Precision engineering and nano-positioning R&D at NSLS-II

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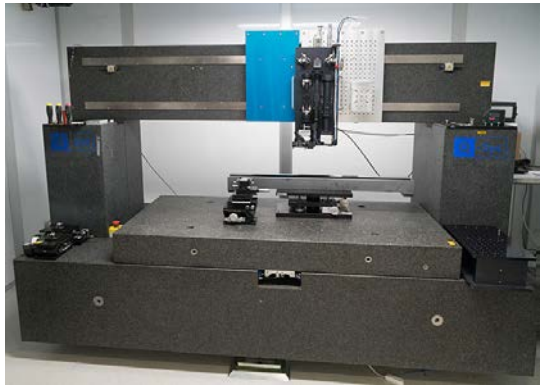
U.S. DEPARTMENT OF ENERGY | Office of Science
BROOKHAVEN NATIONAL LABORATORY

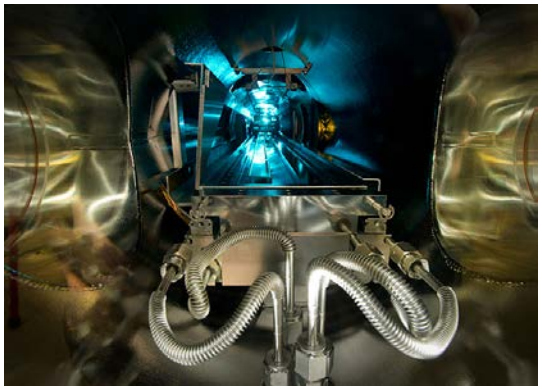
R&D program at NSLS-II

Facility supported R&D program is an integral part of the NSLS-II operations. The role of the R&D program is to support and foster NSLS-II leadership in the field of synchrotron science and technology. The scope of the R&D program consists of research and development activities primarily focused on *detectors development, optical metrology, development of advanced x-ray optics, precision engineering and nano-positioning, theory and simulations*.

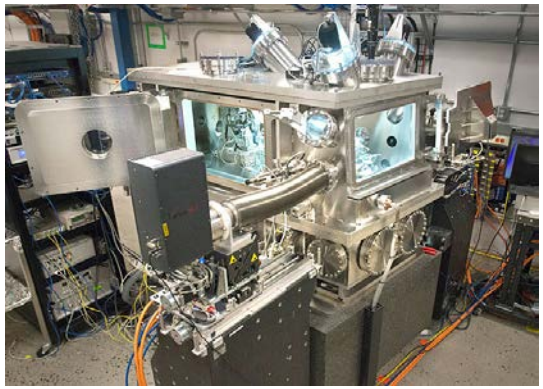
Optics Group https://www.bnl.gov/ps/groups/rd/



Optical and At Wavelength Metrology



Optics Fabrication

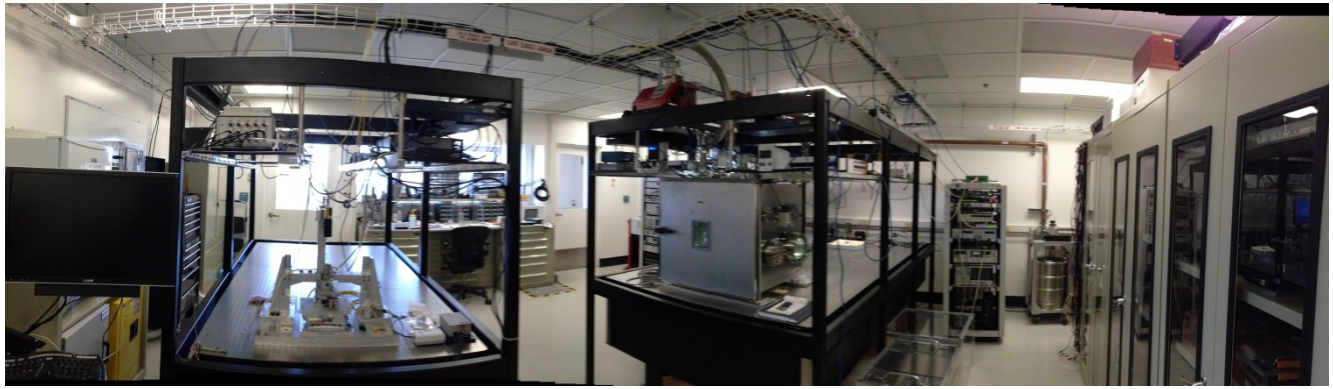


Precision Engineering and Nanopositioning

Precision Engineering and Nanopositioning R&D

Nikon LV150 Microscopy System
Signatone S-1160 Probe station

EOL JCM-6000PLUS NeoScope Benchtop SEM



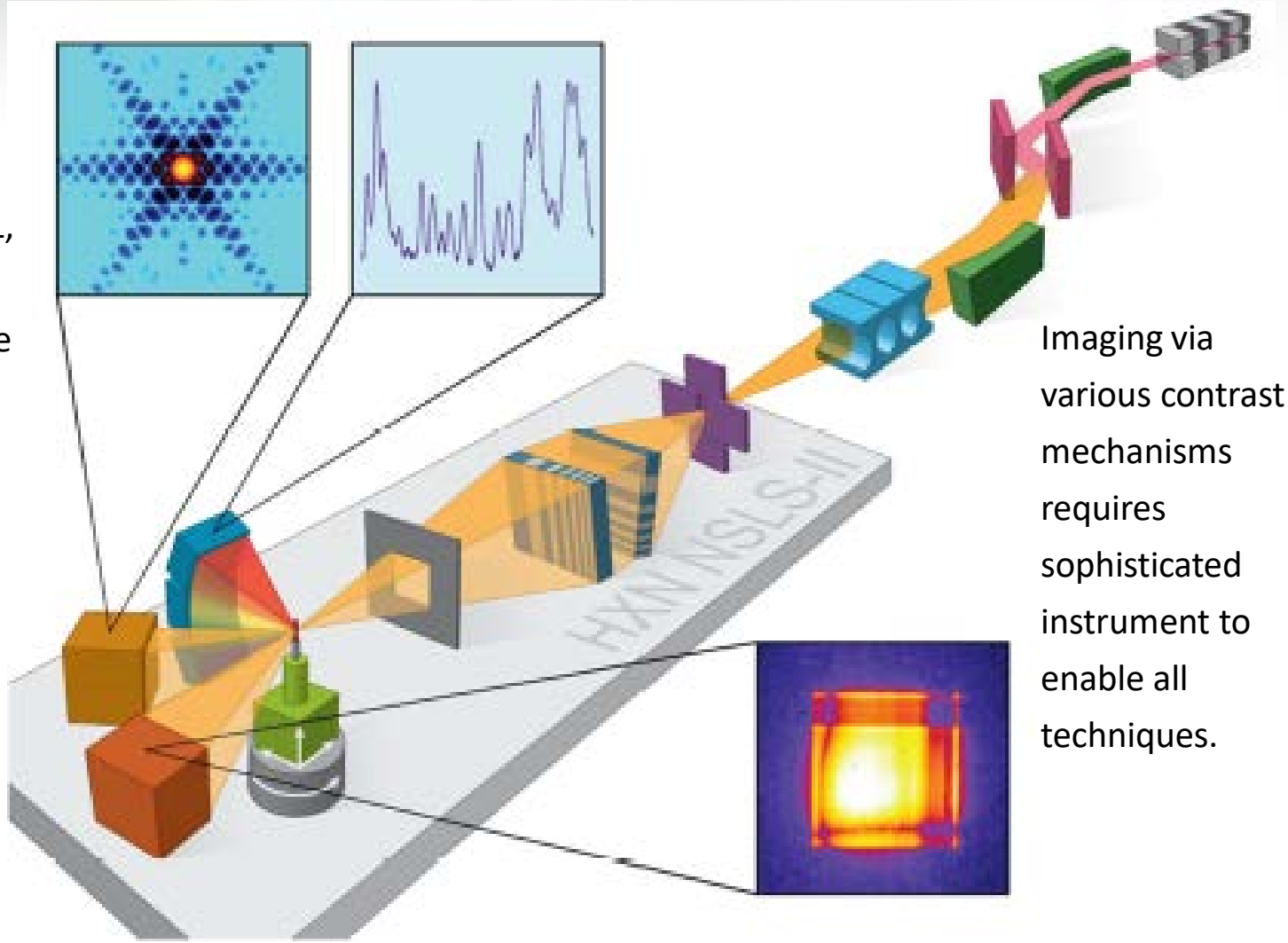
Hirox RH-2000 3D Microscope:
visualization and characterization of
3D objects with sub mm resolution

