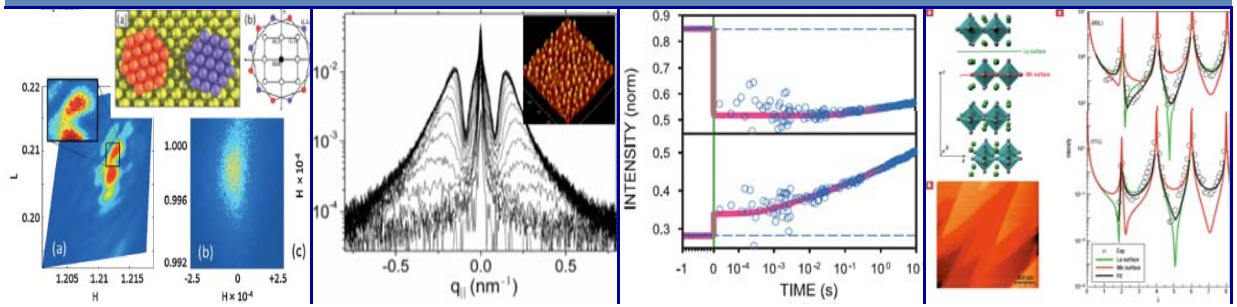
Scientific Scope Overview

Surface and Thin-Film Growth Processes

Surface XPCS	Time-resolved GISAXS	Time-resolved surface scattering	Orbital ordering truncation rod



Pierce *et al.* PRL 2009 | Ozaydin *et al.* 2009 | Tischler *et al.* PRL 2006 | Wakabayashi *et al.* Nature Materials 2007

Functional Surfaces and Interfaces

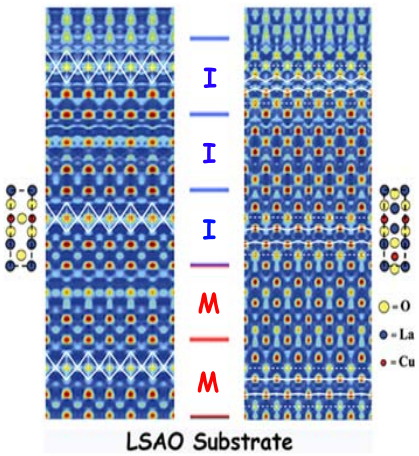
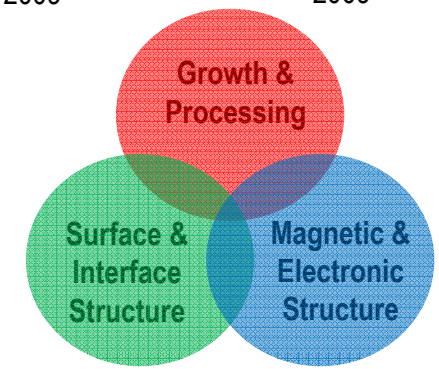
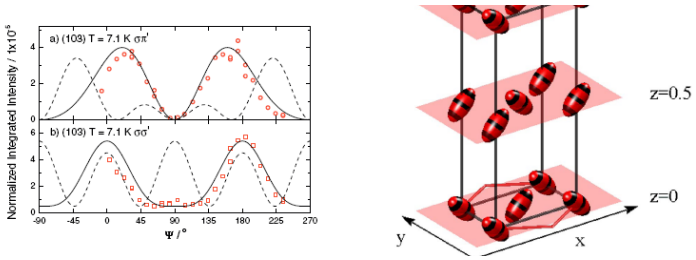


Figure. 7



Electronic densities in M-I junction with interfacial superconductivity, Zhou *et al.*, PNAS **107**, 8103 (2010)

Correlated Electron Materials



Anti-ferro-quadrupolar order in UPd_3, Walker *et al.*, PRL **97**, 137203 (2006)

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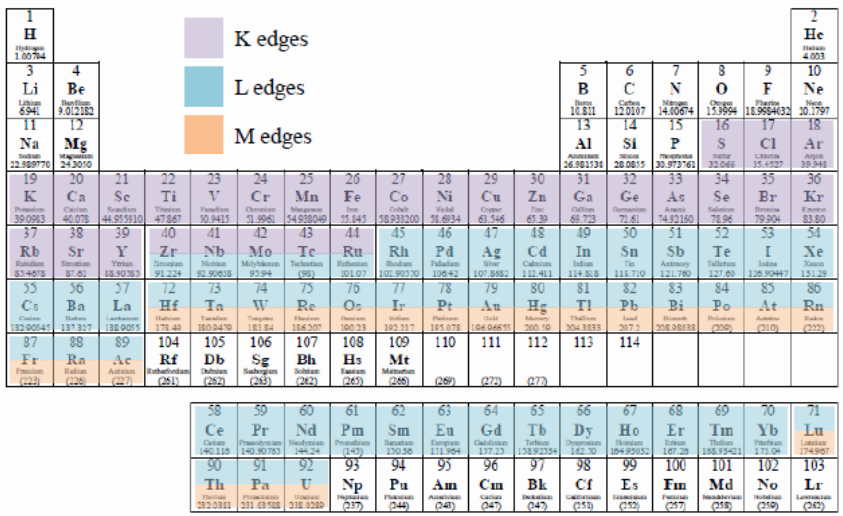
Technical Requirements

- Resonant scattering with energies 2.4-23 keV, and variable energy resolution
- Controllable incident polarization and polarization analysis
- Microfocusing with large (>1 m) working distance
- Highly flexible range of sample environments
  - large UHV chambers with different deposition and processing techniques
  - large (>10 T) magnet compatible with x-ray scattering
  - variable temperatures
- On-the-fly scanning, large area detectors
- Planned canted build-out

