Development of Scanning Hard X-ray Microscopy at 1~10nm

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U.S. DEPARTMENT OF ENERGY

X-ray Interface Science at Advanced Photon Source
APS, Jan 10-11th, 2012

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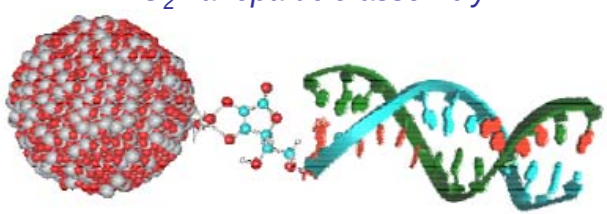
Scientific Scope of Hard X-ray Nanoprobe at NSLS-II

- Explore new frontiers of hard x-ray microscopy with the highest achievable spatial resolution:
  - Initial goal: 10 nm using multilayer Laue lenses (MLLs) and 30nm using x-ray zone plate
  - long-term goal: 1~10 nm using MLLs
- Complement existing electron microscopy capabilities with higher elemental sensitivity, and enable *in-situ* imaging in a variety of realistic environmental conditions.

Supported Techniques at x-ray energies, 6~25 keV
- Primary: Scanning X-ray Fluorescence & Diffraction/Scattering
- Secondary: Differential Phase Contrast Imaging & Ptychography

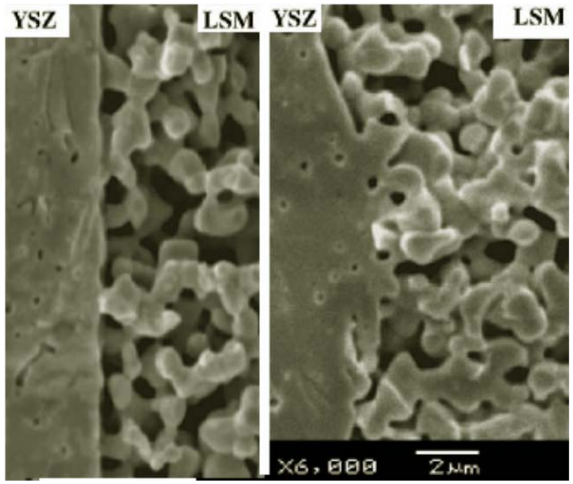
metals in biological systems

DNA-TiO_2 nanoparticle assembly

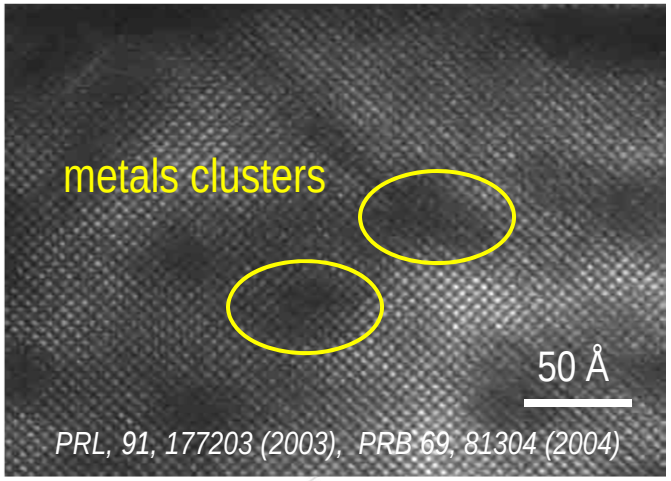


Nat. Mat. 2, 343-346 (2003)

solid oxide fuel cells



magnetic semiconductors



PRL, 91, 177203 (2003), PRB 69, 81304 (2004)

Hard X-ray Nanoprobe (HXN) Team

HXN beamline Group

- Yong Chu: Group leader
- Hanfei Yan : MLL theory and experiments
- Evgueni Nazaretski: HXN microscope design
- Jungdae Kim (postdoc): Nanopositioning research
- Dennis Kuhne (technician)
- Ken Lauer (controls. Eng): microscope control
- Postdoc: OPEN
- Mechanical Engineer: OPEN

- Deming Shu (ANL, joint work project)
- Ken Evans-Lutterodt (MOU staff)

NSLS-II Optics Fabrication Group

- Ray Conley (GL): MLL fabrication and processing
- Nathalie Bouet: MLL RIE etching
- Juan Zhou (RA): Crystal optics & MLL sectioning

Beamline Advisory Team Members

C. Noyan (Columbia) – Chair
D. Bilderback (Cornell);
C. Jacobsen (APS/Northwestern)
T. Lanzirotti (U. Chicago);
T. Buonasisi (MIT)
S. Vogt (ANL)
M. Holt (ANL);
K. Evans-Lutterodt (BNL)

Key External Collaborators on MLL

- Deming Shu (ANL, Nanopositioning)
- Jörg Maser (ANL, MLL theory & experiment)
- Hyon ChoL Kang (Chosun Univ, Korea, MLL experiment)
- Volker Rose (ANL, MLL experiments)
- Martin Holt (ANL, MLL experiments)
- Albert Macrander (ANL, MLL experiments & metrology)
- Lahsen Assoufid (MLL fabrication/processing)

Nanoprobes around the world

Beamline @ SR	keV	Focusing optics	Focus size, flux, enegy resolution	Primary Imaging modality	Major Infrastructure	Operation Status
HXN@NSLS-II	6-25	MLL, ZP	~1 nm, ultimate goal. 10nm ($1x10^{10}$), 20nm ($9x10^{10}$), 45nm ($2x10^{11}$), at 10keV, w/MLLs, dE/E~10^{-4}	XRF, XRD	satellite bldg, microscope with 1nm stability	in 2015
SRX@ NSLS-II	4.65 – 23.2	KB	300 x 300 nm: > 10^{13}, dE/E~10^{-4} 60 x 60 nm: 10^{11} – 10^{12}, dE/E~10^{-4}	XRF, XAS, EXAFS		In 2015
INP@APS	4-30	KB	50nm, 9 $x10^{11}$ at 10keV, dE/E~10^{-2}	XRF, XAS	sample T cntrl at 40k~1000C	in ~2016
Nanoscp'm @ SOLEIL	5 – 20	KB (ZP)	30~1000 nm, 1.4 x 10^{10} (110x110 nm^2), KB, dE/E~10^{-2}	XRF, XAS, CDI	Satellite bldg	in 2012
NA @ ESRF	5-70	KB	50~1000 nm, ~10^{11}, dE/E~10^{-4}	XRF, XAS, XRD	Satellite bldg, cryo for sample	in 2014
NI@ ESRF	11-34, descrte energies	KB	10 ~ 100 nm, ~10^{12}	XRF, full-field Imaging	Satellite bldg, cryo for samle	in 2014
BioNano@APS	3-30	ZP	30~100nm, ~10^{9}, dE/E~10^{-4}	XRF, XAS	Cryo + sample exchanger	Commissioning
26-ID@ APS	3-30	ZP	30nm (~10^{9}) – 100nm, dE/E~10^{-4}	XRD, XRF, TXM	sample T cntro at 100K-600C	Operating
ID21@ESRF	2 –7.5	KB, ZP	200~1000 nm , ~10^{9}, dE/E~10^{-2}	full-field imaging, XRF, XAS		Operating
XFM@AS	4 – 25	KB, ZP	5um (1.5x 10^{10}, KB, dE/E~10^{-2}) 100 nm (10^{9}, ZP, dE/E~10^{-2})	XAS, XRF, transm. Imaging		Operating
P06@Petra III	4-80 15-30	KB CRL	< 1000nm (KB) 50nm (~10^{9}, CRL, dE/E~10^{-2})	XRF, XAS, CDI		Operating

* Compiled from information available on the Web.

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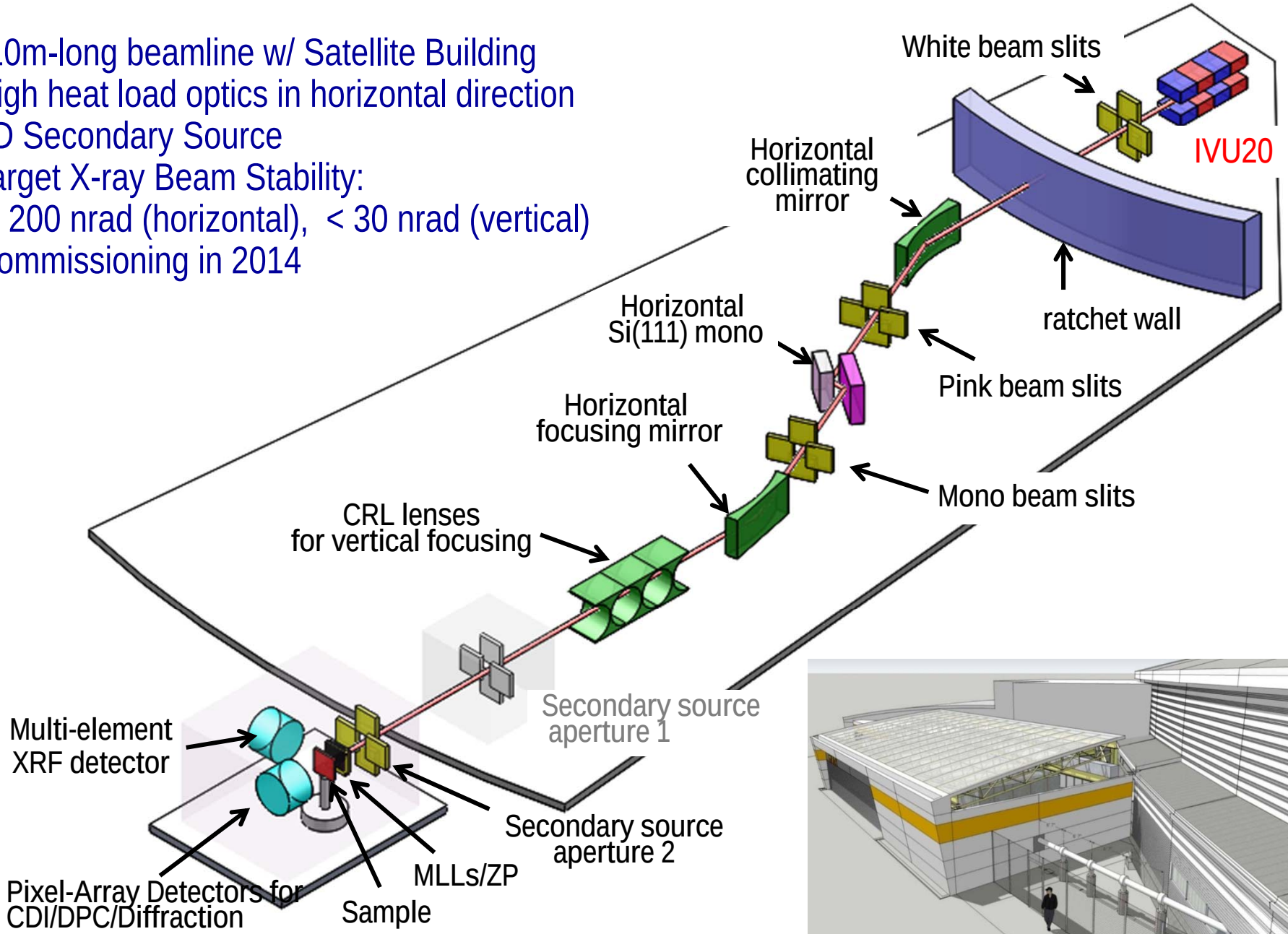
Technical Challenges