Attocube/Smaract interferometry systems
Range: 0 – 5 m, Resolution: >0.5 nm

Hard X-ray Nanoprobe beamline

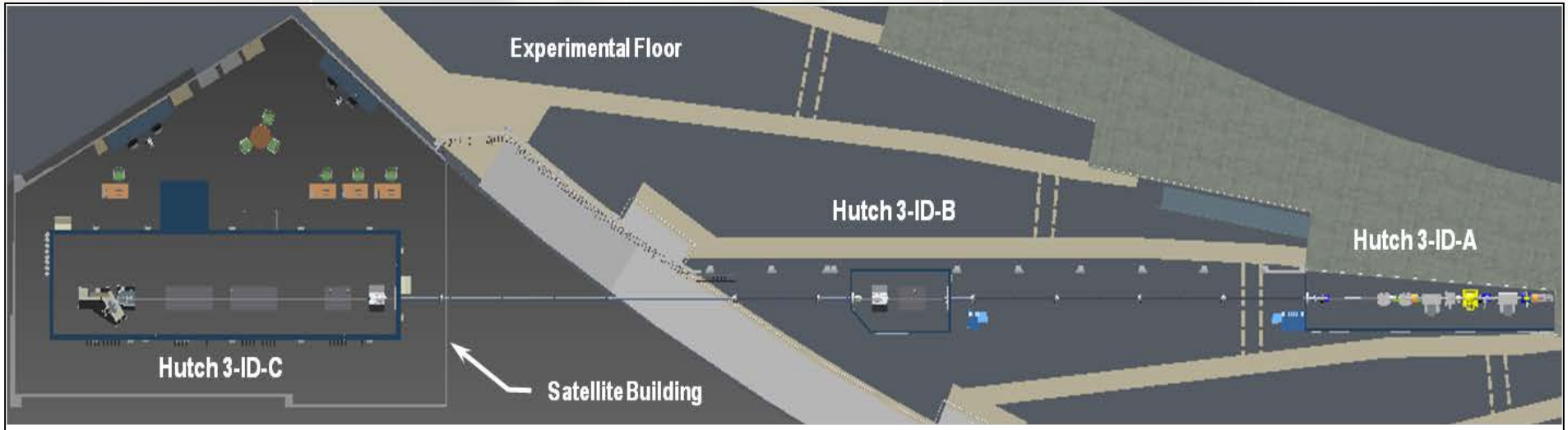
Unique Capabilities:

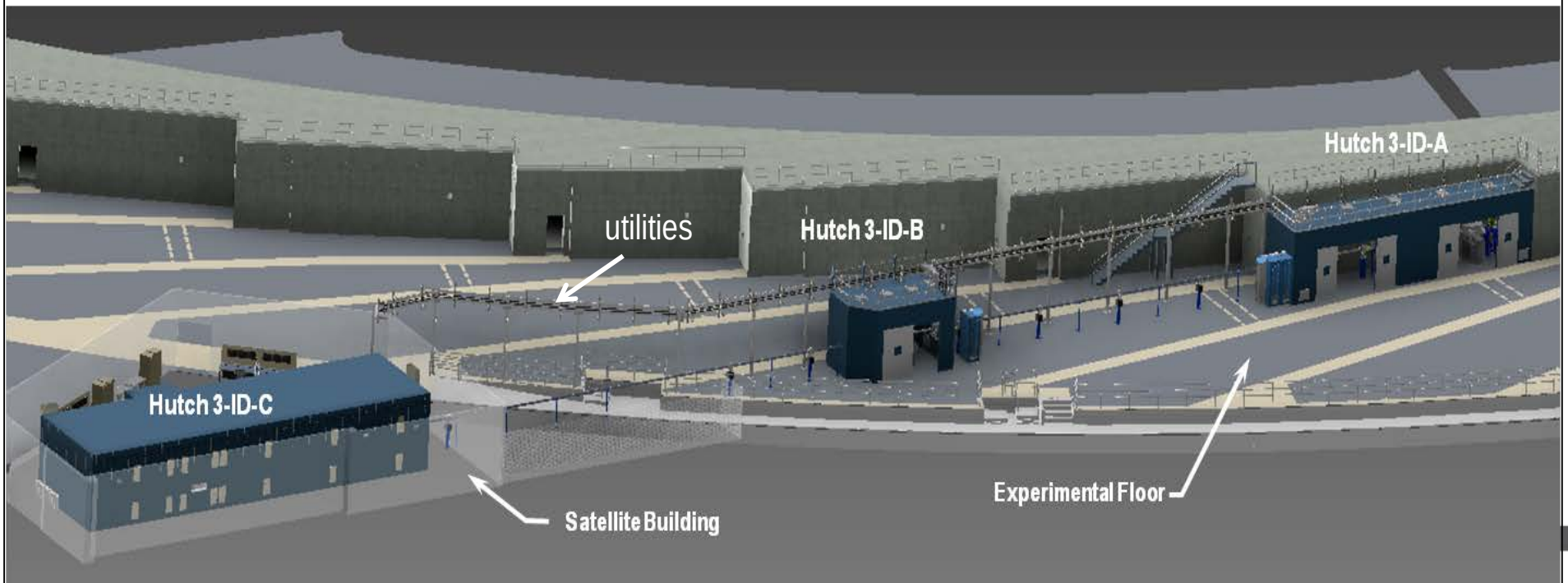
- High resolution (MLL, 10 nm) and high scientific flexibility (zone plate, 30nm)
- Imaging via different contrast mechanisms in 2D and 3D: absorption, phase-contrast, fluorescence, diffraction, Ptychography
- Sample temperature regulation



Imaging via various contrast mechanisms requires sophisticated instrument to enable all techniques.

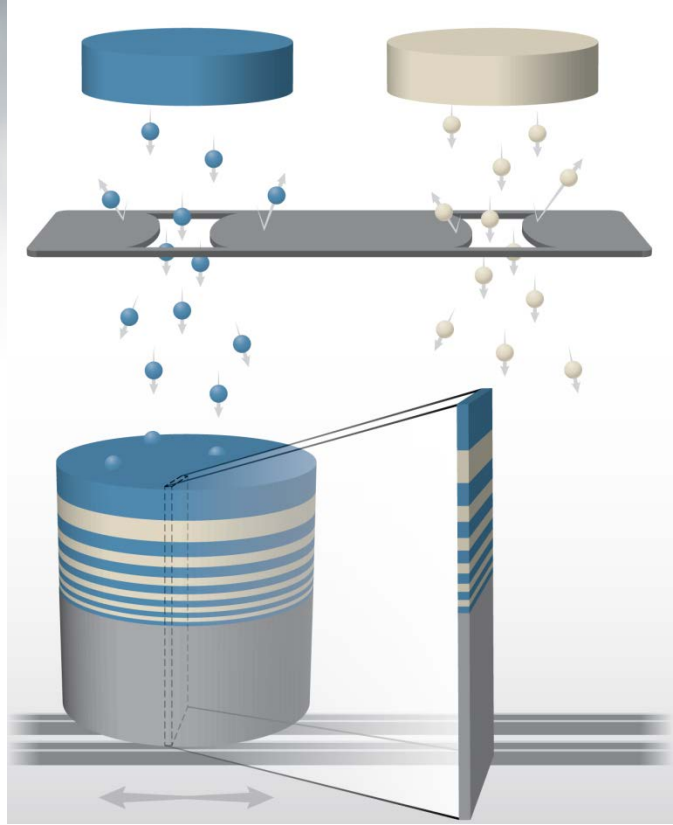
Overview of the HXN beamline

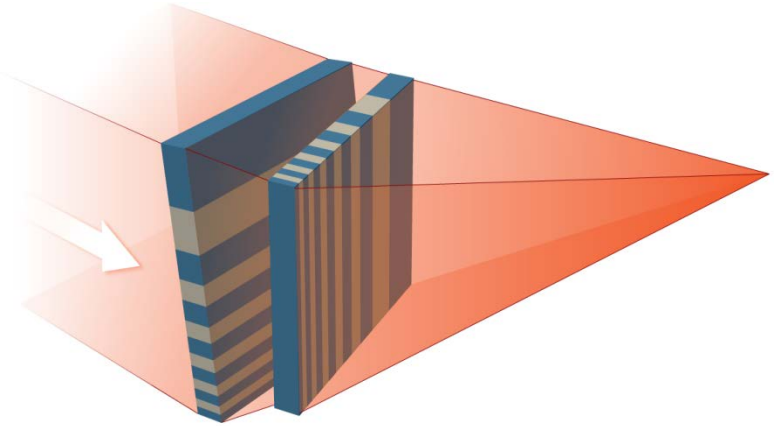




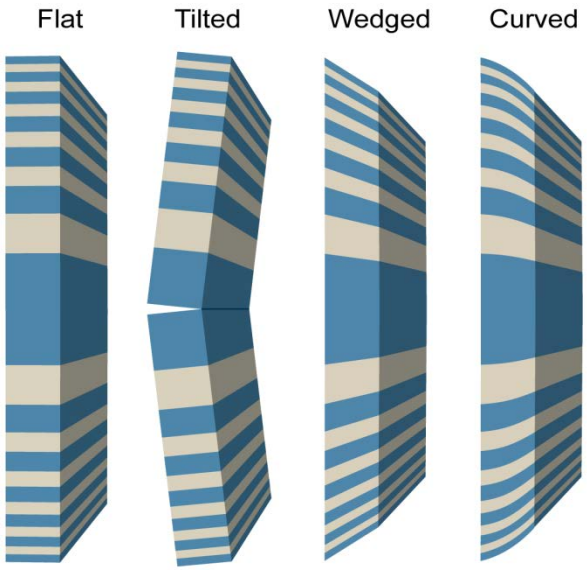
Multilayer Laue Lens (MLL)