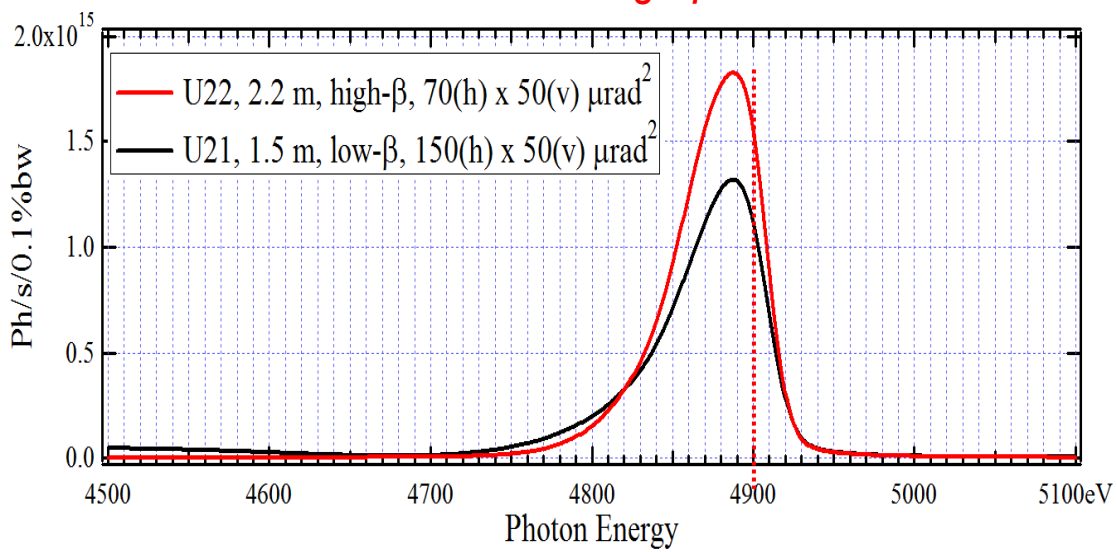


Insertion Device Optimization

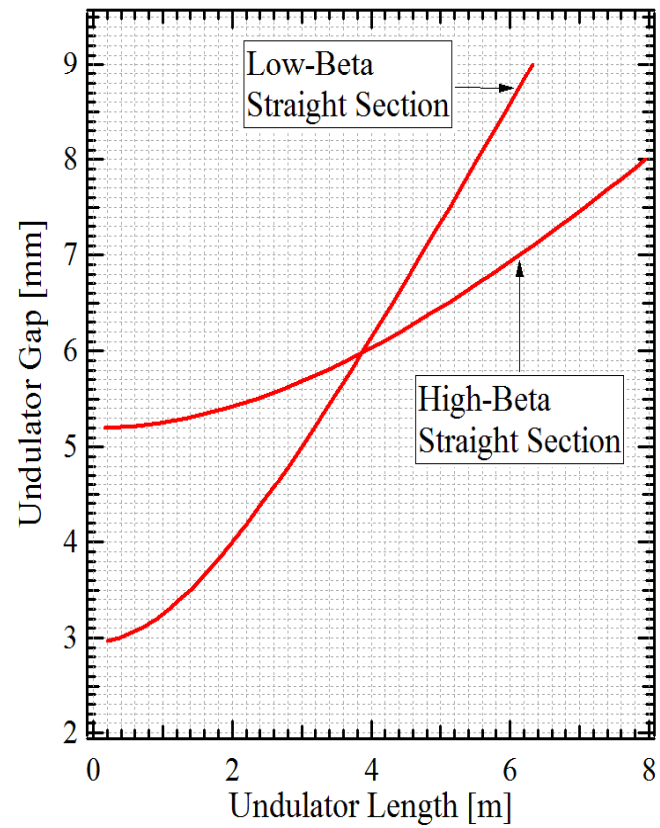
Requirements:

- Highest flux over entire 2.4-23 keV energy range
- Low divergence
- Future canting
- Third harmonic starting below Ti K edge

Low- vs. High-β



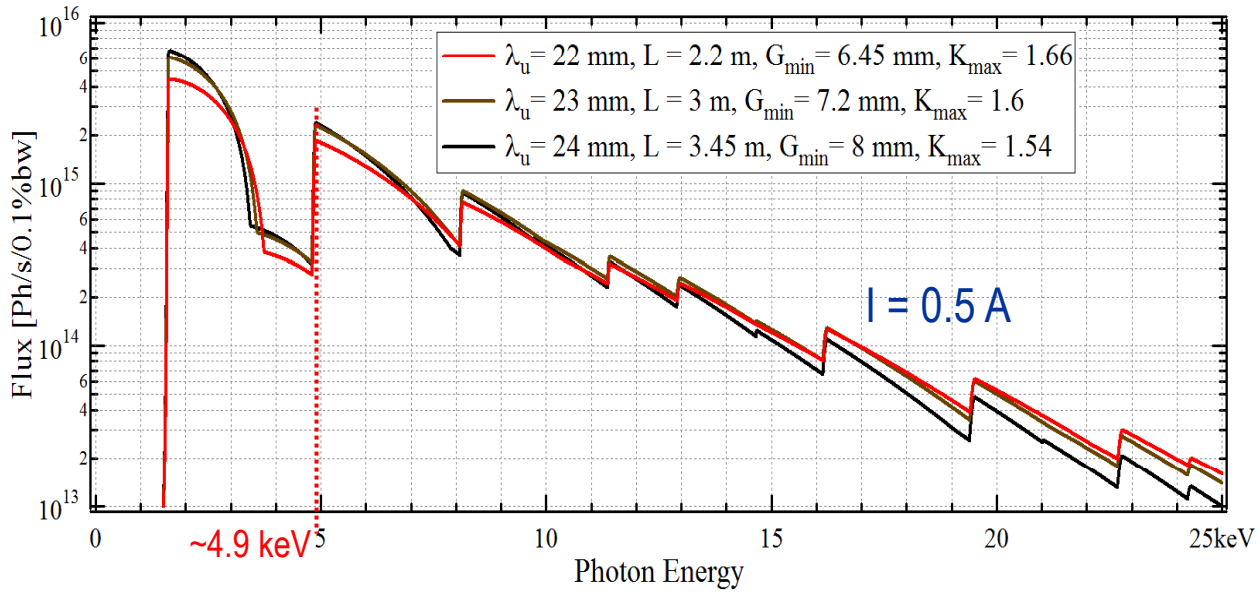
Stay-clear Constraints



Courtesy Oleg Tchoubar

Insertion Device Optimization

ISR Beamline (one of two Canted IVU in High-β Straight; 70 H x 50 V μrad² Ap.)