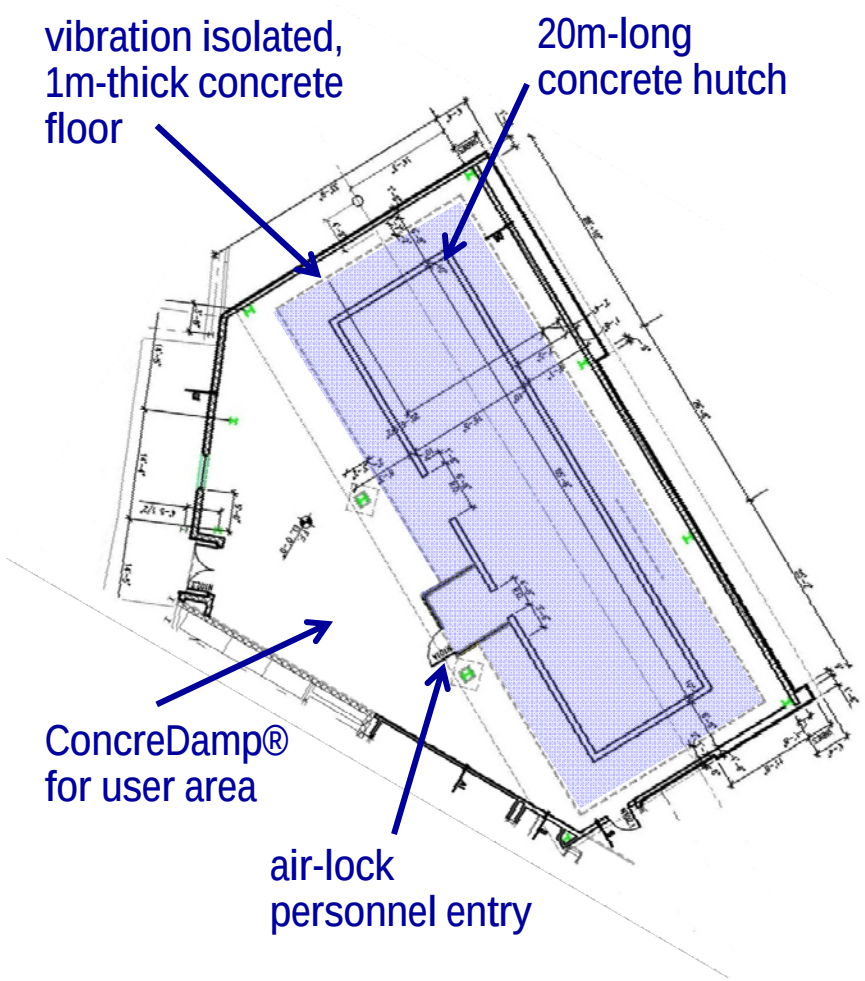
- Stability of X-ray Beam and Experimental Environment
- X-ray Optics at 1~10 nm
- X-ray Microscope with Sub-1nm Stability

Optical Layout of HXN Beamline at NSL-II

- 110m-long beamline w/ Satellite Building
- High heat load optics in horizontal direction
- 2D Secondary Source
- Target X-ray Beam Stability:
  < 200 nrad (horizontal), < 30 nrad (vertical)
- Commissioning in 2014



HXN Satellite Building

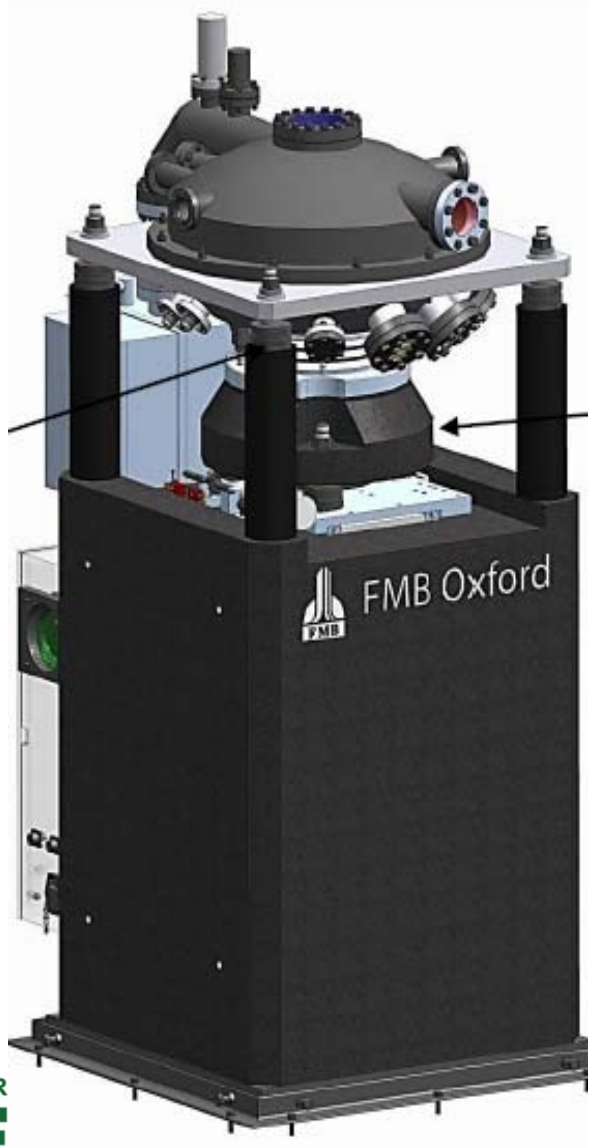


~520 ton concrete block

Major Environment Specifications:

- Vibration specification:
  NIST VC-F (1.56um/s floor, 1-80 Hz).
- Temperature specification
  < 0.1º C (< 0.05º C in 1 hr-period)
  Reach equilibrium temperature within 30 minutes

Development of Ultra Stability DCM



Key Features:

- Horizontal Diffraction Geometry
- Psudo-channel cut with small gap
- Small lever arms for reducing vibration amplification
- Target: 50nrad angular stability
- Vibration measurements on the prototype (work in progress):

crystal separation: ~0.5 nm rms @40Hz
crystal relative roll: ~4 nrad rms @40Hz
crystal relative pitch: ~8 nrad rms @40Hz

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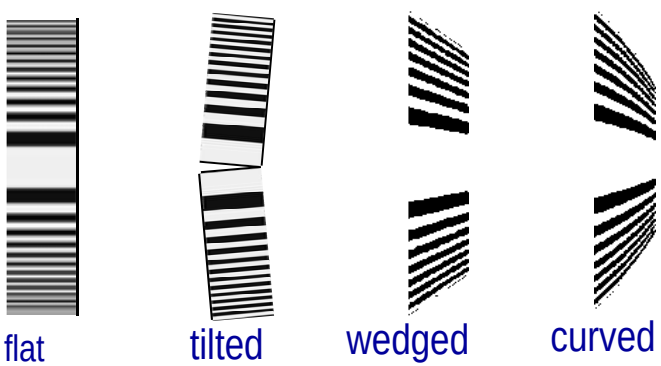
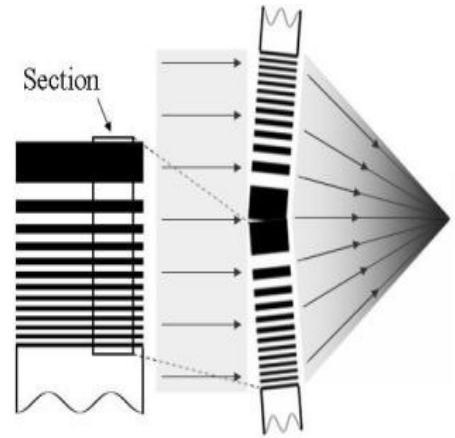
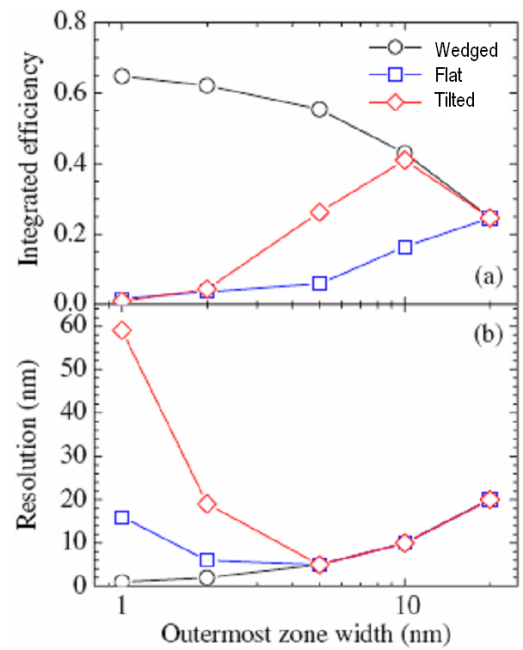
Multilayer Laue Lens (MLL)

- Originally conceived by Jorg Maser, and later developed at the APS
- Overcome "aspect ratio" problem of ZP by thin-film deposition
  → achieve virtually limitless aspect ratio, but requires sectioning
  → ~1nm layer spacing feasible
  → 2D focusing requires two linear lenses ("KB" configuration)
- Volume optic
  → strong dynamical effect
  → high efficiency
  → chromatic focusing
  → no theoretical limitation for 1nm focusing
- Current material system: WSi_2/Si
- Achieved resolution limit:
  1D: 16nm (Kang et. al. APL 92, 2008)
  2D: 25 x 27 nm (Yan et. al., Optics Express 19, 2011)