- Overcome "aspect ratio" problem of ZP by thin-film deposition
  - → achieve virtually limitless aspect ratio
  - → ~1nm layer spacing feasible
- Volume optic (i.e. thick)
  - → high efficiency
  - → chromatic focusing
- Current material system: WSi_2/Si





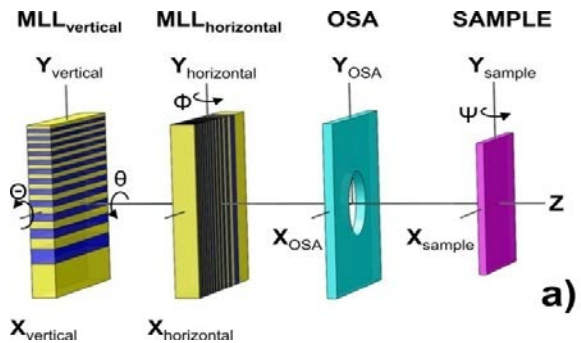
Typically:
7000-10000 layers
Aperture ~ 50 μm
Working distance
~ 1mm at 10 keV



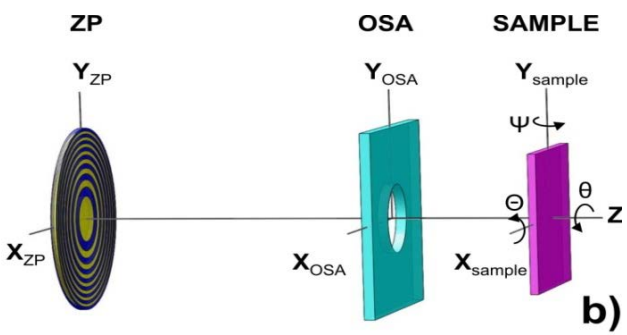
Off-axis focusing by half-MLLs, see MLLs overview
H. Yan, et al., J. Phys. D (2014), Synch. Rad. News (2016)

MLL/ZP scanning microscope

- **In-house development**
  - No commercially available MLL microscope
  - Commercial ZP microscopes are not suitable for MLL optics
  - Retrofitting of a commercial ZP microscope is not possible

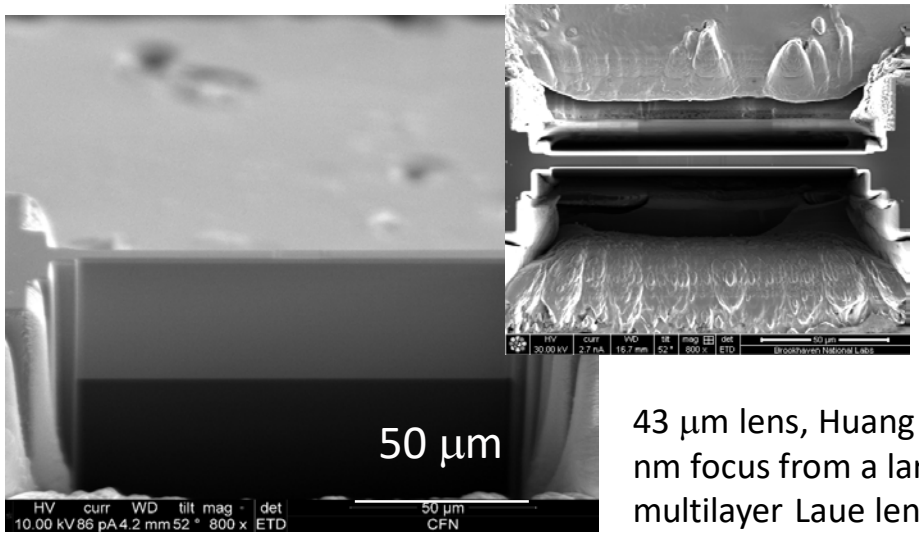


MLLs: 3 rotations + 5 translations



ZP: 2 translations

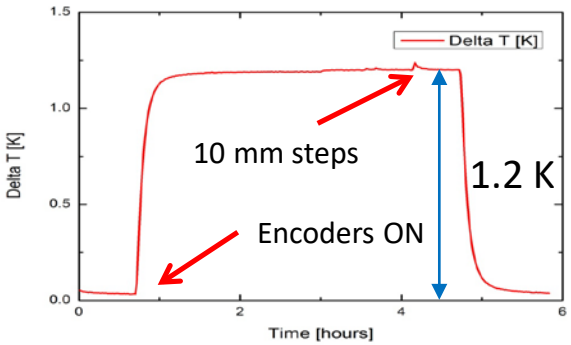
- Overcome "aspect ratio" problem of ZP by thin-film deposition
  - ✓ achieve virtually limitless aspect ratio
  - ✓ ~1 nm layer spacing feasible
- Volume optic (i.e. thick)
  - ✓ high efficiency
  - ✓ chromatic focusing
- Current material system: WSi_2/Si



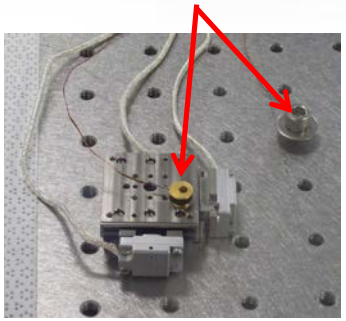
43 μm lens, Huang et. al., "11 nm focus from a large-aperture multilayer Laue lens", Scientific Reports, **3**, 35622 (2013)

Components evaluation : positioners and sensors

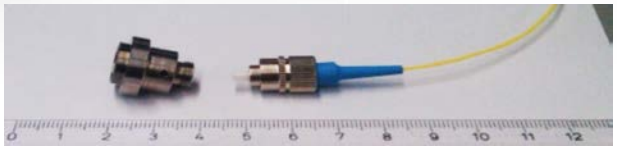
- Heat dissipation (encoders, motors)



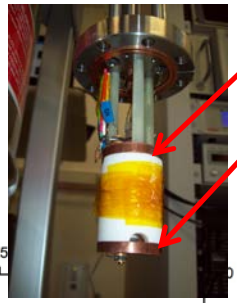
Thermometer A, B



- Fiber optic interferometry



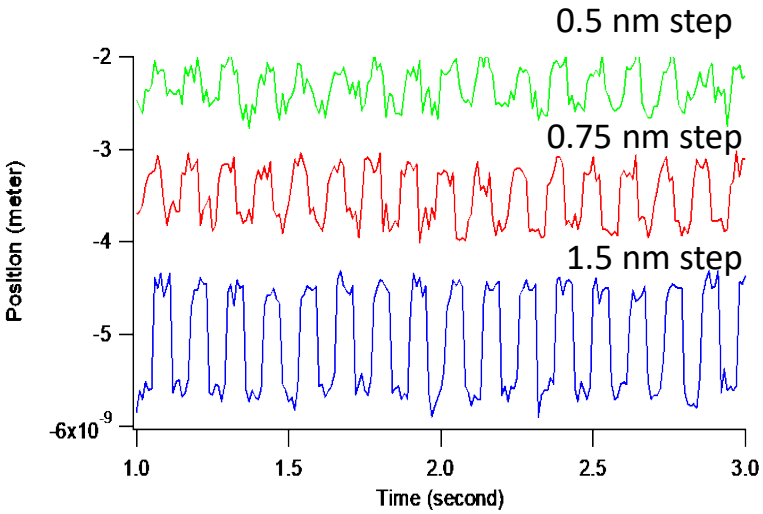
Compact design, vacuum compatibility, flexibility, wavelength stabilization

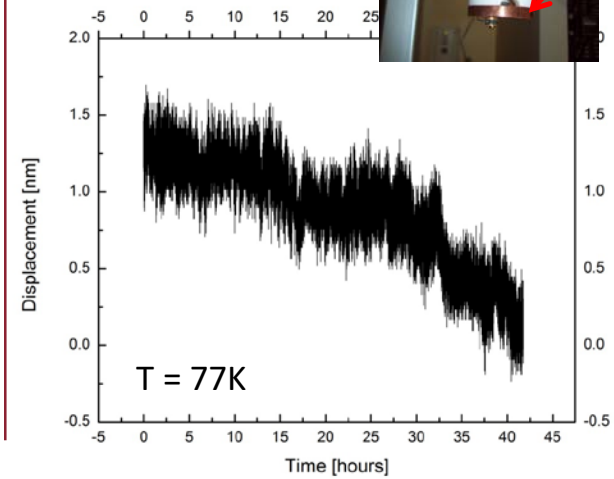


Interferometer head

Reflector

- Minimal incremental motion (piezo scanners)