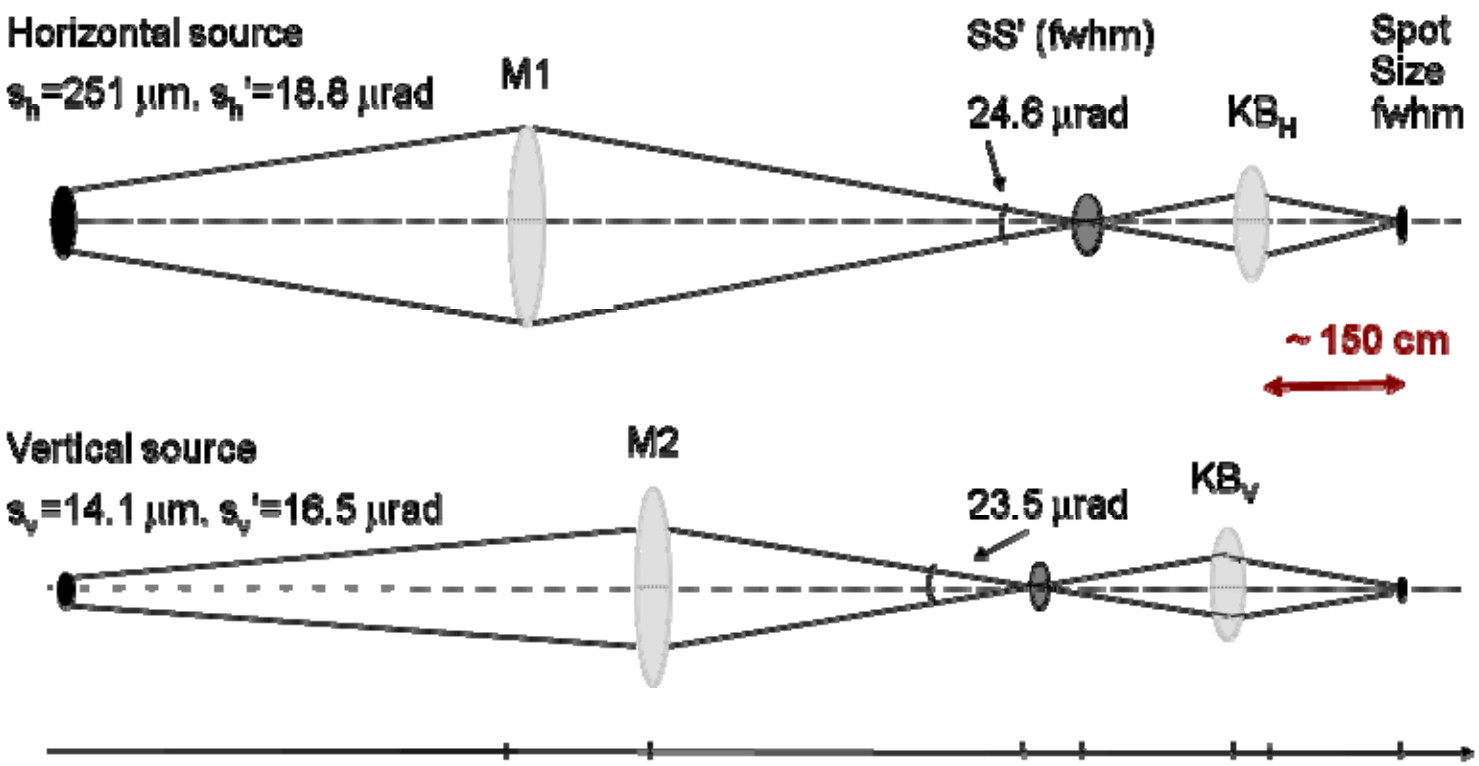


Courtesy
Oleg Tchoubar

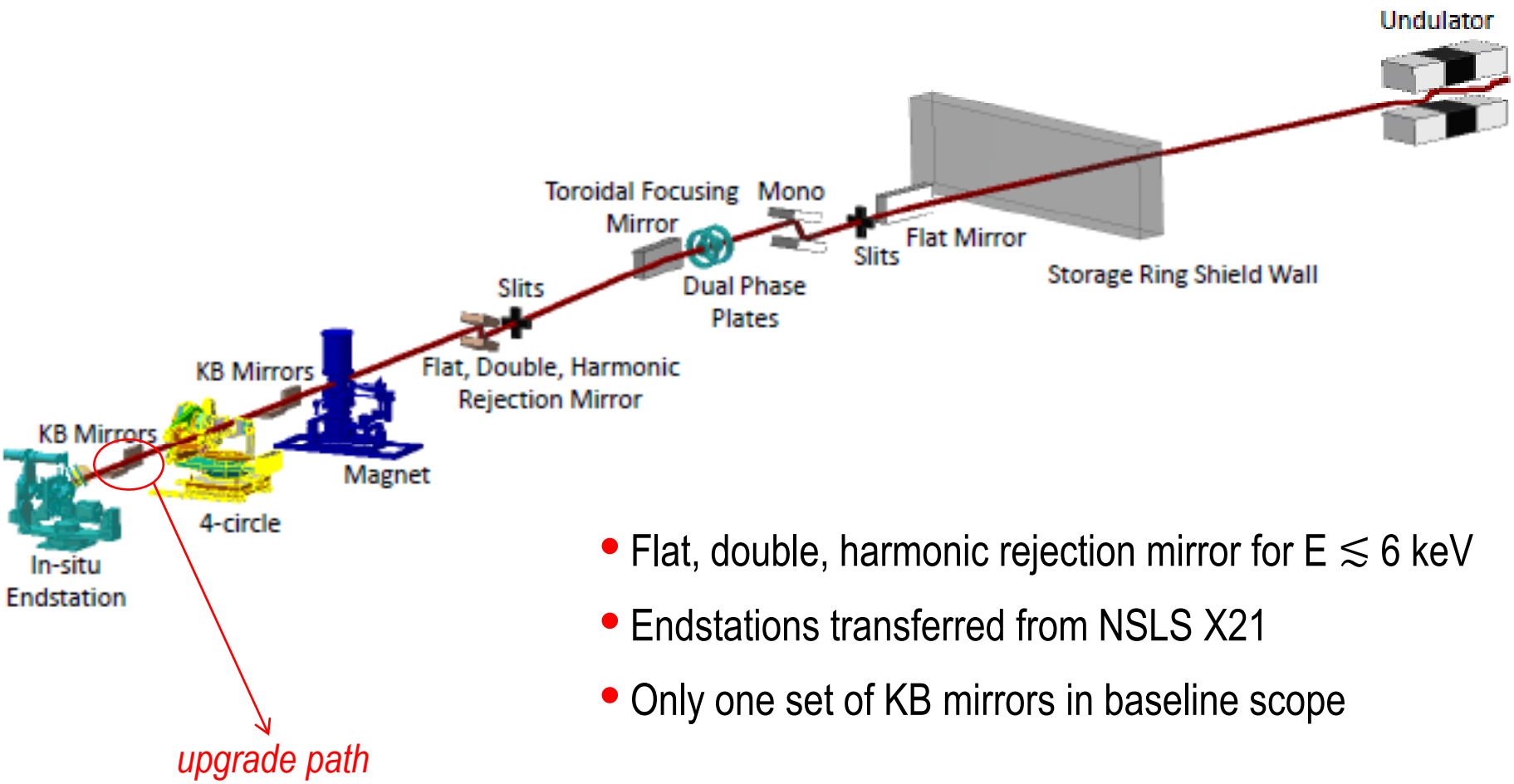
→ 3 m long IVU-23 preferred

	3 keV	10 keV	20 keV
flux in Si(111) bandpass	4×10^{14} ph/s	6×10^{13} ph/s	7×10^{12} ph/s
coherent flux in Si(111) bandpass	4×10^{12} ph/s	3×10^{11} ph/s	1×10^{10} ph/s

Focusing Scheme



Beamline Schematic Layout



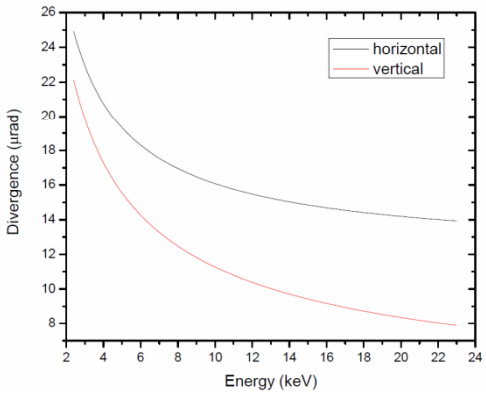
- Flat, double, harmonic rejection mirror for E $\lesssim$ 6 keV
- Endstations transferred from NSLS X21
- Only one set of KB mirrors in baseline scope