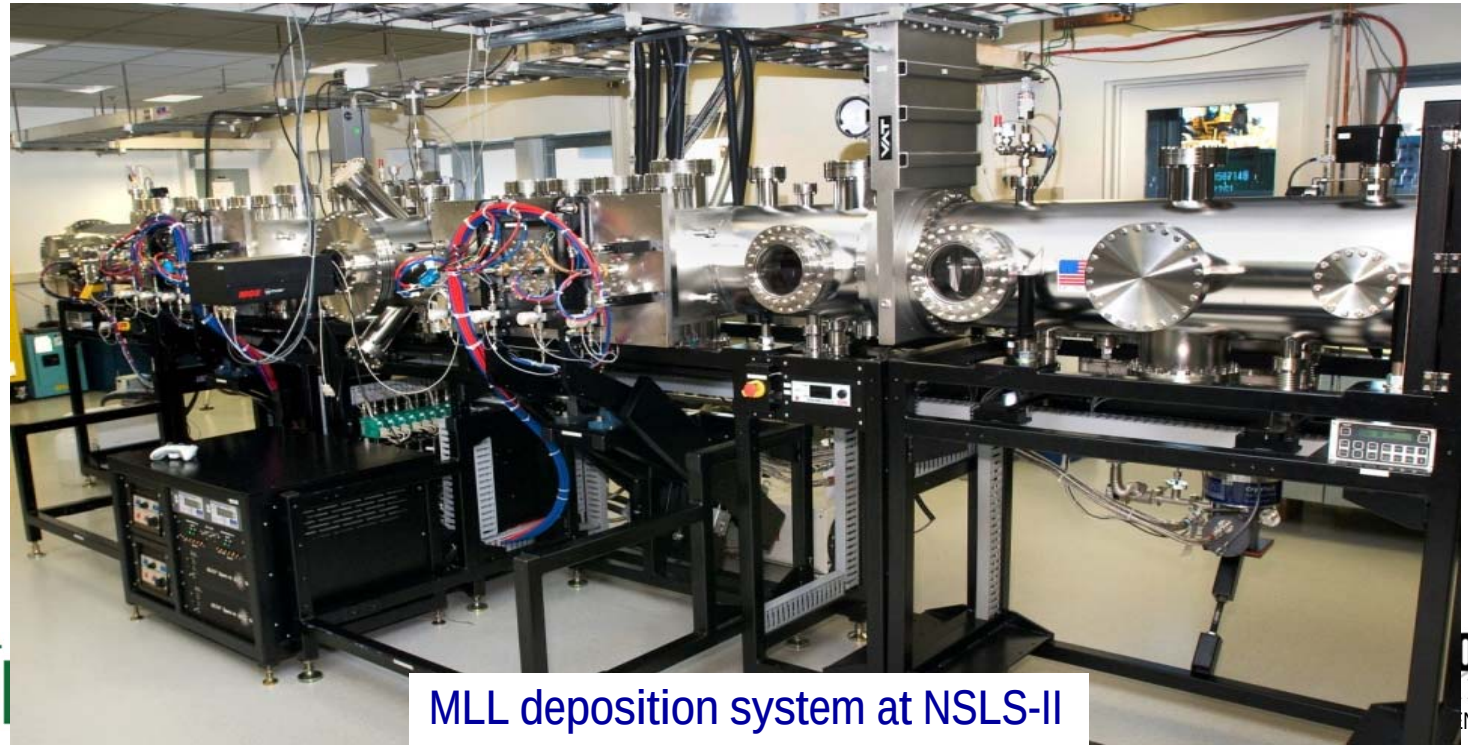


Kang et. al. PRL. 96 (2006)

MLL Fabrication Capability at NSLS-II

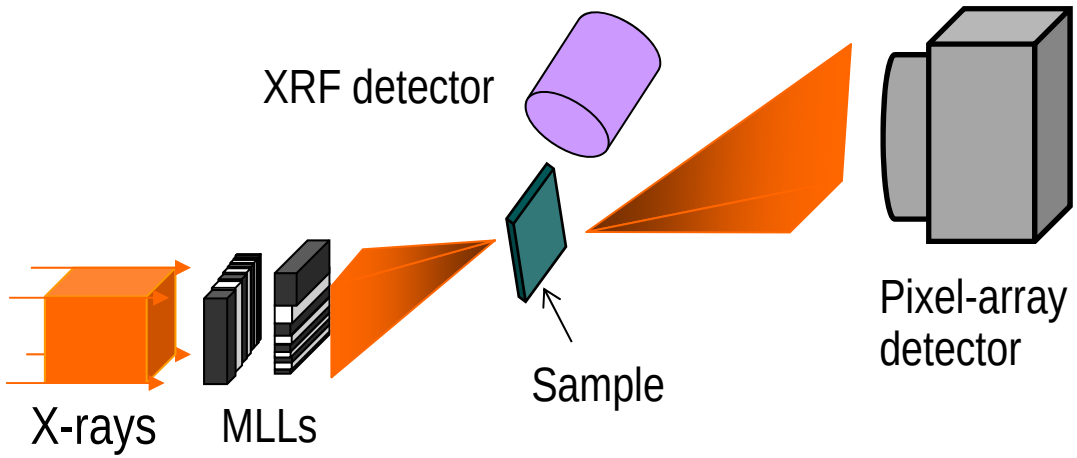
R. Conley & N. Bouet

- Advanced MLL deposition system is commissioned in May, 2010.
  - 7.7m-long with 9 sputter targets, horizontally mounted
  - Super-precision linear-drive system with ~1μrad trajectory straightness
  - capable of growing MLLs with ~300μm aperture
- Target by 2013: ~150μm tilted MLLs (dr_N <4nm) , ~50μm wedged MLLs (dr_N < 2.5nm)
- Current Achievement:
  - 68μm titled MLLs with 4nm outmost zone width (dr_N)

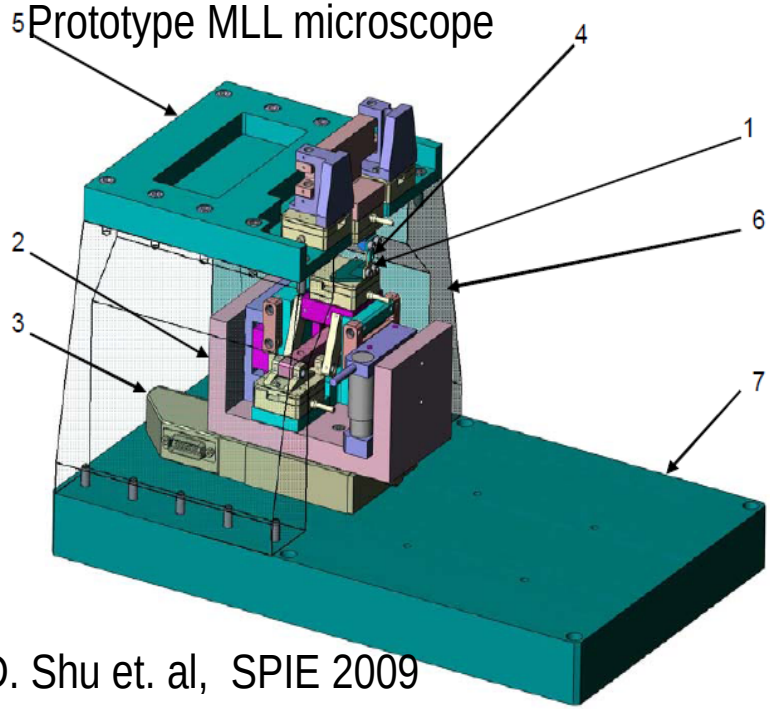


MLL deposition system at NSLS-II

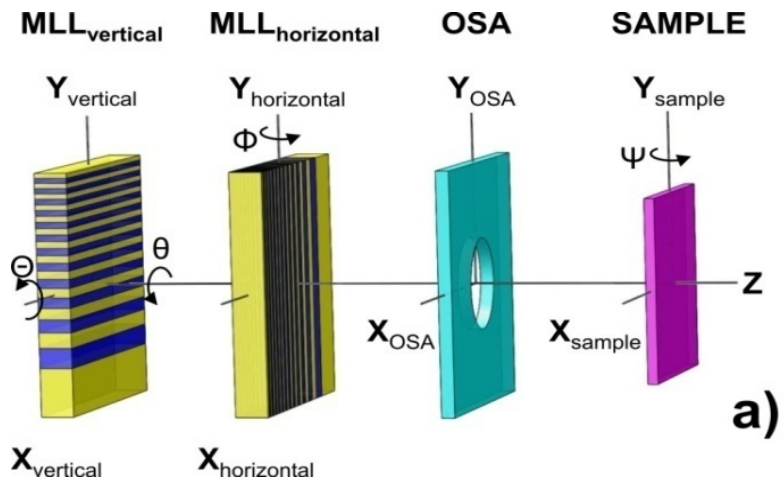
Microscopy Experiment using MLLs
at APS 26-ID