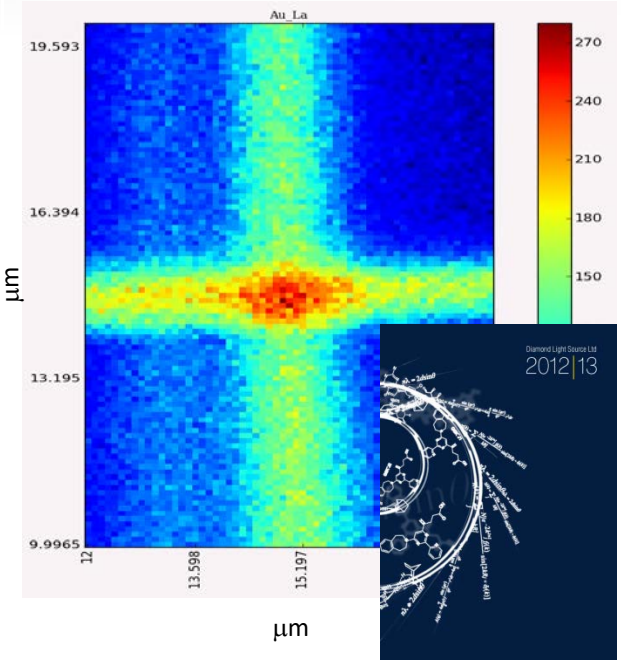


Prototype microscope for MLL characterization

MLL prototype instrument developed in collaboration with APS (D. Shu)

- Stand alone ambient system (capable of MLL alignment and sample scanning)
- System installed and tested at I13 Diamond Light Source, reliable scanning demonstrated over a few hours period of time

'Optomechanical design of a multilayer Laue Lens test-bed for 10-nm focusing of hard x-rays', D. Shu, E. Nazaretski, J. Kim, H. Yan, K. Lauer, B. Mullany, D. Kuhne, J. Maser, and Y. S. Chu', Journal of Physics: Conference Series **463**, 12029 (2013)

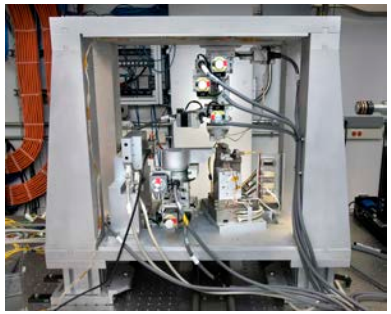


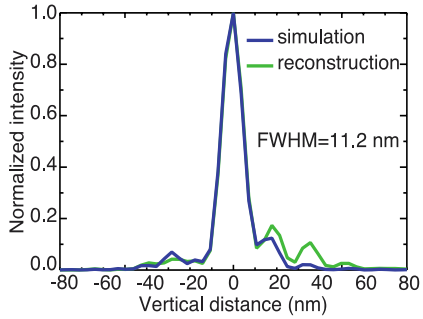
Fluorescence mapping of an Au test pattern at I13, DLS

'Characterization and performance of a prototype nm-scale spatial resolution scanning MLL microscope', E. Nazaretski, J. Kim, H. Yan, K. Lauer, D. Eom, D. Shu, J. Maser, and Y. S. Chu, *Rev. Sci. Inst.*, **84**, 033701 (2013)

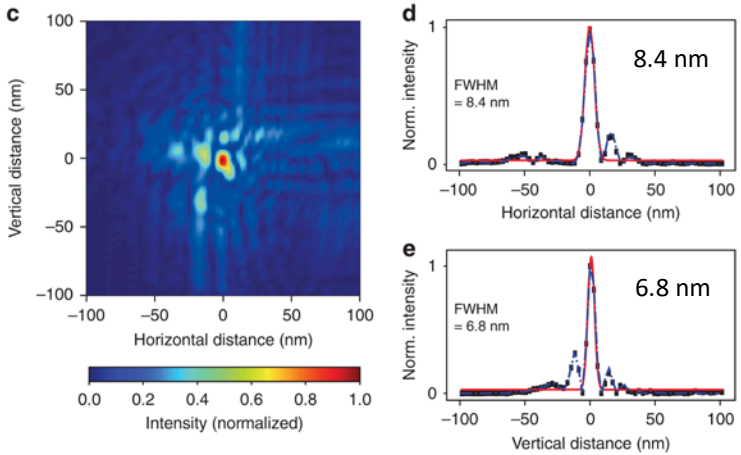
Ptychography instrument for MLL characterization; COC measurement systems for tomography

- Stand alone ptychography system developed to characterize focusing performance of MLL optics
- Characterization experiments performed at ID-34, APS



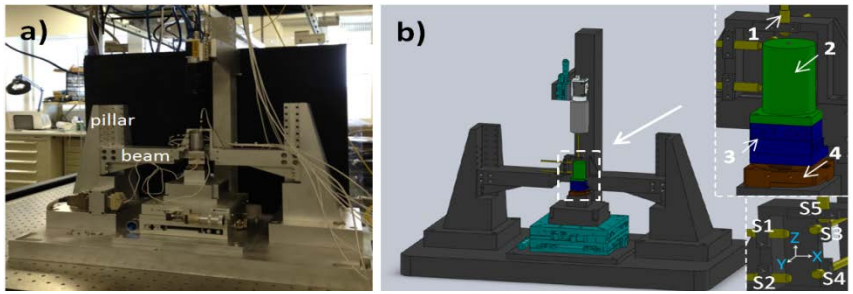


E. Nazaretski, et al., *Rev. Sci. Instr.* **85**, 33707 (2014), Huang et. al., *Scientific Reports*, **3**, 35622 (2013), 7.6 nm (2018)



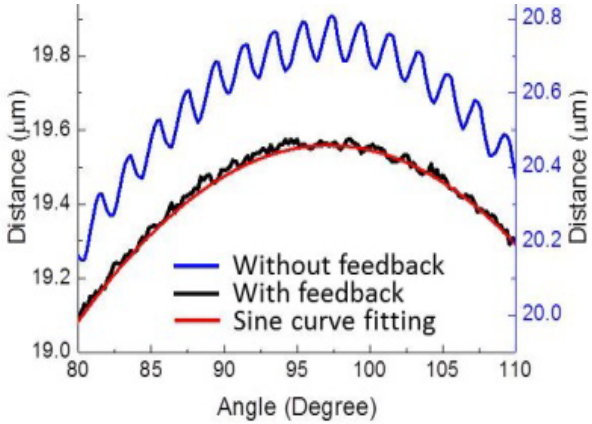
S, Bajt, et al., *Light: Science and Applications* , **7**, 17163 (2018)

- Dedicated system developed to map circle of confusion in various rotary stages – direct benefit for tomography experiments



'A high precision instrument for mapping of rotational errors in rotary stages', Weihe Xu, Kenneth Lauer, Y. S. Chu, and E. Nazaretski, *Journal of Synchrotron Radiation,* **21**, 1 (2014)

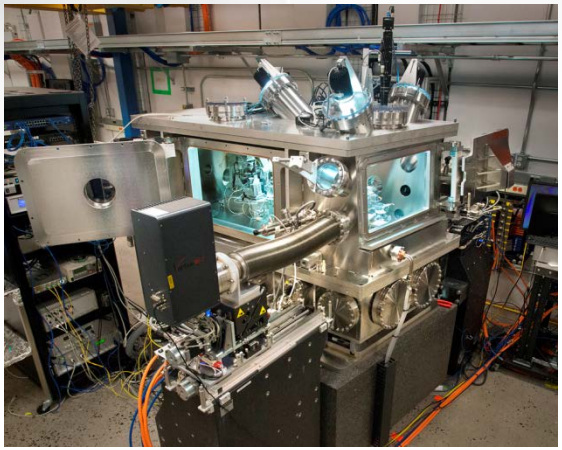
- Optics methods (interferometry) used in a prototype device to measure and correct rotational errors

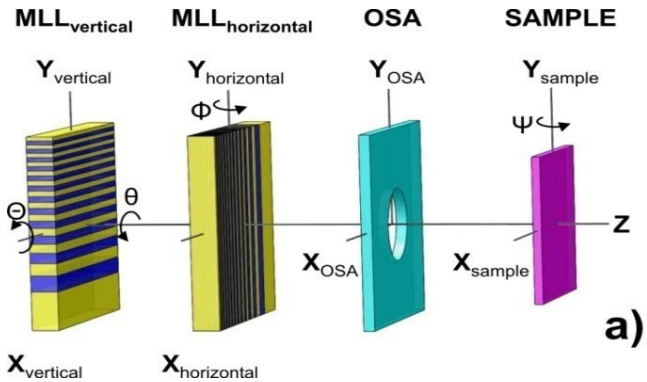


'Compact apparatus for reducing the circle of confusion down to 40 nm for x-ray nanotomography', J. Kim, K. Lauer, H. Yan, Y. S. Chu, and E. Nazaretski, *Rev. Sci. Inst.* **84**, 035006 (2013)