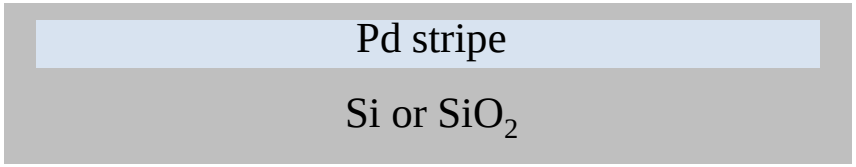
Horizontally-Deflecting Flat Mirror

Purpose:

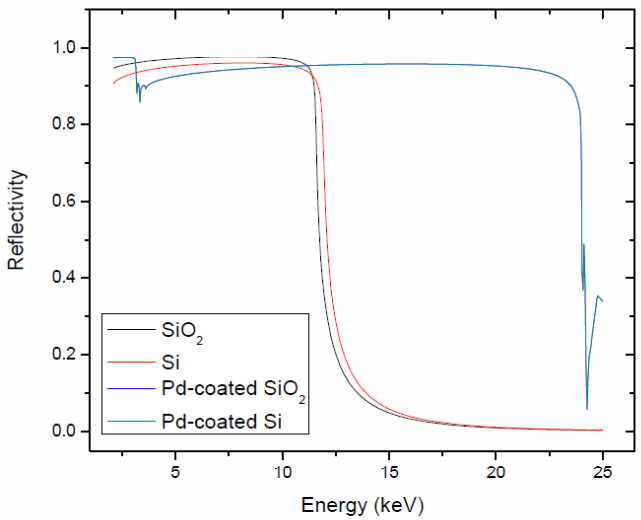
- Increase beam separation for future canted (2 mrad) beamline
- Deflect beam out of Bremsstrahlung
- Remove some (~10%) power
- Maintain small, nearly isotropic beam divergence
- Provide harmonic rejection for E $\gtrsim$ 6 keV

Beam Divergence (3 m ID, high-β)





0.15° Incidence Angle

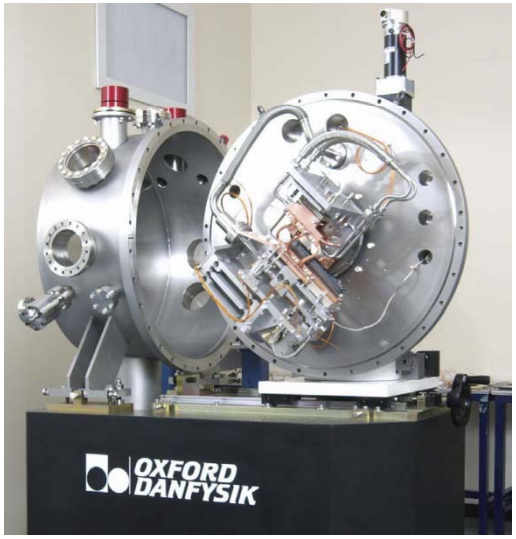


Double-Crystal Monochromator

Purpose:

- Provide high ($\Delta E/E \sim 10^{-4}$-10^{-5}) energy resolution for 2.4-23 keV energy range
- Minimize negative effects on beam coherence
- *Provide low ($\Delta E/E \sim 10^{-2}$) energy resolution for ~6-12 keV energy range (upgrade path)*

Cryocooled DCM (at NSLS X9)



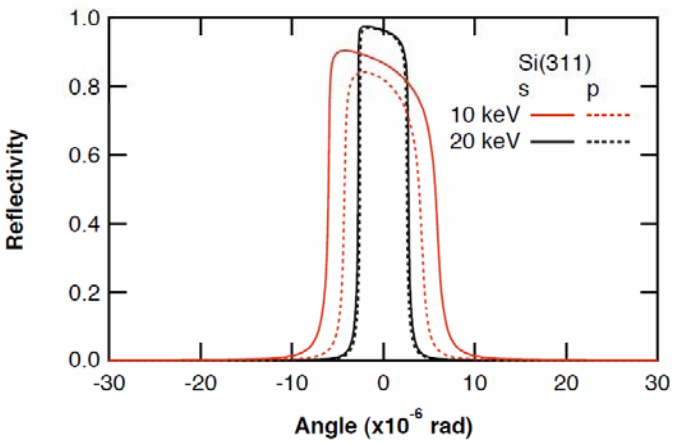
Approach:

→ mount Si(111) and Si(311) crystal pairs side-by-side

→ use angular range of 5-55° (E ≥ 4.6 keV for Si(311))

→ *install separate ML mono (upgrade path)*

Si(311) Reflectivity



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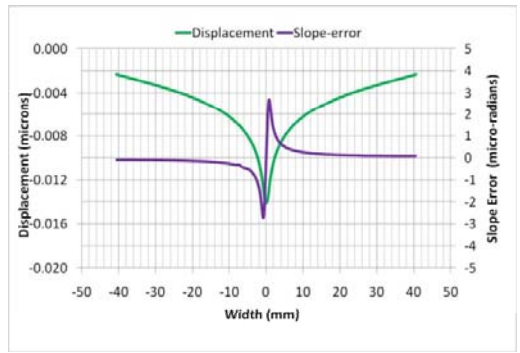
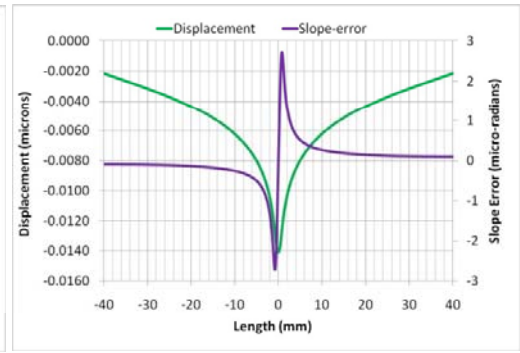
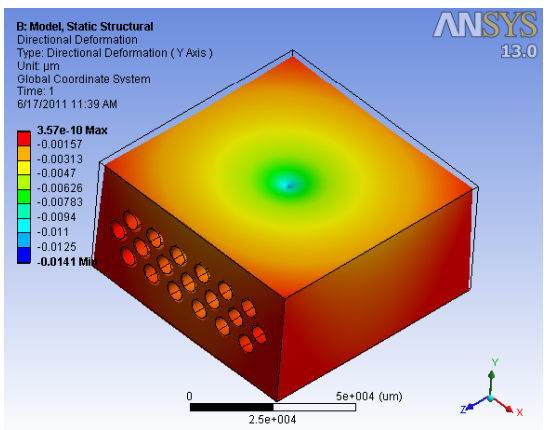
Double-Crystal Monochromator: Heat Load

Finite element analysis calculations:

- at 2.4 and 4.9 keV
- with direct cooling of monochromator
- with 1 mm x 1 mm beam cross-section at 28 meters







At 2.4 keV:

→ peak temperature of 90.8 K

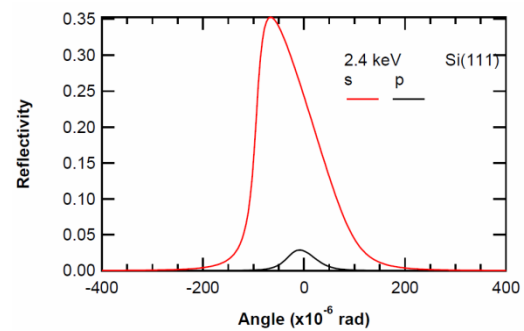
→ peak displacement of -0.014 μm

→ slope errors of ±3 μrad

→ peak power density 20 W/mm^2

→ total deposited power of 30 W

Si(111) Reflectivity



Courtesy Vishy Ravindranath

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Dual Phase Plate Assembly

Purpose:

provide polarization (circular and variable linear) control over 2.4-13.5 keV energy range

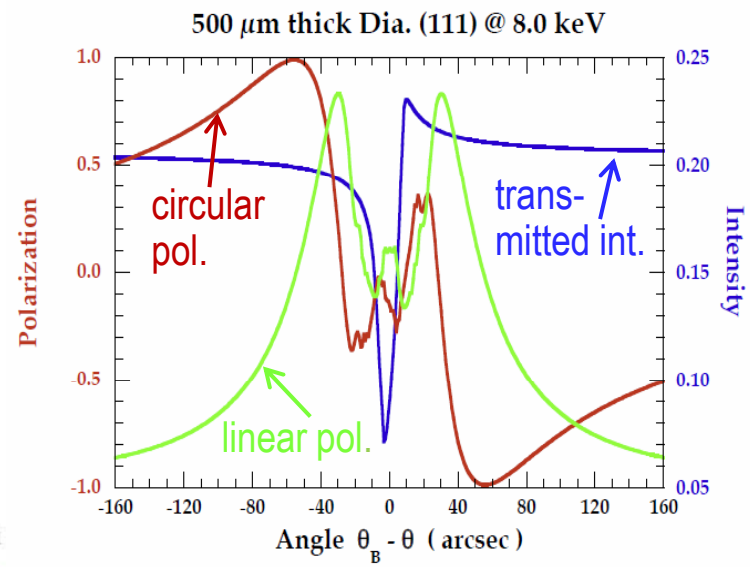
Approach:

→ use dual phase plate assembly

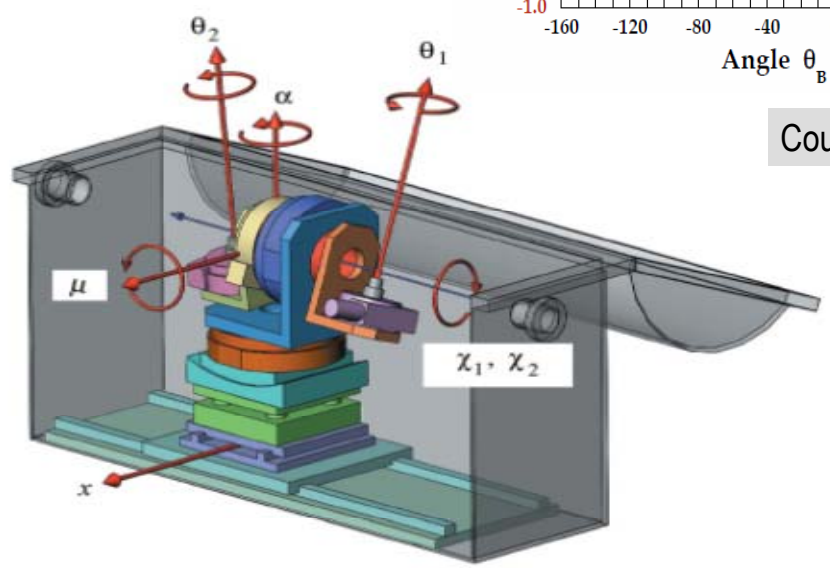
→ use diamond(111) transmission phase plates for E = 3.5-10 keV

→ use diamond(220) transmission phase plates for E = 10-13.5 keV

→ begin development work on Si(111) phase plates for E = 2.4-3.5 keV

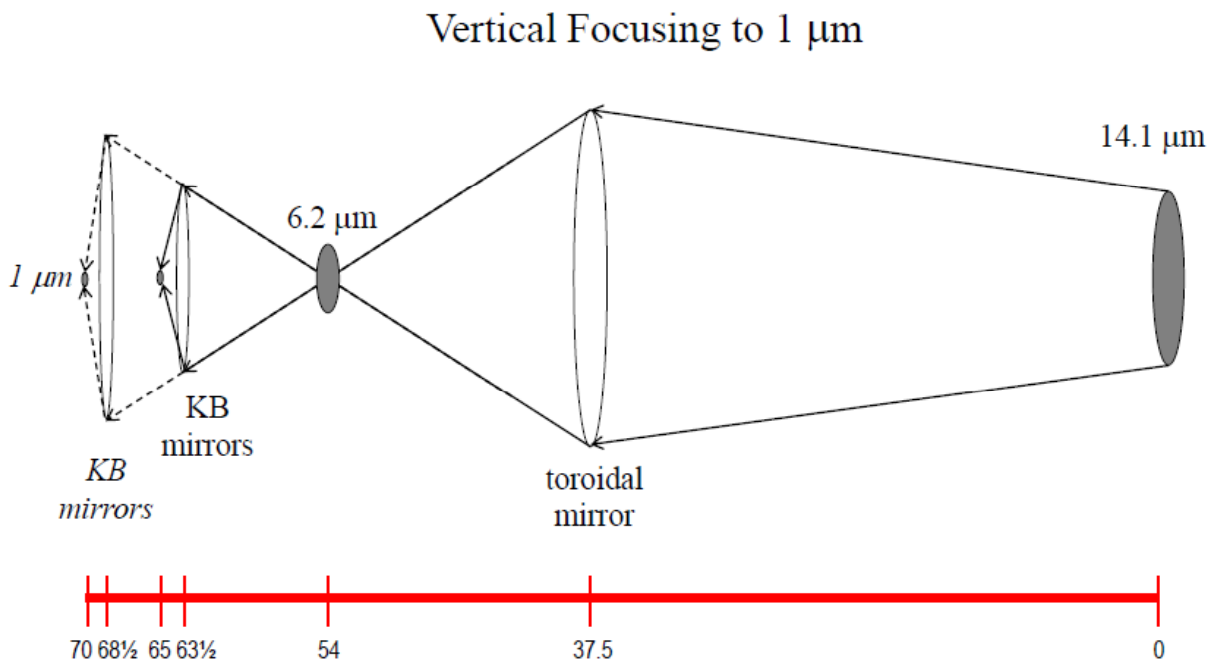


Courtesy Jonathan Lang



from Scagnoli *et al., J. Synch. Rad.* **16**, 778 (2009)

Focusing Scheme



	V x H (μm²) FWHM
13 Tesla Magnet and Diffractometer	300 x 400
4-Circle Diffractometer	1 x 20
Large UHV Chambers and Base Diffractometer	4 x 70
Large UHV Chambers and Base Diffractometer	<1 x 10