Prototype MLL microscope

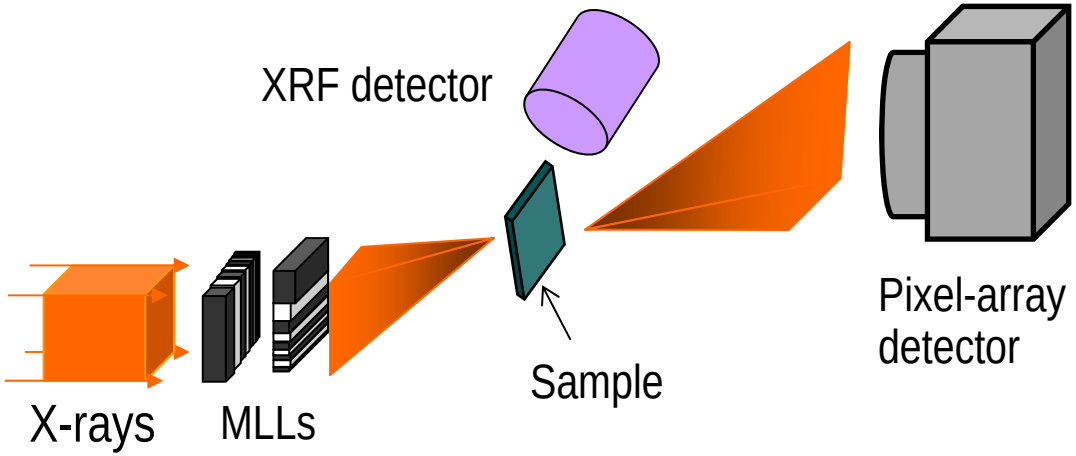


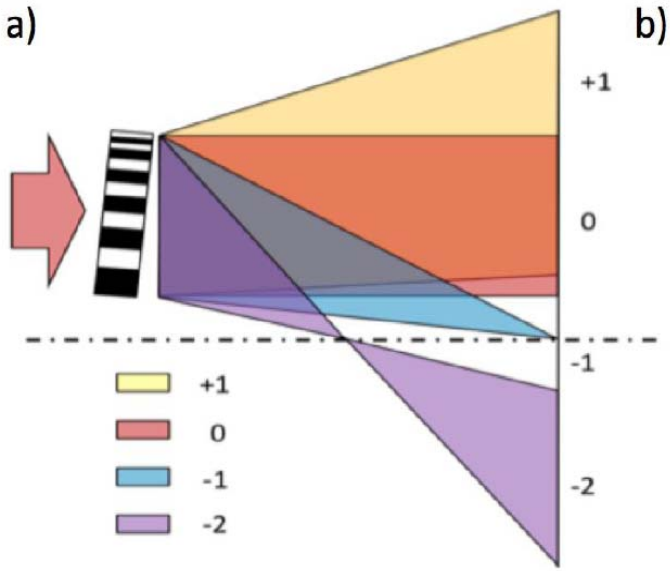
D. Shu et. al, SPIE 2009



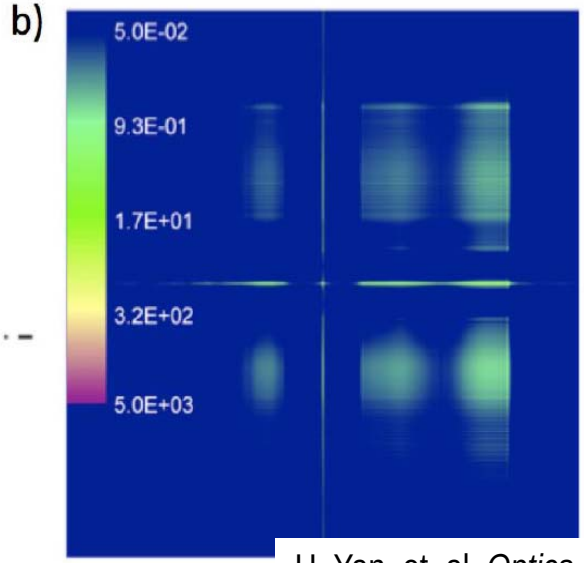
Provides 8 degrees of freedom
5 rotations + 3 translations

Microscopy Experiment using MLLs
at APS 26-ID

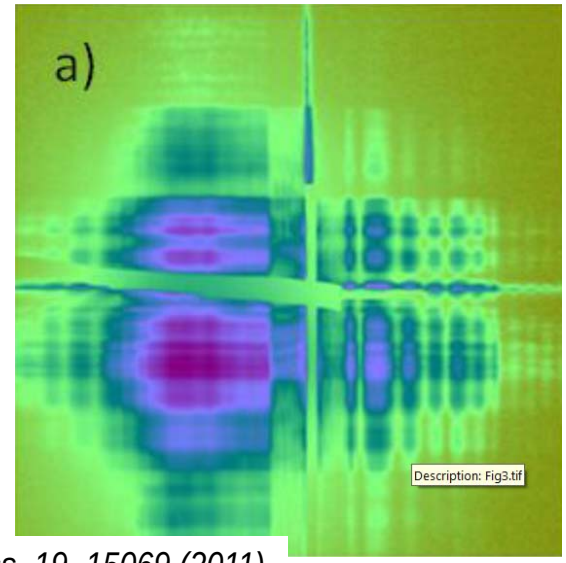




at focus



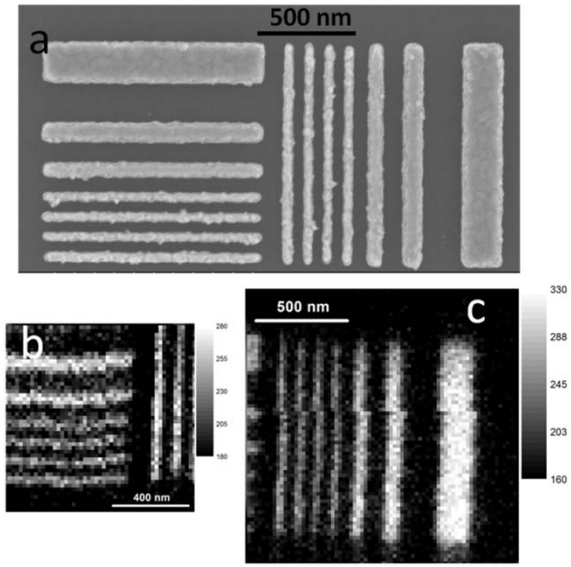
at far-field

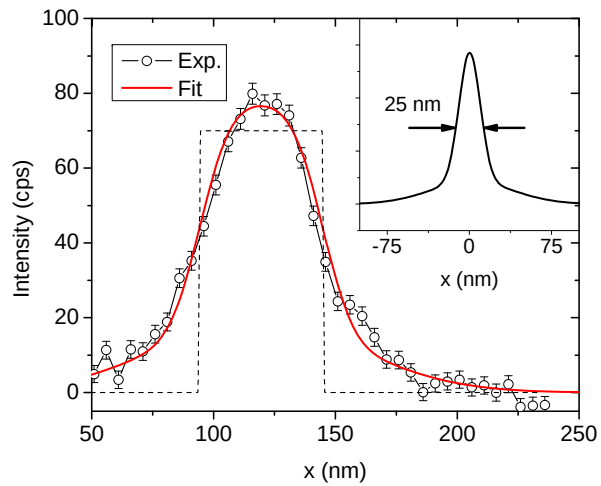


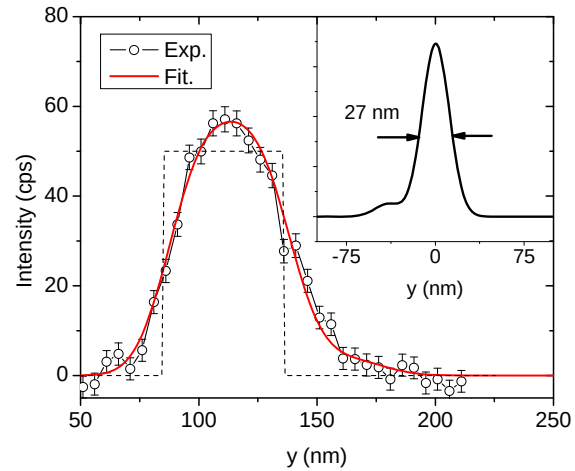
H. Yan, et. al. *Optics Express, 19, 15069 (2011)*

Experimental Results in 2010

at APS 26-ID







25 x 27 nm^2 at 12 keV

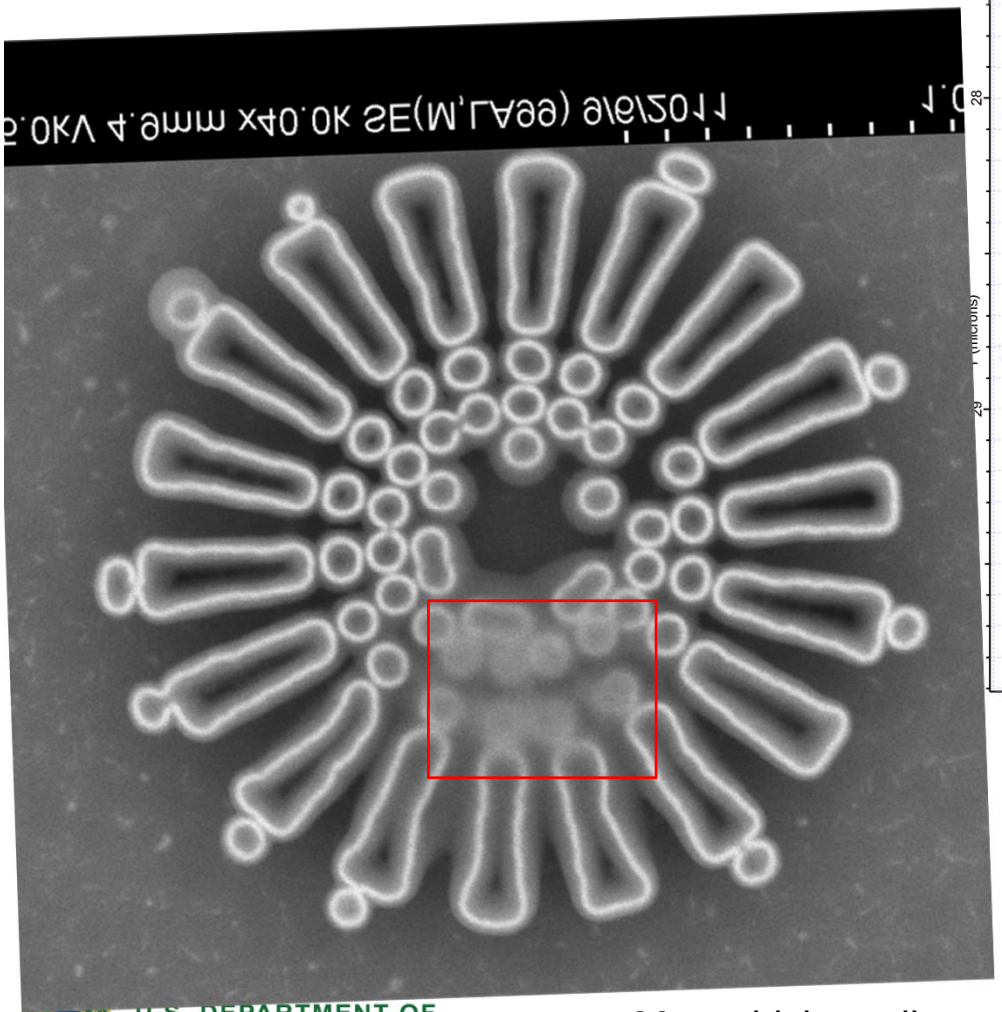
Measured Efficiency:
2% at 12 keV
17% at 19.5 keV

H. Yan, V. Rose, D. Shu, E. Lima, H. C. Kang, R. Conley, C. Liu, N. Jahedi, A. Macrander, G. B. Stephenson, M. Holt, Y. S. Chu, M. Lu, J. Maser, *"Two dimensional hard x-ray nanofocusing with crossed multilayer Laue lenses", Optics Express, 19, 15069 (2011)*