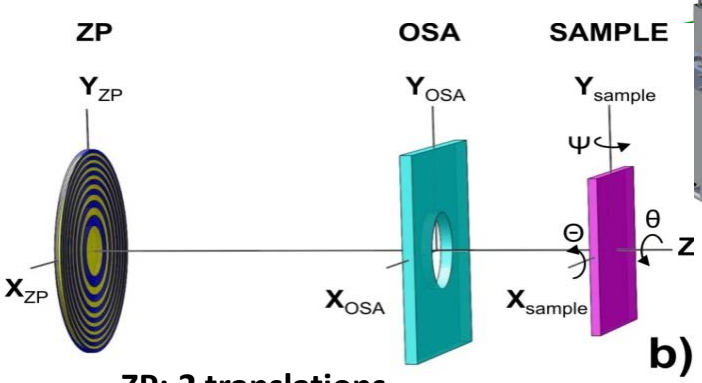
HXN endstation

- In-house development (in collaboration with D. Shu, APS)
- Allows scanning fluorescence, diffraction, ptychography, differential phase contrast, tomography measurements
- Targeted resolution: 10 nm and below (MLL), 30 nm ZP

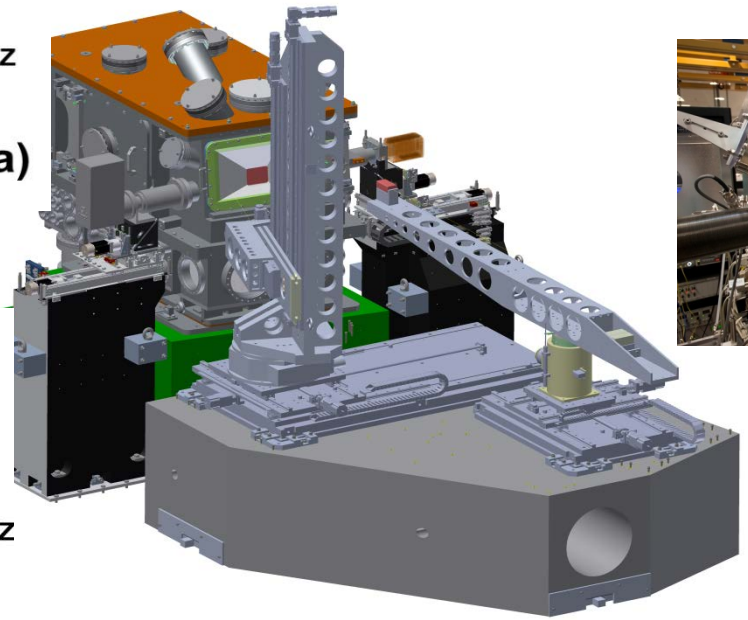


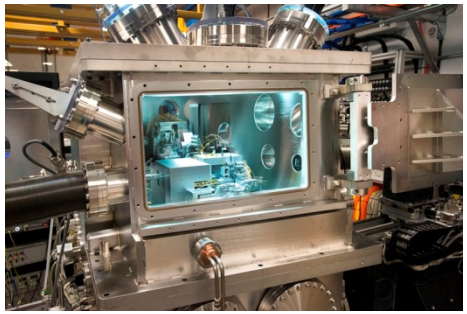


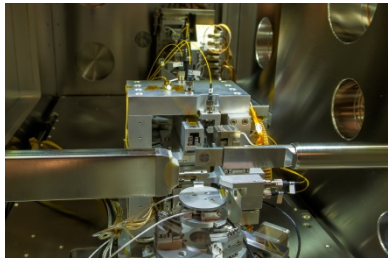
MLLs: 3 rotations + 5 translations



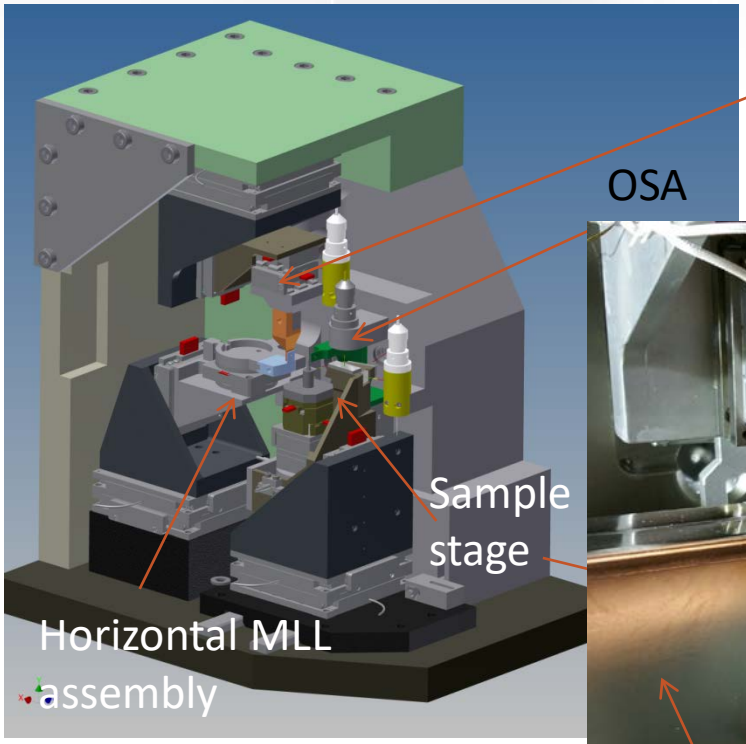
ZP: 2 translations



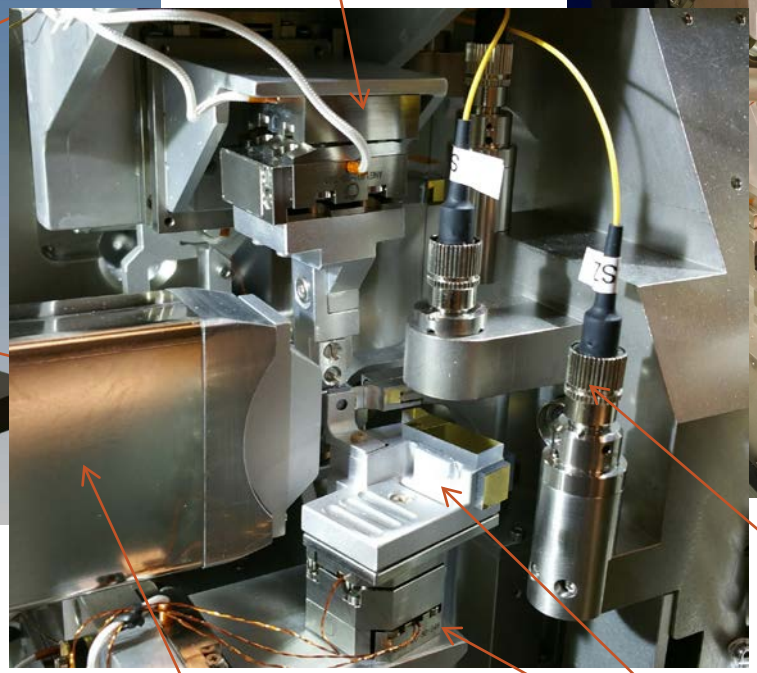


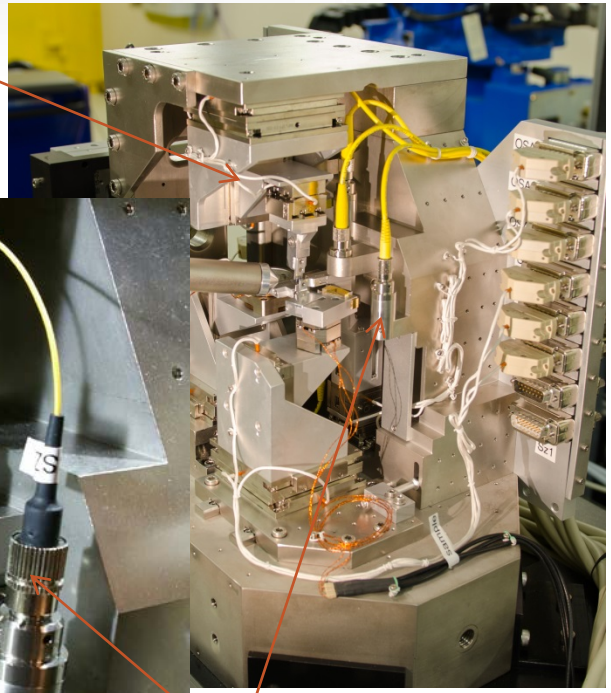


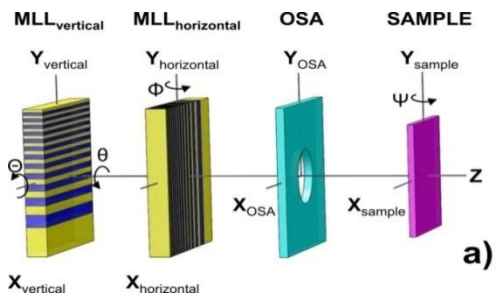
HXN endstation (MLL module)