Spot Sizes at Various Endstations with Perfect Mirrors

upgrade path with second set of KB mirrors

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Hutch A (front): 13 Tesla Magnet and Diffractometer

superconducting, 13 T, vertical field magnet

NSLS X21

analyzer

detector (APD or CdTe diode)

x, y, and z translation stages

2 sets of arcs

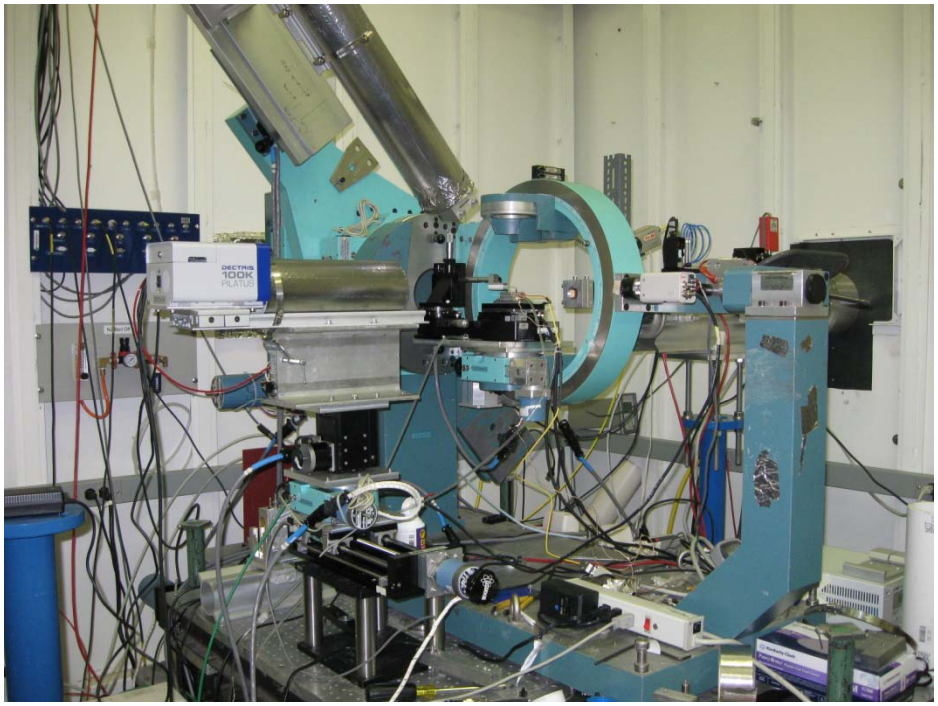
2-circle (θ-2θ)

- H: 0-13 Tesla
- T: 1.8-300 Kelvin
- X-ray energies $\gtrsim$ 6 keV
- Mounted on rails for easy endstation changeover

Replace magnet in upgrade path.

Hutch A (back): 4-circle Diffractometer

NSLS X21



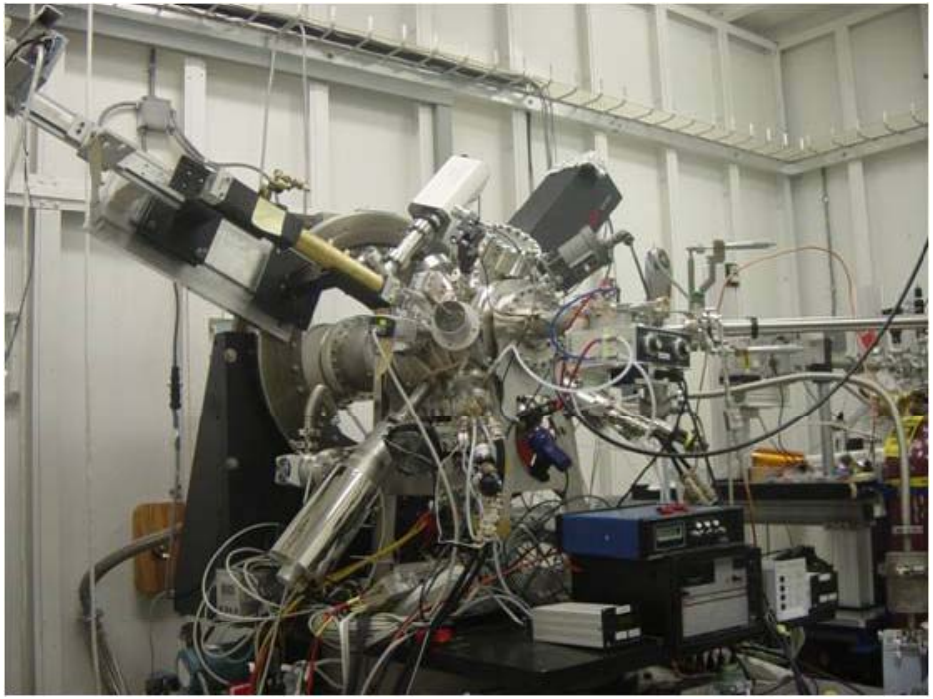
Additional endstation equipment from X21:

- Displex cryostat (temperature range of 6.5-350 K)
- Pilatus and ADSC area detectors (shared with X20)
- Vortex detector

Replace 4-circle with 6-circle in upgrade path. Also, buy motorized displex mount and in-vacuum polarization analyzer.

Hutch B: Large UHV Chambers and Base Diffractometer

NSLS X21



Courtesy Karl Ludwig and Randy Headrick

- Custom diffractometer to accommodate interchangeable UHV growth and processing chambers for *in-situ* studies
- Tender x-ray compatible
- Vibrations minimized for micro-diffraction and coherent scattering

Upgrade base diffractometer and buy a second set of KB mirrors in upgrade path.

Current X21 Surface/Interface User Community

The Roll-On/Roll-Off chamber approach has been very productive at NSLS X21:

- Surface Morphology Evolution during Ion Bombardment (Ludwig, Headrick – Vermont, Aziz – Harvard, Allain – Purdue)
- Plasma-Assisted MBE Growth of Nitrides (Ludwig)
- Atomic Layer Deposition (Detavernier – Ghent)
- Evolution of Surface Roughness during Mulilayer Growth by Sputter Deposition (Headrick – Vermont; Macrander – ANL)
- PLD of Semiconductors and Oxides (Headrick – Vermont)
- Reactive Sputter Deposition of Oxides (Dawber – Stony Brook)
- Evaporation of Thin Film Photocathodes (Smedley – BNL)

ISR Beamline Cost Estimate Breakdown

Burdened/Escalated Dollars

WBS	Total Labor ($)	Total M&S ($)	Total Budgeted Cost ($)
2.06.01 Photon Delivery System	1,487,860	3,467,995	4,955,855
2.06.02 Shielded Enclosures	146,005	1,262,660	1,408,665
2.06.03 Endstation Equipment	487,107	742,462	1,229,569
2.06.04 Controls and Data Acquisition	532,341	339,930	872,271
2.06.05 Beamline Management	255,597	237,161	492,758
2.6 ISR Beamline Total	**2,908,911**	**6,050,208**	**8,959,119**