Experimental Results in 2011

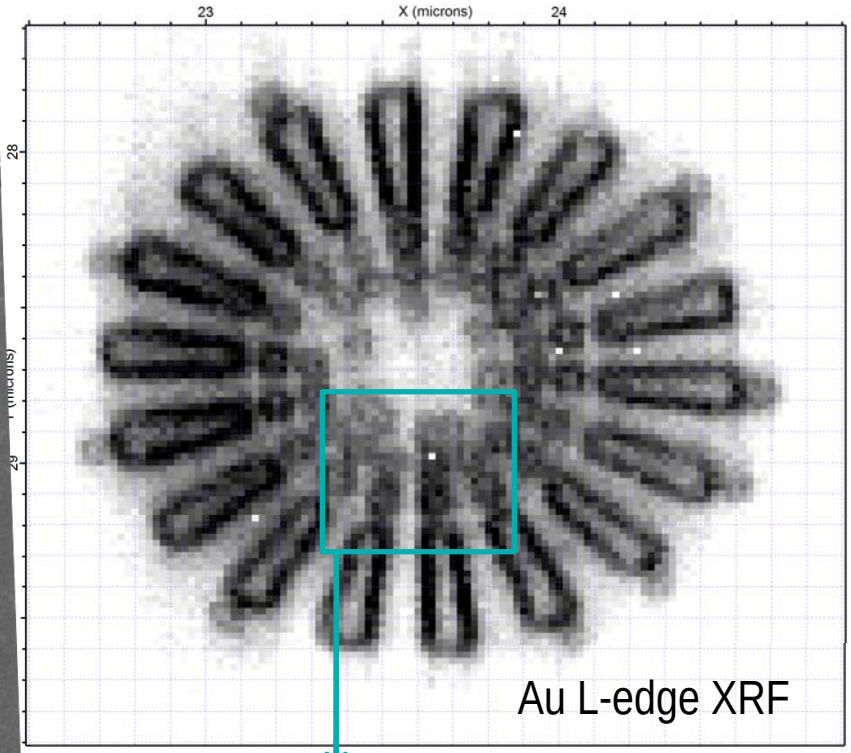
at APS 26-ID

SEM

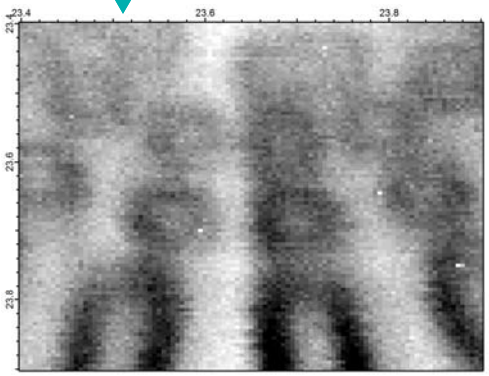


20nm-thick Au lines

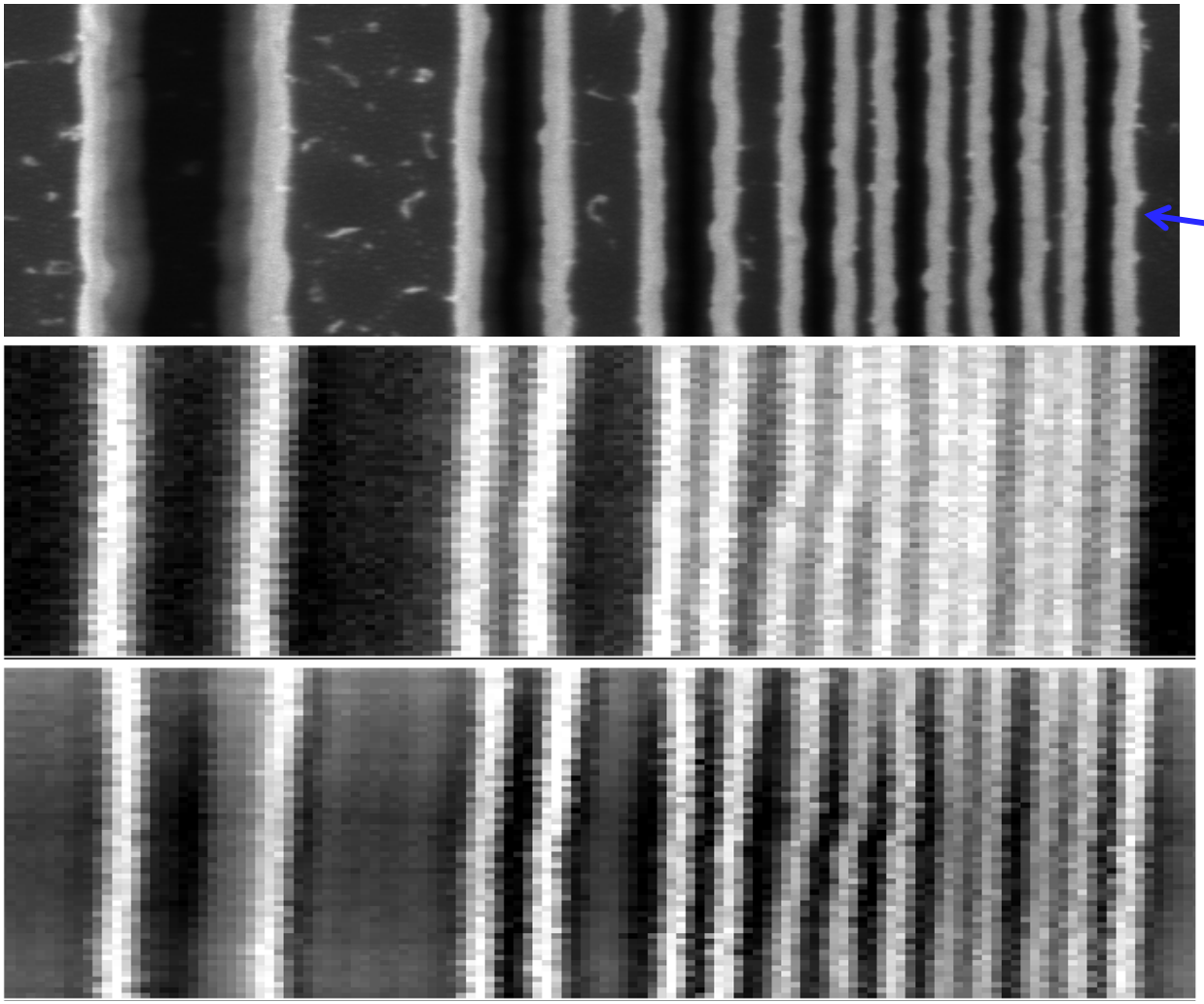
E= 12 keV



Au L-edge XRF



Demonstration of Differential Phase-Contrast Imaging



SEM

20nm Pt line
200 nm tall

Pt L-edge fluorescence

Differential Phase Contrast using Pilatus 100k

Nanofocusing Optics for HXN

Initial Goal

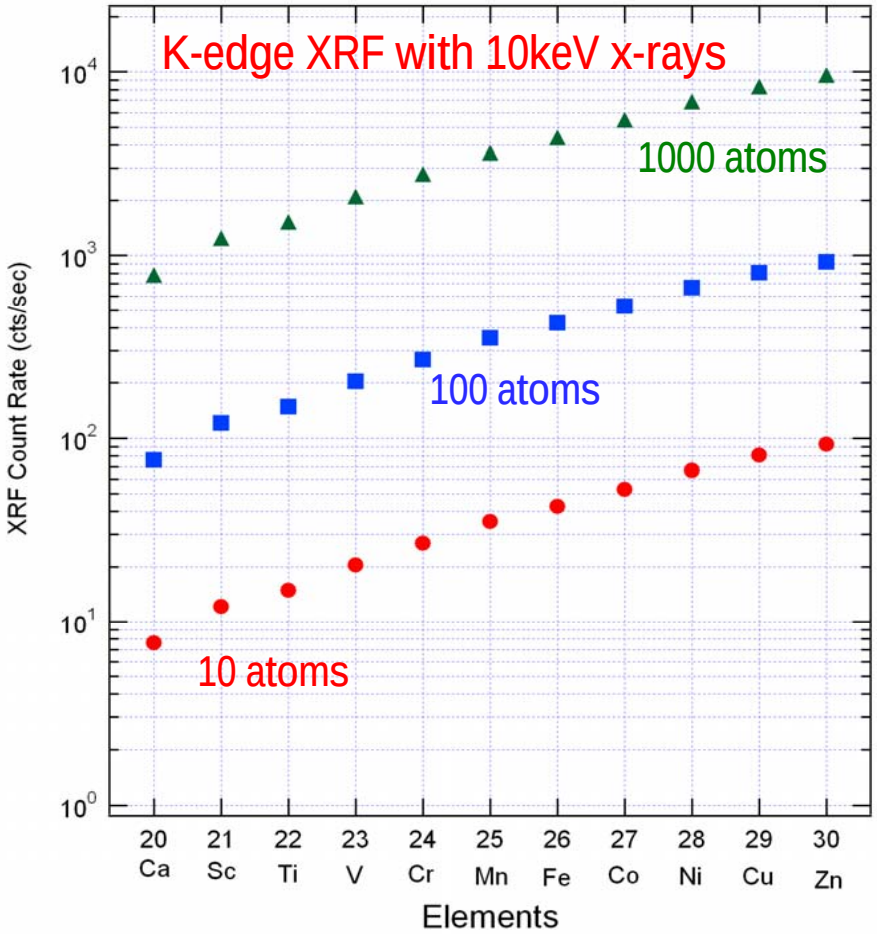
- 10 nm spatial resolution (~1 mm working distance) using MLLs
- 30 nm spatial resolution (5~9 mm working distance) using ZP
- Using the monochromaticity of Si (111)

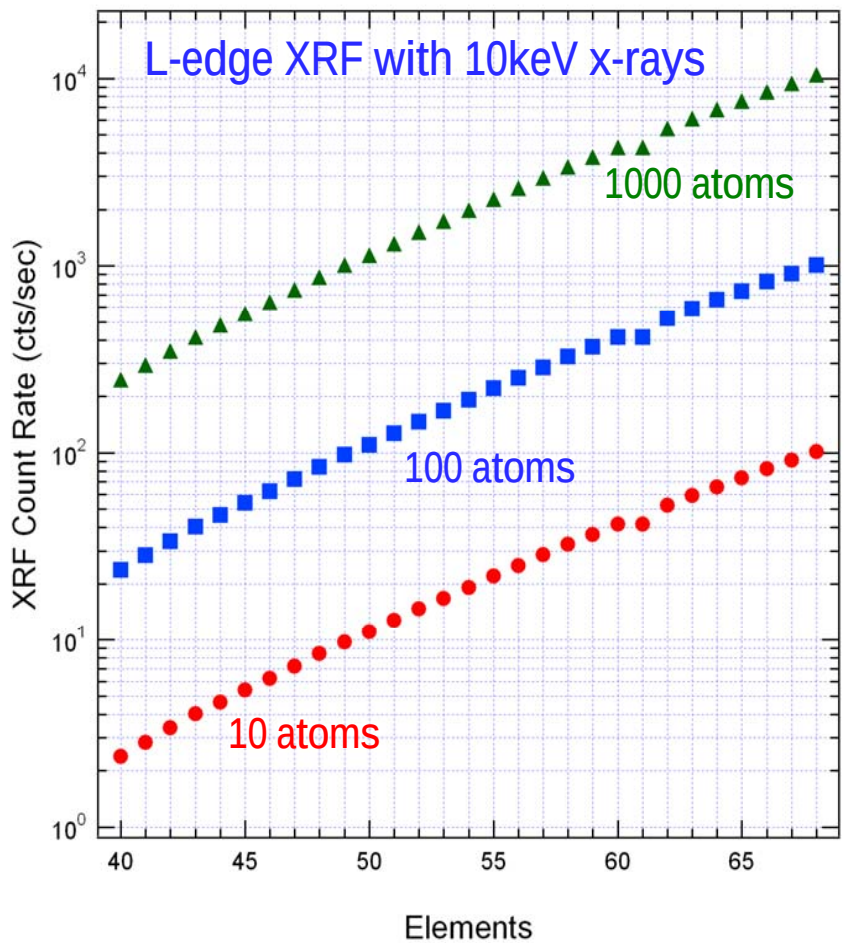
optics	resolution (nm)	energy range (keV)	aperture (µm)	depth of focus (µm)	focal length (mm)	working distance (mm)	2D efficiency*
flat MLL	10	10~25	62 (10keV) 62 (20keV)	3.2 (10keV) 6.4 (20keV)	5	1	2.3% (10keV) 4.0% (20keV)
wedged MLL	10	10~25	32 at10keV 32 at 20keV	3.2 at 10keV 6.4 at 20keV	2.6	1.1	48% (10keV) 48% (20keV)
ZP	30	6~12	150	17.4 (6keV) 34.8 (12keV)	17.8 (6keV) 35.7 (12keV)	~5 (6keV) ~9 (12keV)	~2% (10keV)