Vertical MLL assembly





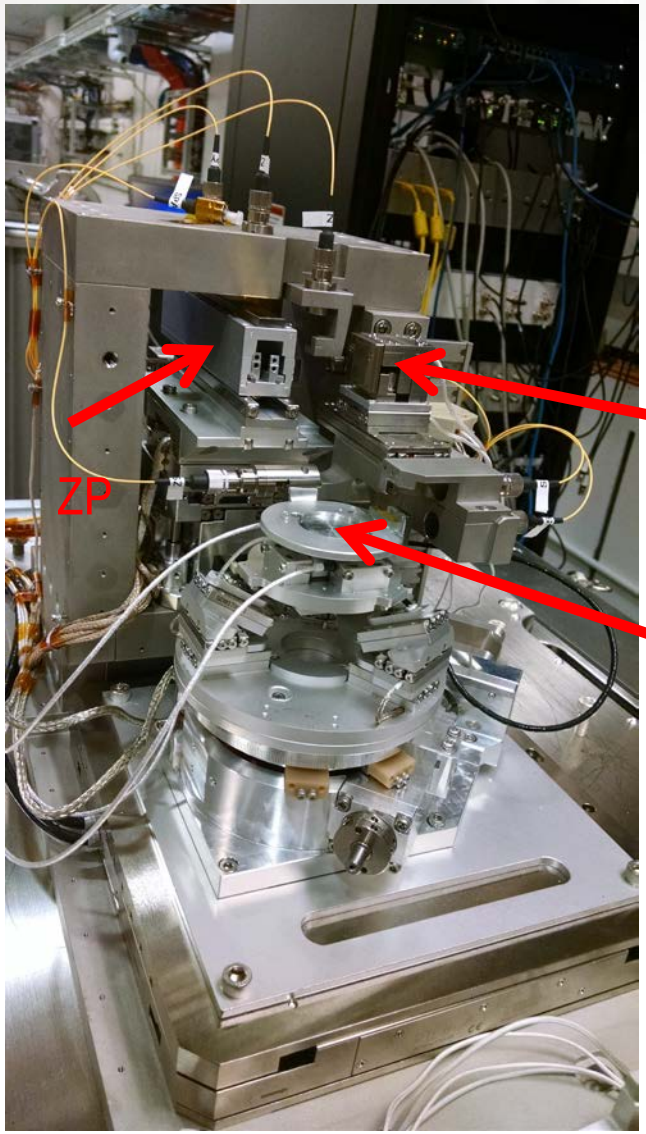


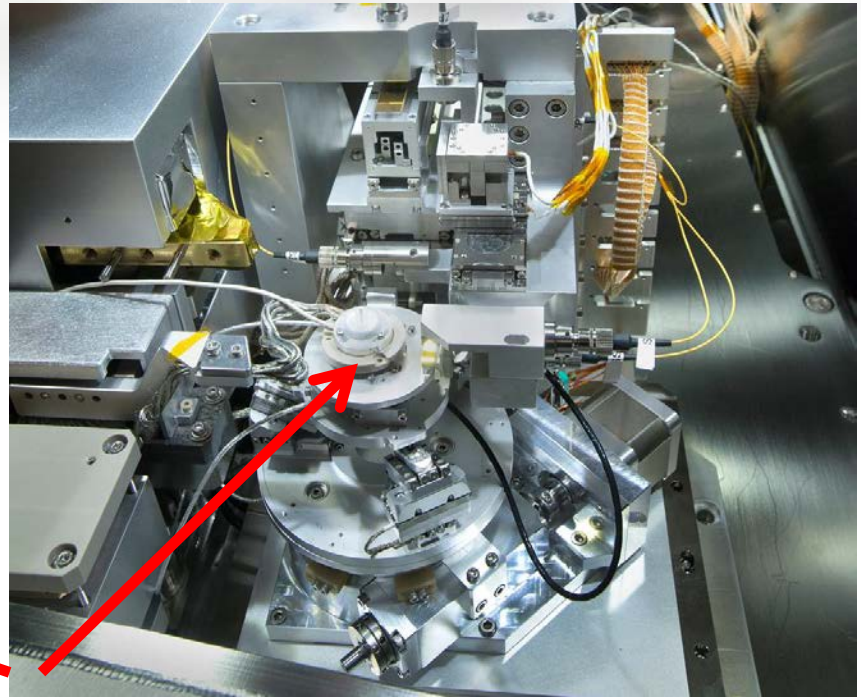
9 fiber-optic interferometer heads

Stick-slip piezo scanners/stages

XRF 3 element detector

HXN endstation (ZP module)





OSA

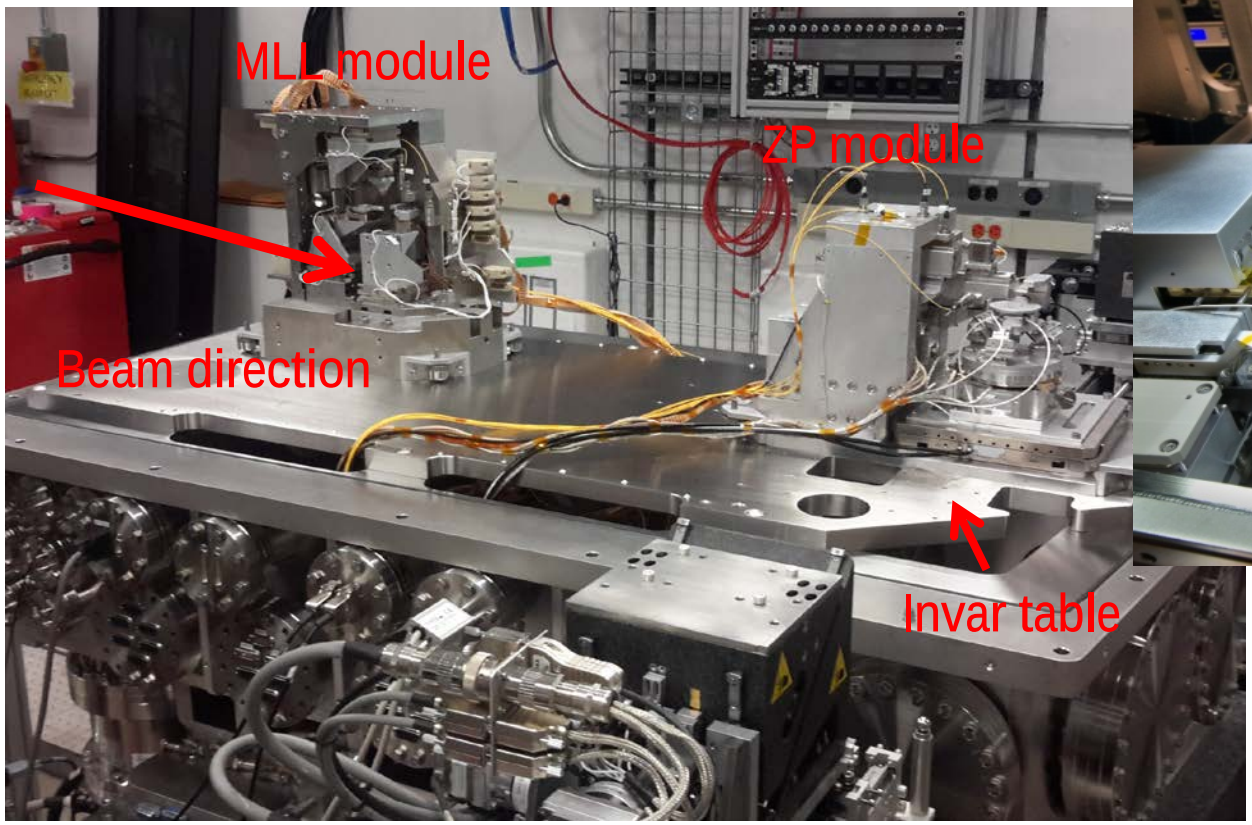
Sample

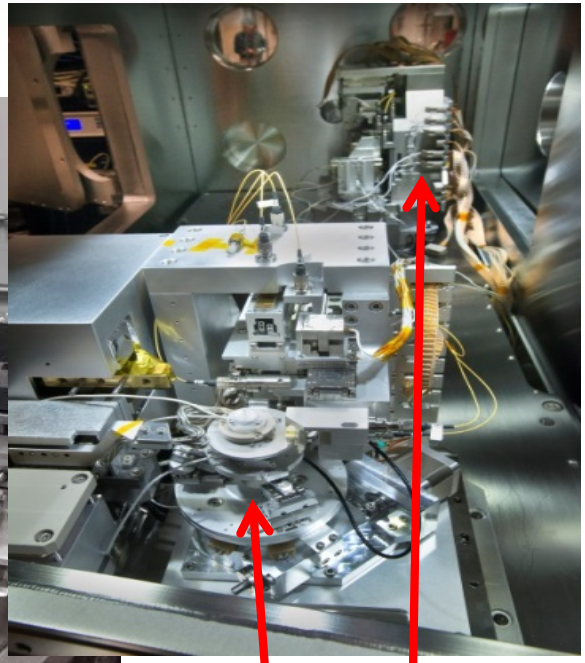
- Possible to install up to 4 Zone Plates
- Piezo-based actuation, interferometric control
- Invar frame for improved thermal stability