Direct Dollars

Photon Delivery System Major Items

- Mirrors (3) $1000K
- DCM w/ cryocooler $720K
- Dual phase plate assembly $450K
- Slit systems (2) $150K
- Non-optics beamline components $750K

$3070K

Endstation Equipment Major Items

Secondary KB focusing optics $520K
Gas handling system $100K
Laser interlock and fluorine detectors $29K

$649K

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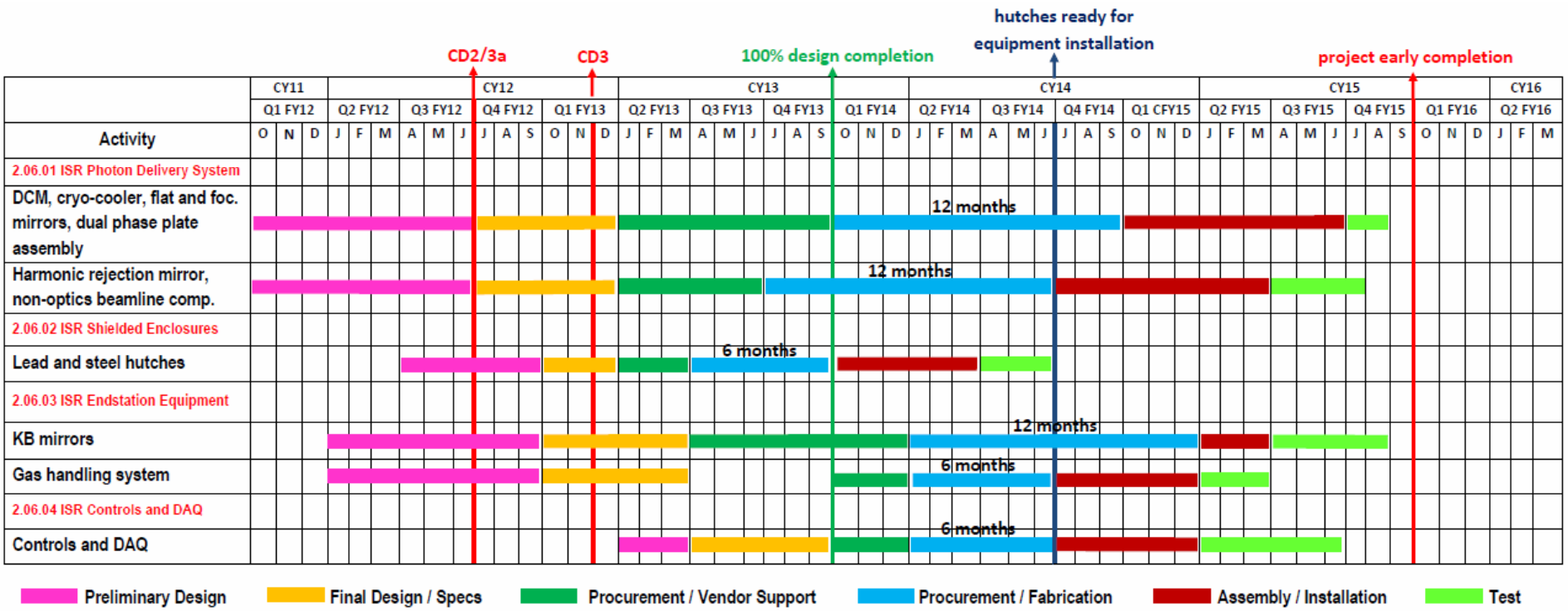
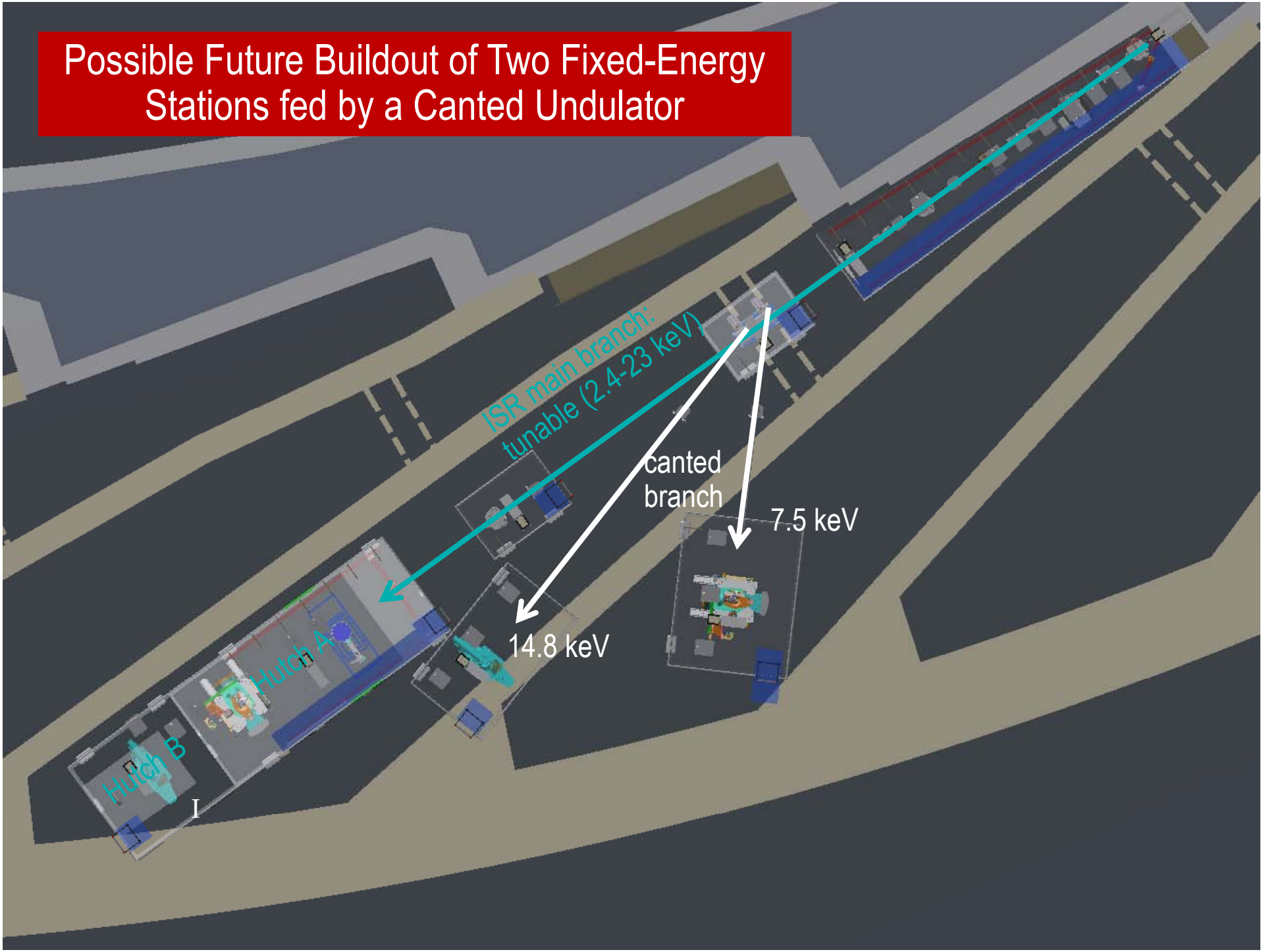
ISR Beamline Schedule Summary