*Efficiency is calculated using WSi_2/Si MLL system

Expected XRF Sensitivity

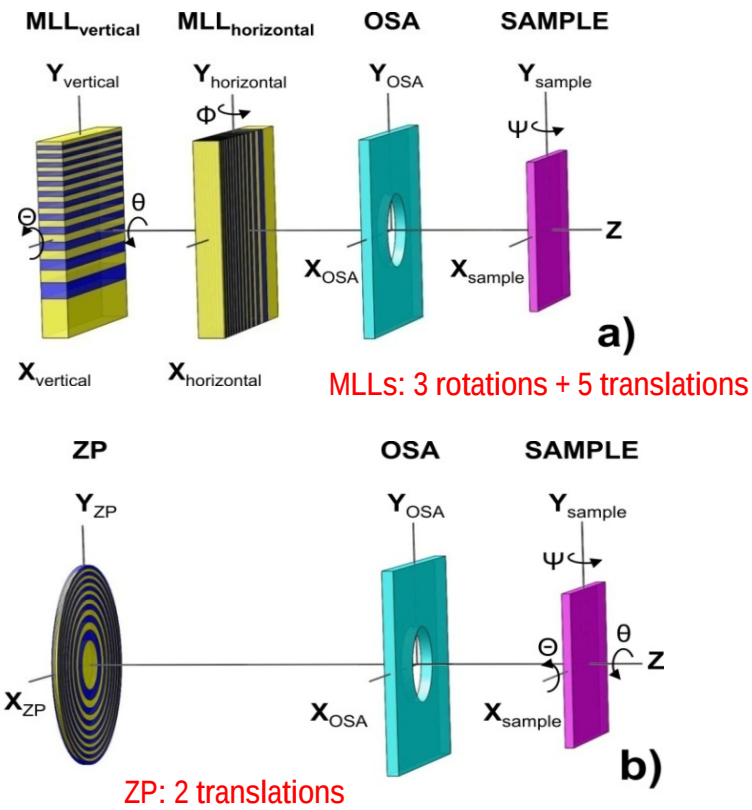
- at 10keV, 10.9nm focus, 1.1 x10^{10} ph/s, using wedged MLLs
- detector solid angle: 10% of 4π SR





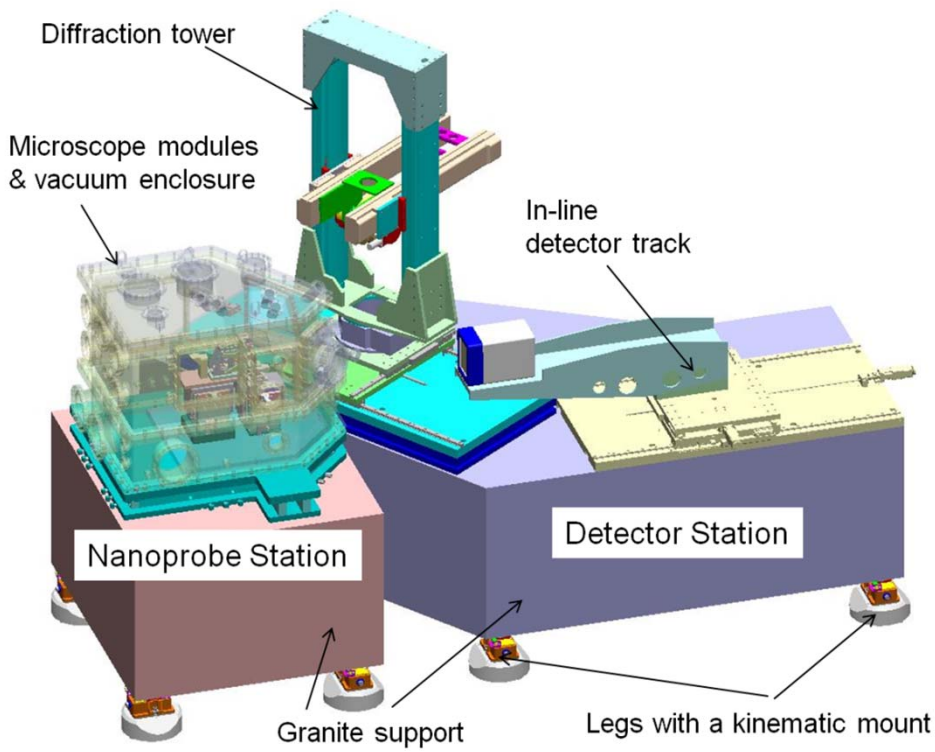
Development of HXN X-ray Microscope

- Single Instrument with multiple modules
  - MLL Optic Module (MOM)
  - Zone plate Optic Module (ZOM)
  - Sample Module for Fluorescence (SAMF)
  - Sample Module for 1-axis Diffraction (SAM1D)
  - Sample Module for 3-axis Diffraction (SAM3D)
- "Hot Swapping" capability
  - change modules while connected



Design Approach

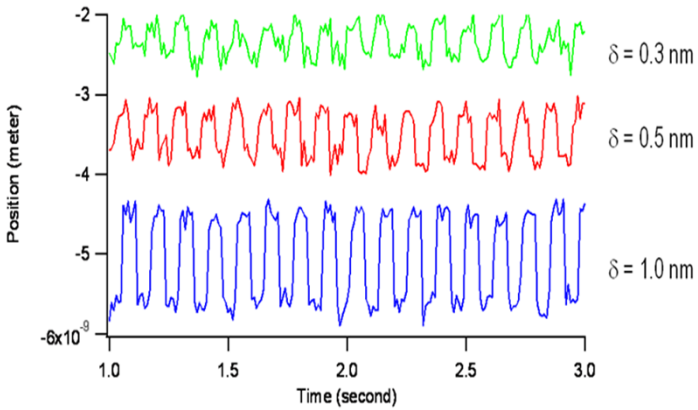
- Target temperature stability ~10mK
- Compact design ensuring high stiffness and minimal thermal drift.
- UHV compatible components
- Large solid angle XRF detectors
- Multiple optics for high-res and high-throughput
- A large exit angle for diffracted x-rays horizontal: -30~70º, vertical: -5~60º

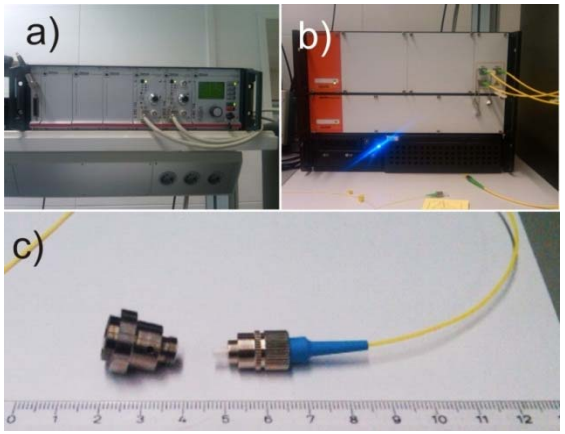


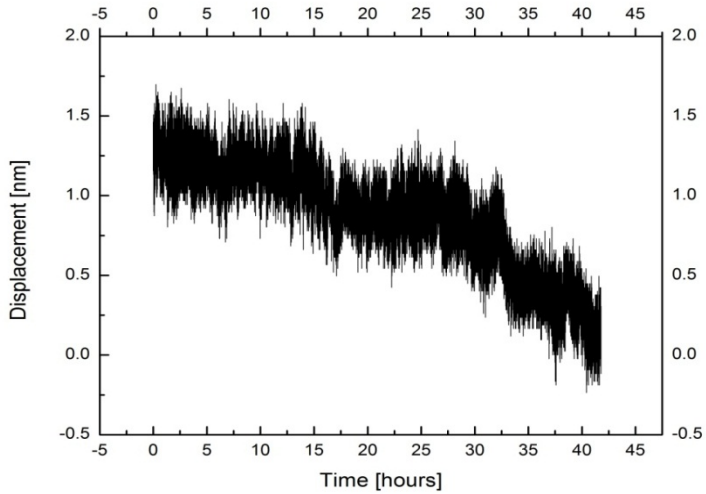
Nanopositioning R&D

E. Nazaretski

- Established In-house nanopositioning metrology capabilities.
- Developed compact fiber-optic interferometers with ~0.3nm sensitivity and stability over days.
- Performed extensive evaluation on commercial nano-stages