HXN endstation (modules exchange)

- **'Hot Swapping' of modules**

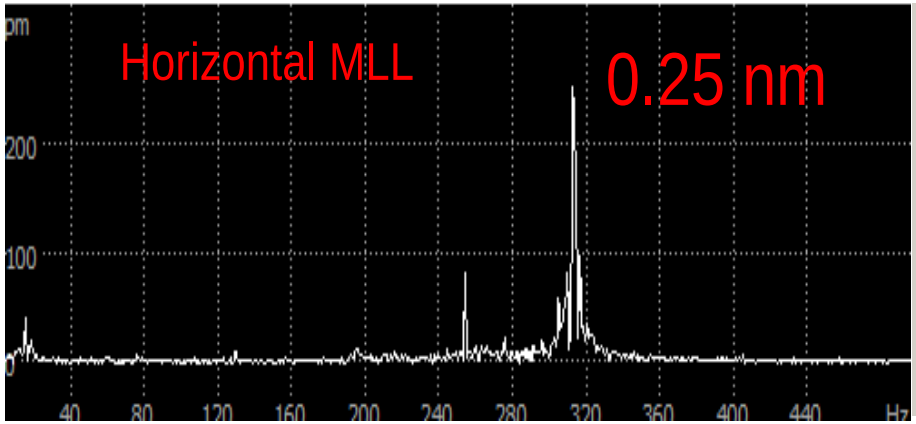
  - Allows to exchange position of both modules within the chamber without disconnecting any cables/interferometers.

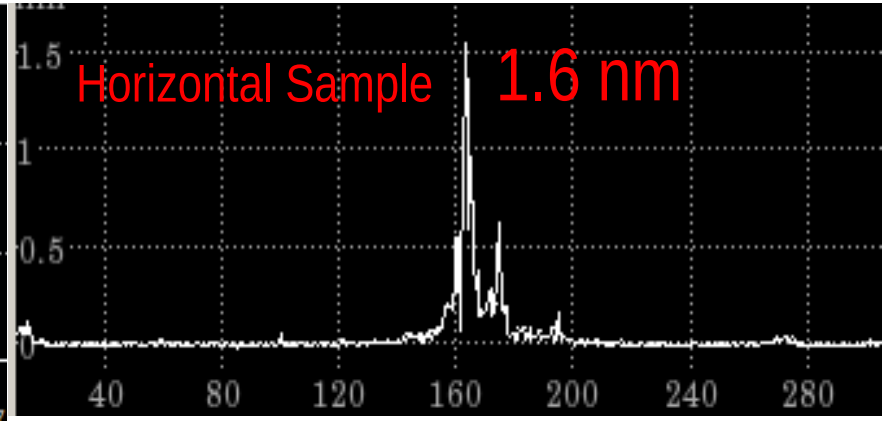


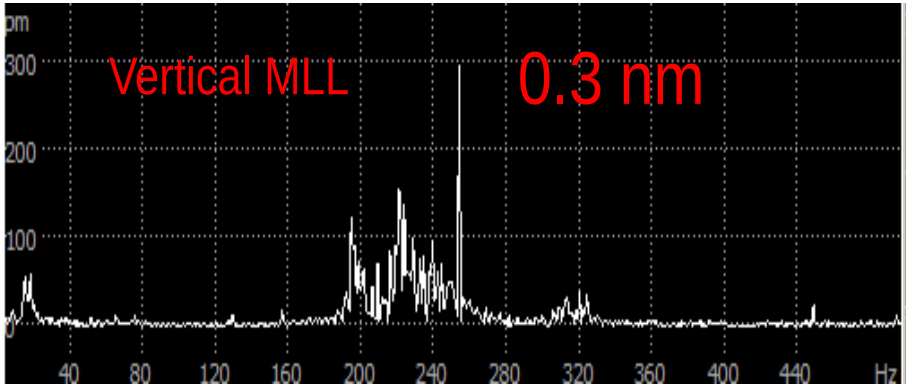


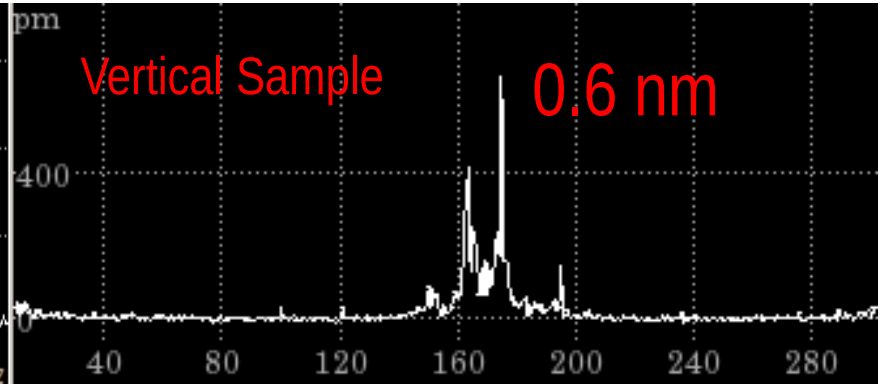
HXN endstation (MLL module)

- **Vibration characteristics for MLL optics and sample (0.6 - 1 ms time constant)**





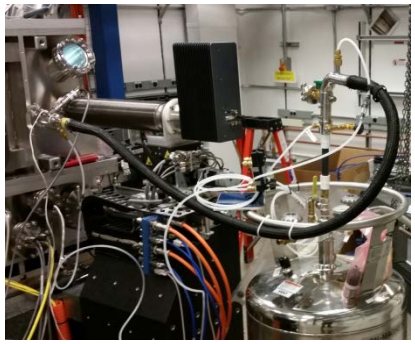


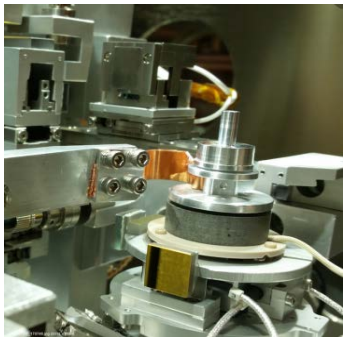


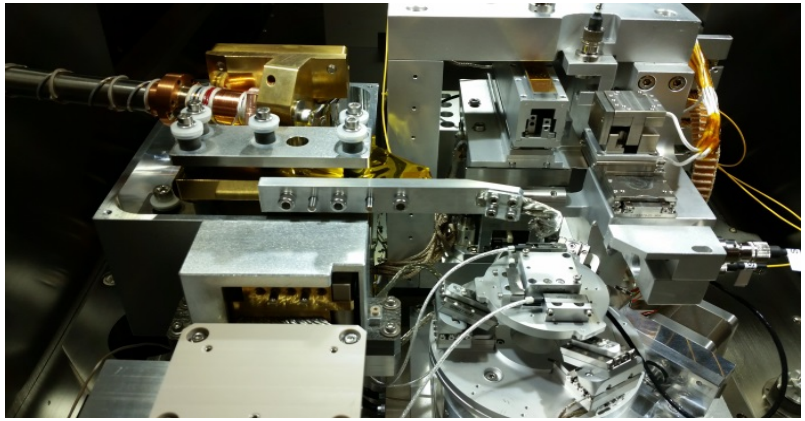
HXN endstation (TRS)

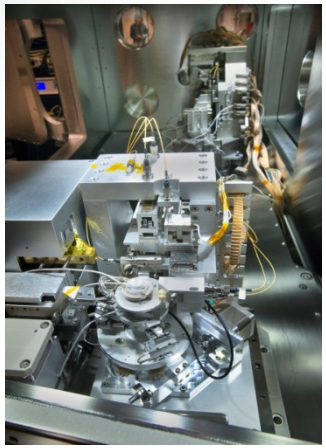
- **Temperature regulation system for HXN endstation**

- Cryogenic and heating holder for the ZP module (85 – 1100 K)
- TEC and heating holder for the MLL module (190 – 500 K)