- 2 years (FY12 and FY13) until 100% design completion
- 1 year between contract award and delivery for DCM, cryo-cooler, mirrors, and dual phase plate assembly
- Endstations in use and being improved until end of NSLS operations (mid-FY14)
- 15 months for assembly/installation and integrated testing of photon delivery system, endstations, and controls

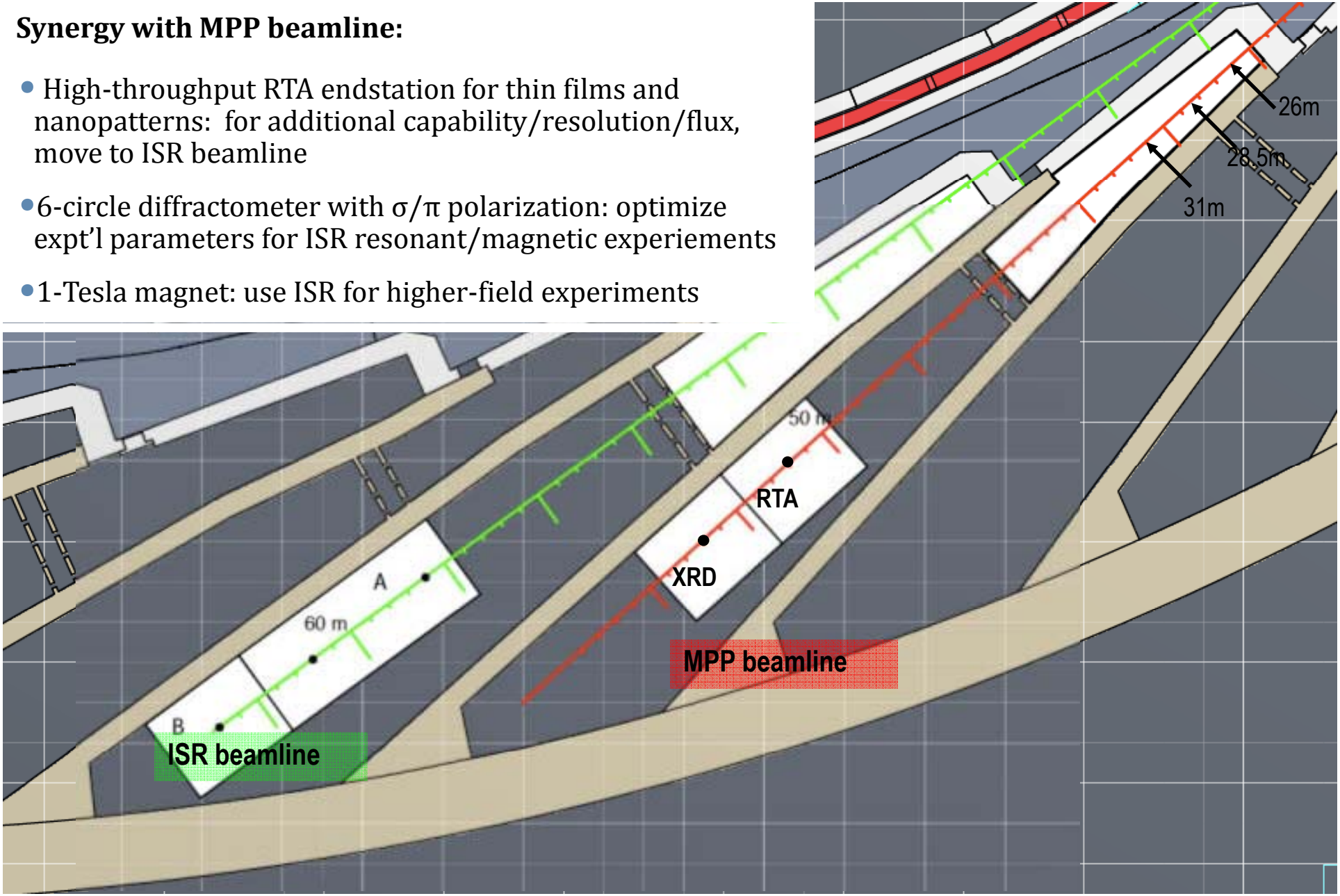
Possible Future Buildout of Two Fixed-Energy Stations fed by a Canted Undulator



MPP: Proposed Materials Physics and Processing – 3PW Beamline

Synergy with MPP beamline:

- High-throughput RTA endstation for thin films and nanopatterns: for additional capability/resolution/flux, move to ISR beamline
- 6-circle diffractometer with σ/π polarization: optimize expt'l parameters for ISR resonant/magnetic experiements
- 1-Tesla magnet: use ISR for higher-field experiments



ISR Summary

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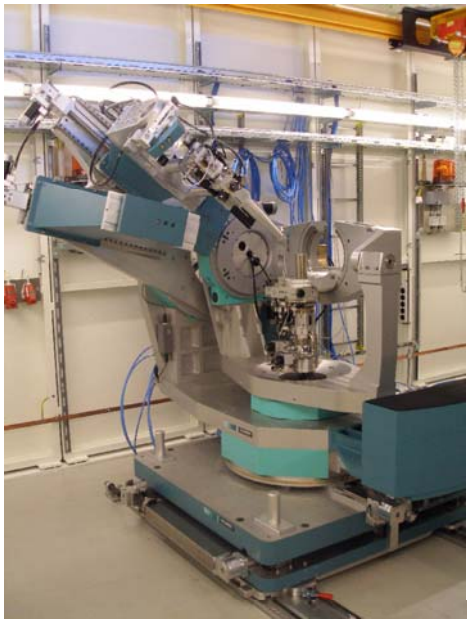

Huber Diffractometer on PETRA III
Resonant X-ray Scattering Beamline

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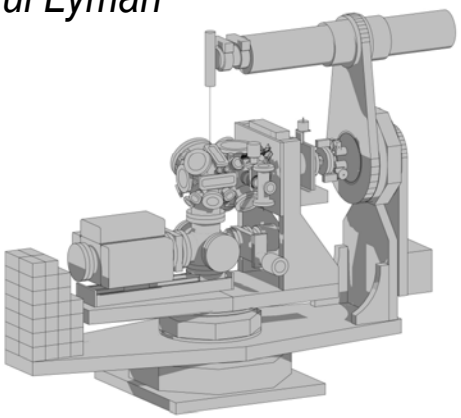
Paul Lyman



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	3 keV	10 keV	20 keV
flux in Si(111) bandpass	$4x10^{14}$ ph/s	$6x10^{13}$ ph/s	$7x10^{12}$ ph/s

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- Possibility of later canted undulator build-out of 2 fixed-energy stations