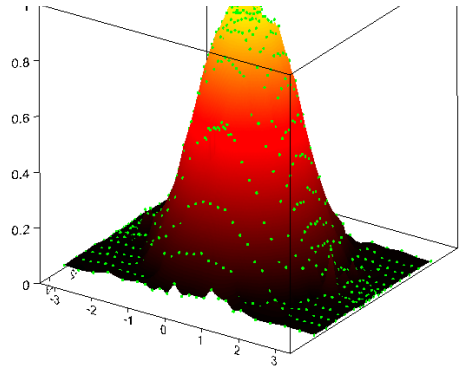




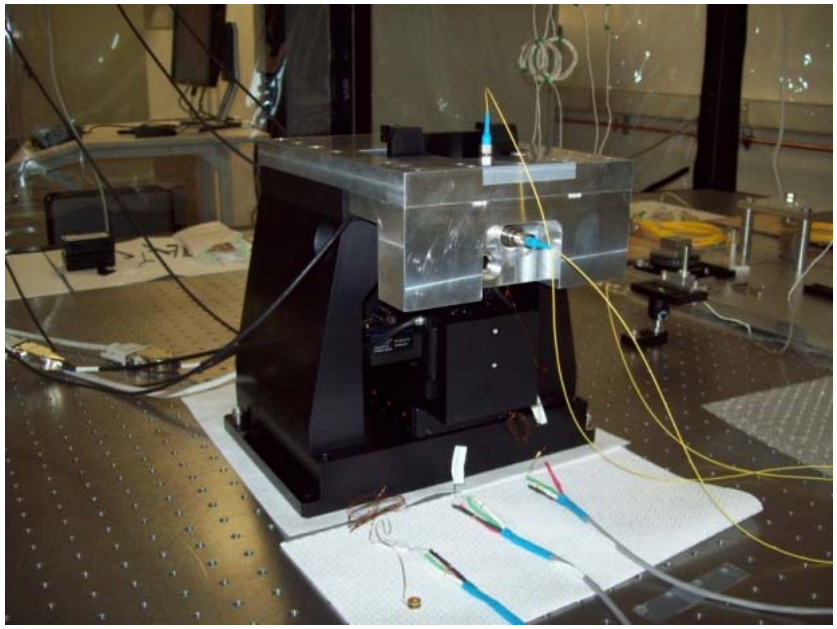


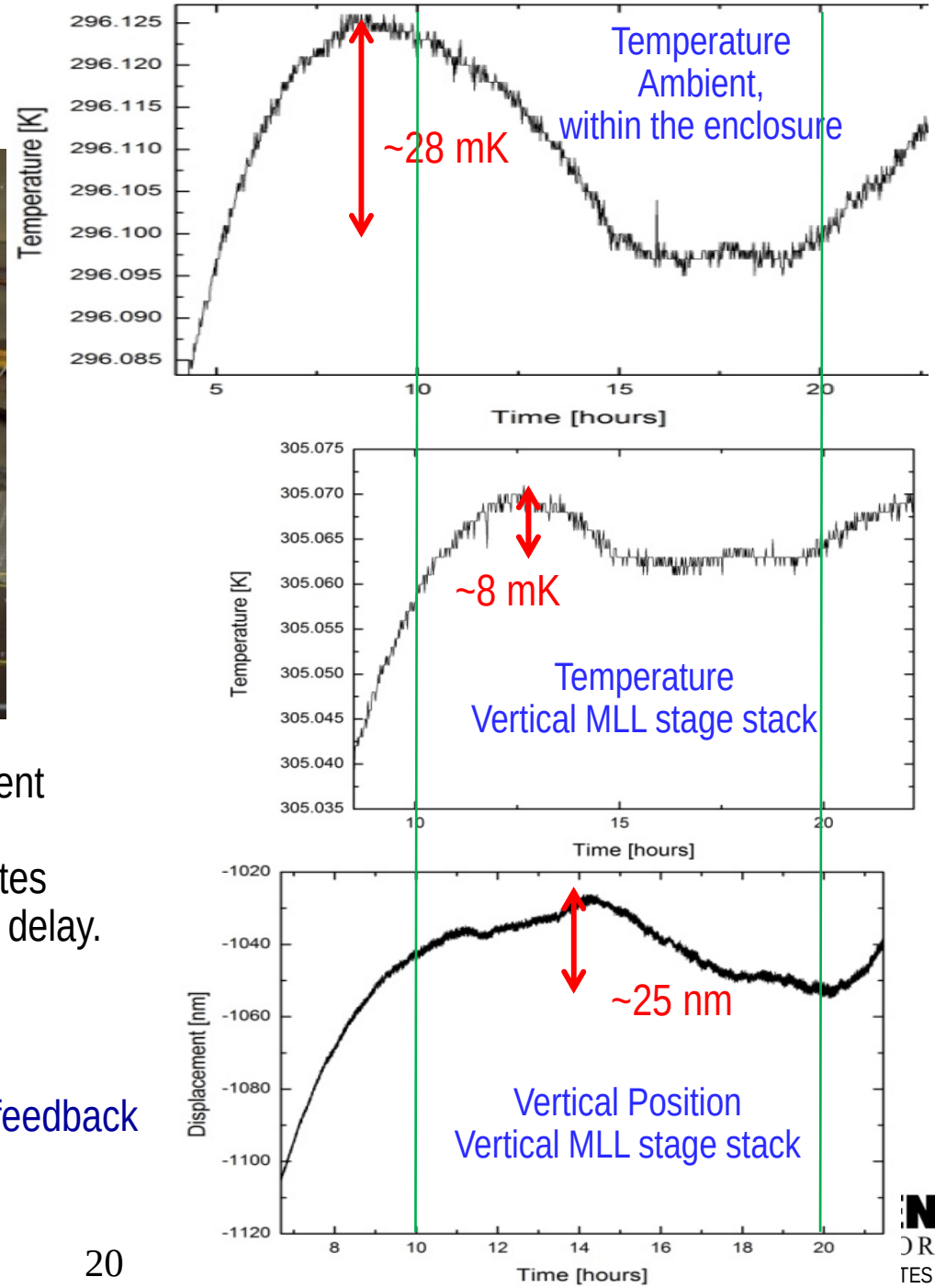
Interferometer angular acceptance of ~7º at 20mm working distance

→ important for tracking MLLs



Stability test of the prototype MLL microscope





- ~9 K temperature difference from the ambient and the MLL piezo stages.
- The temperature at the MLL stages correlates well with the ambient temperature with ~3.5hr delay.
- The vertical MLL position correlates with the temperature of the piezo stages.

→ Require better T. stability + interferometric feedback

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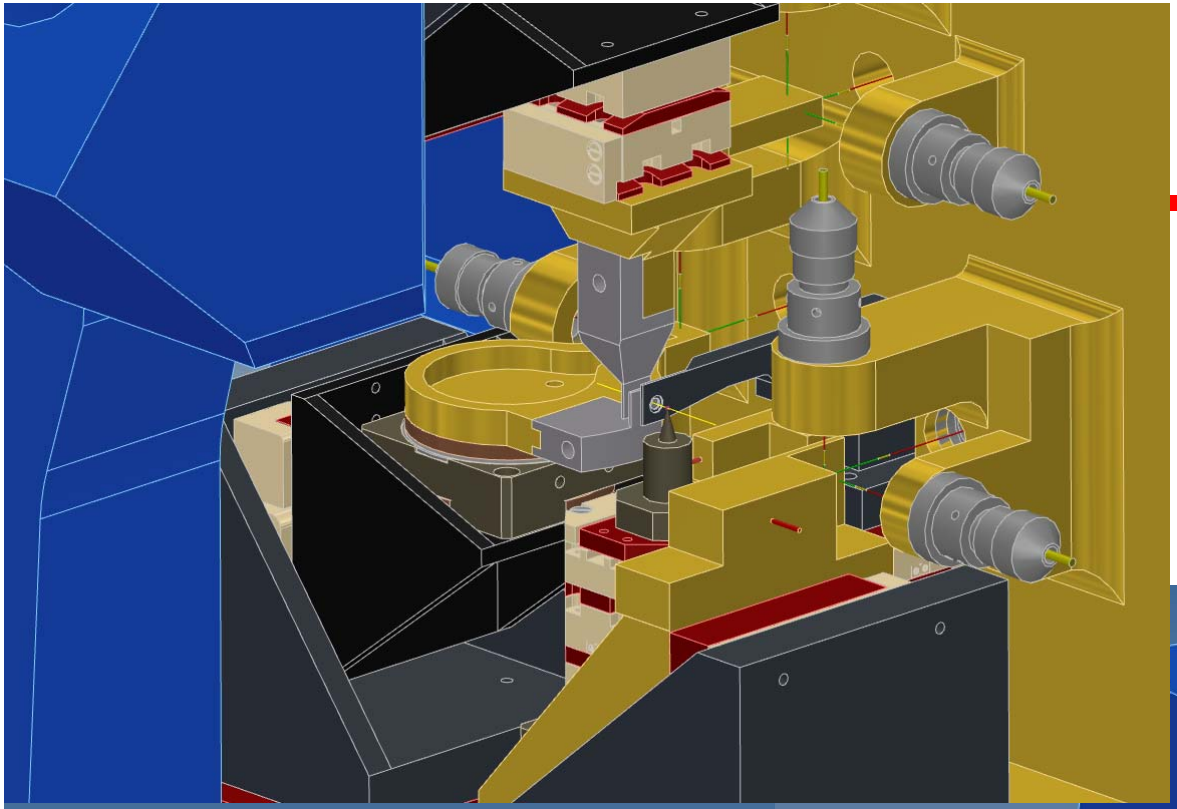
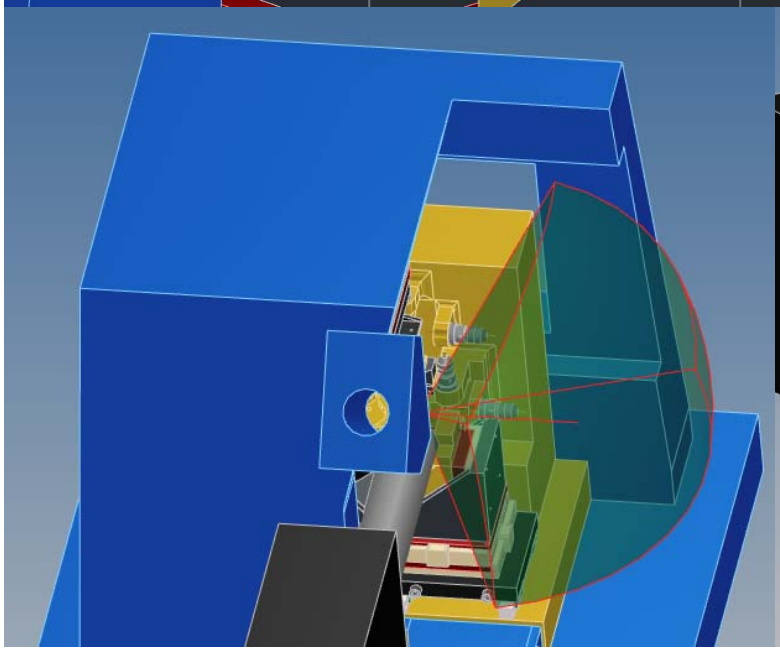
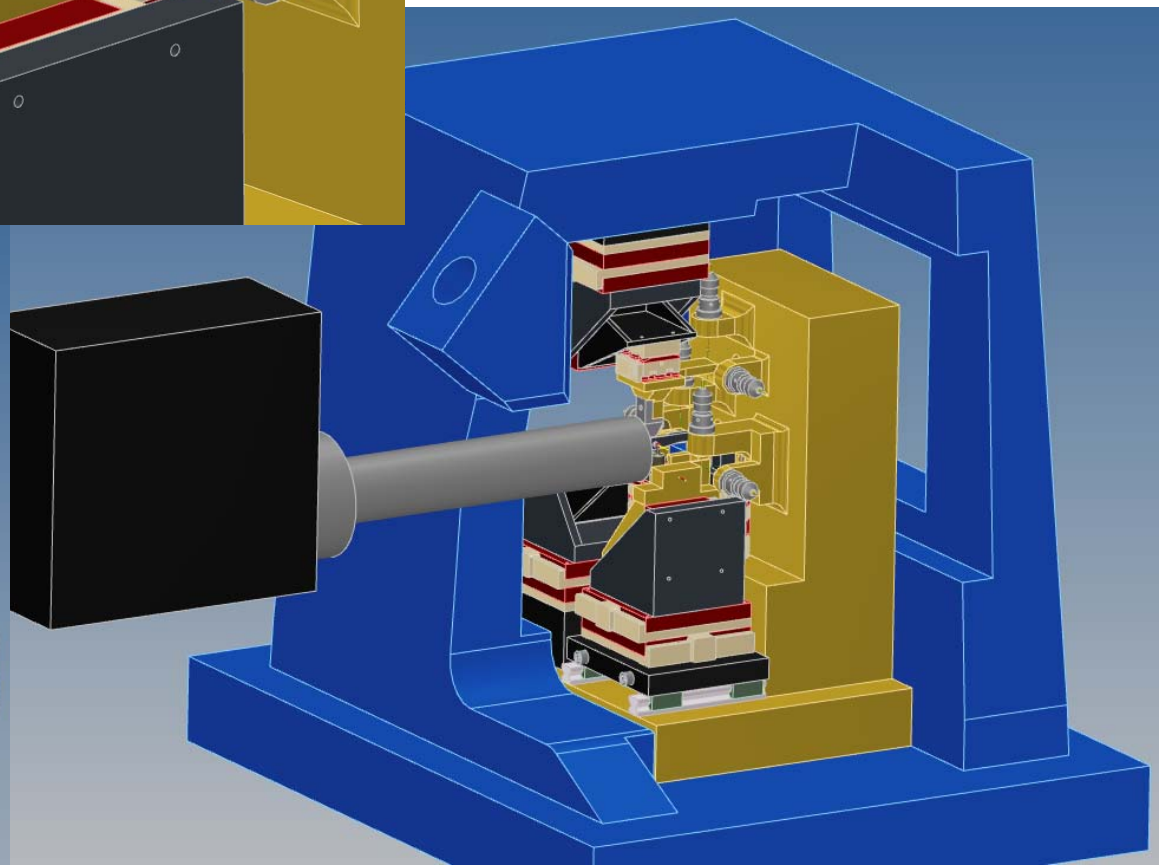
MLL Module

E. Nazaretski & E. Feldman

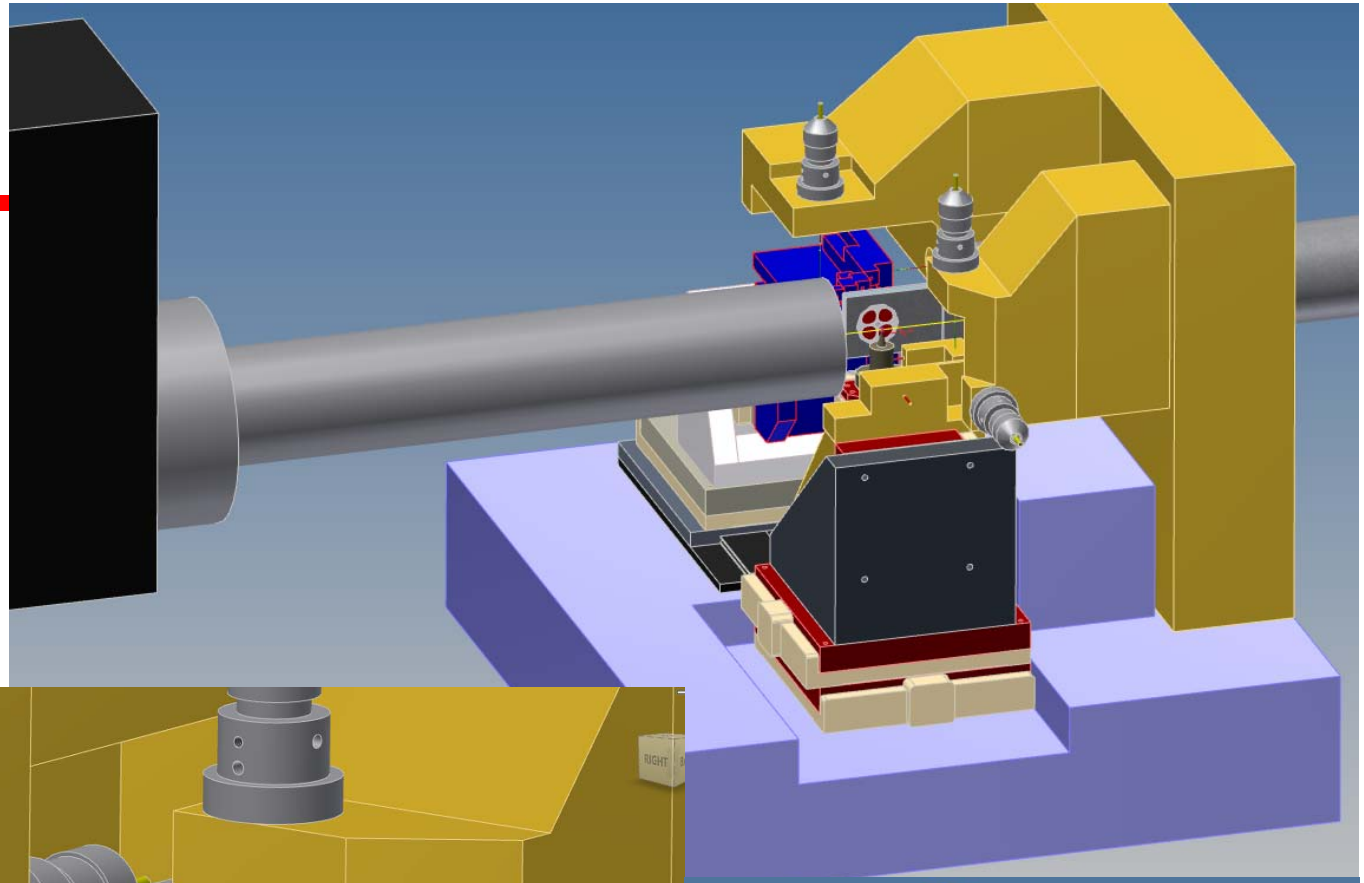


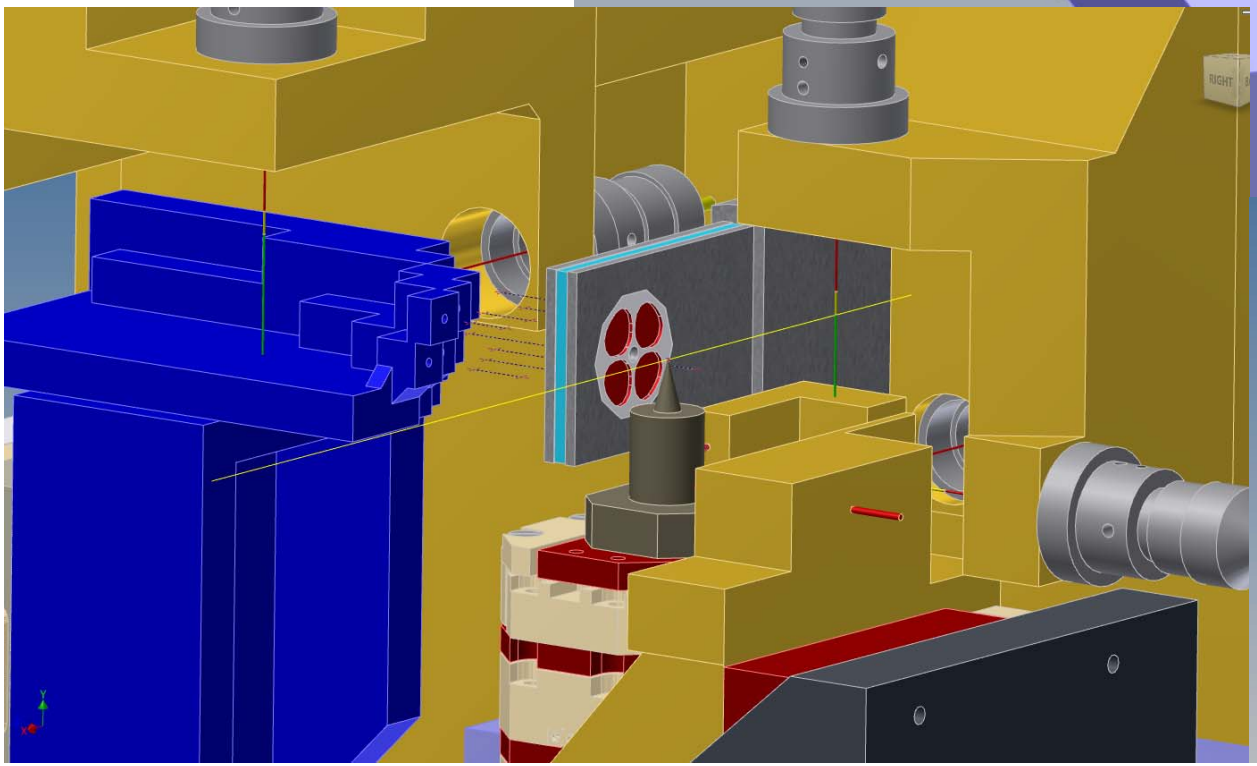


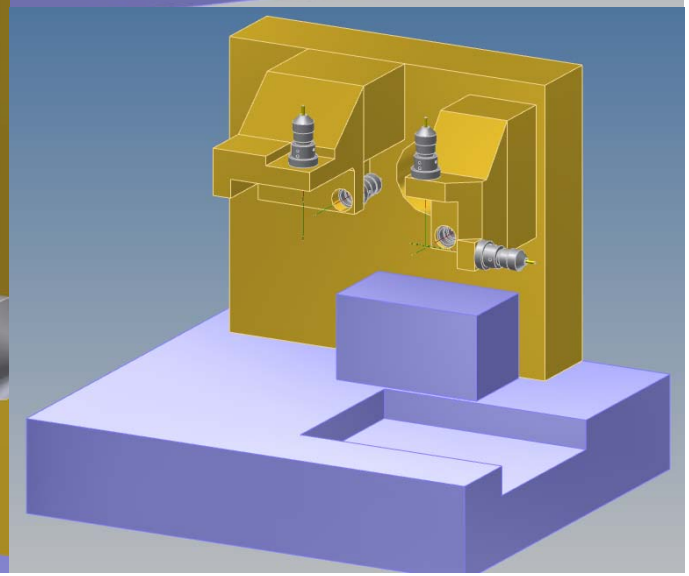


ZP Module

- OSA on XRF detector
- Two detectors for solid angle > 1 Sr !







Thank you for your attention