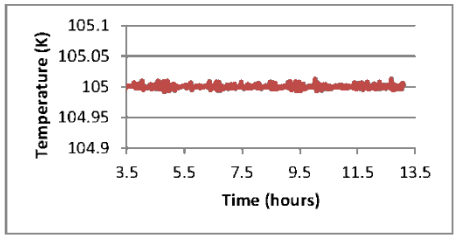


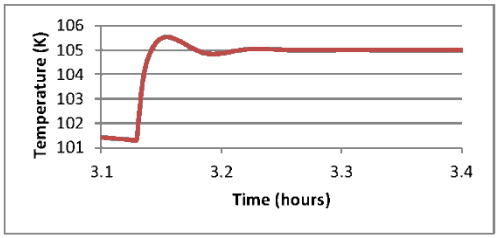


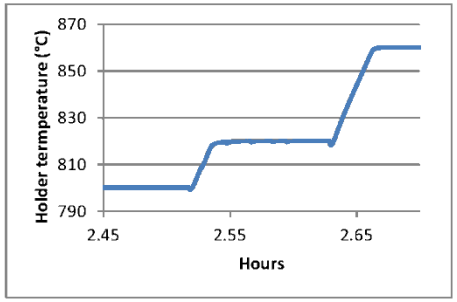


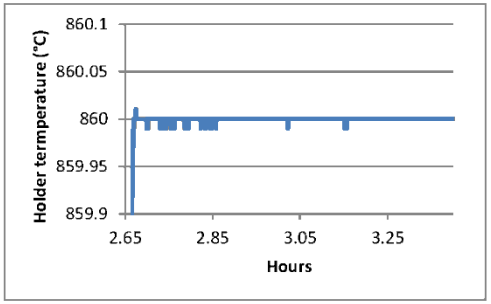


Thermal performance of the Cryo and Heating holders installed at the ZP module





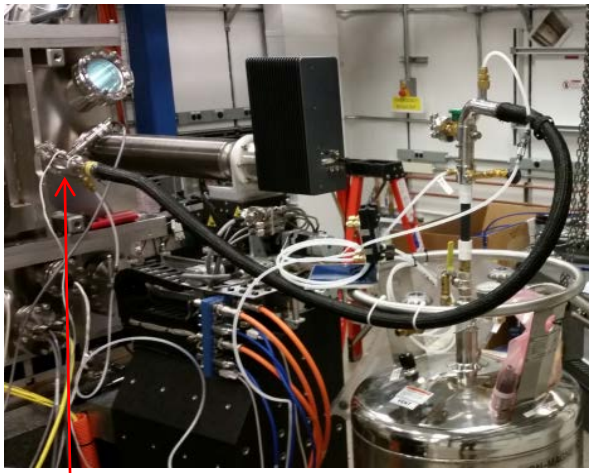




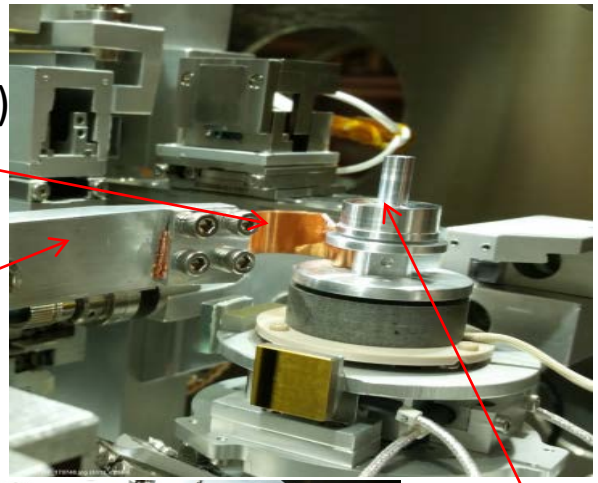
Commissioning of the Temperature Regulation System (TRS) for the HXN endstation

- **Temperature regulation system for the Zone Plate (ZP) module of the HXN endstation**

- Cryogenic and heating holders (90 – 1000 K), XRF (2D, tomography), diffraction



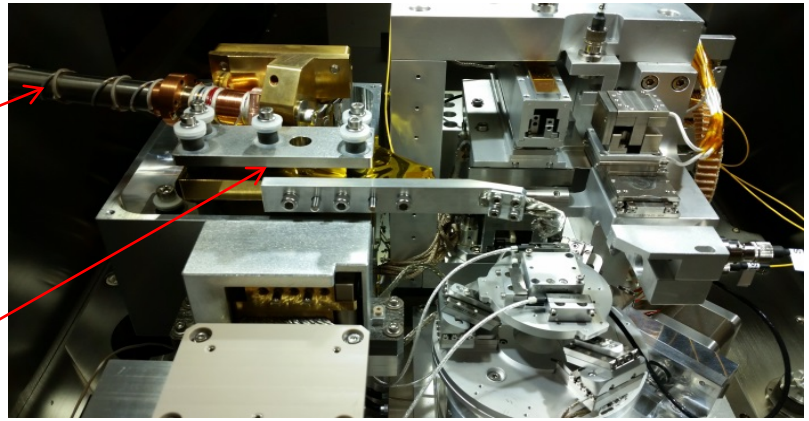
Braid to the sample (copper or graphite)



'cold arm'

Ultra-low vibration Flow cryostat

'Cold finger' inside the vacuum chamber

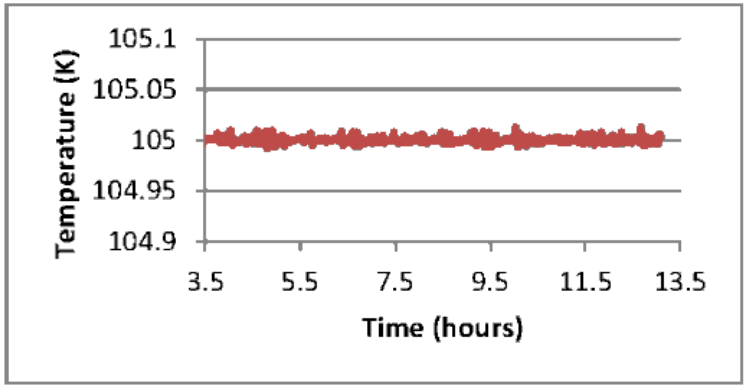


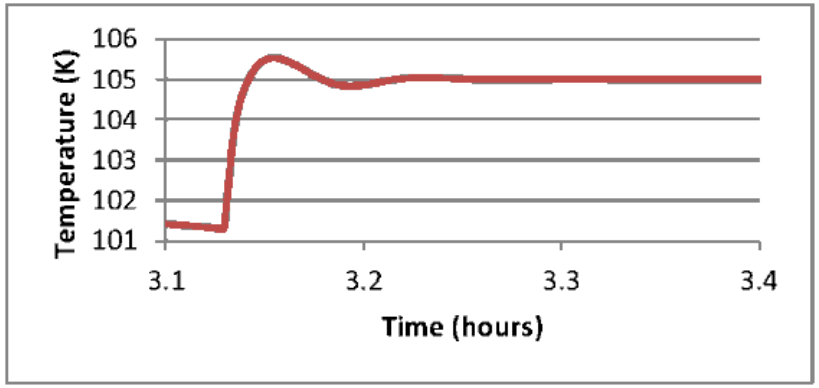
Multi-stage vibration isolation with a 'cold arm'

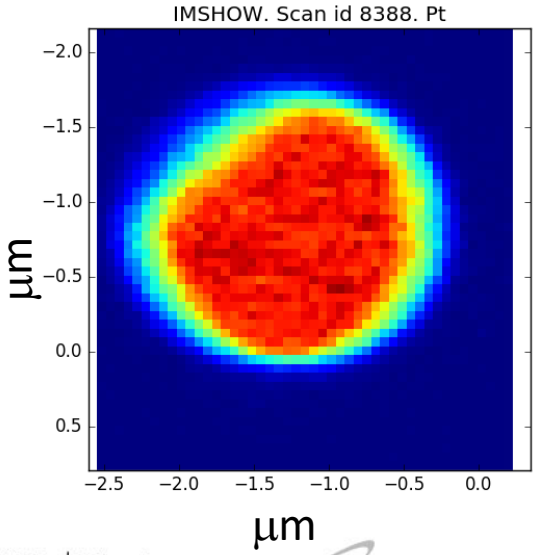
Sample mount with radiation shield around

Commissioning of the Temperature Regulation System (TRS) for the HXN endstation

- Thermal performance and XRF imaging

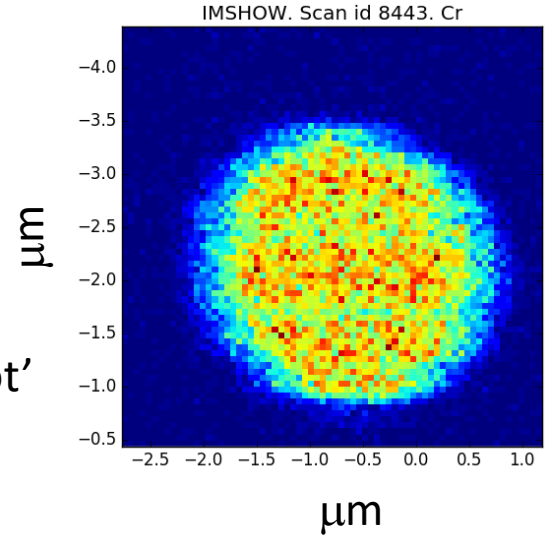




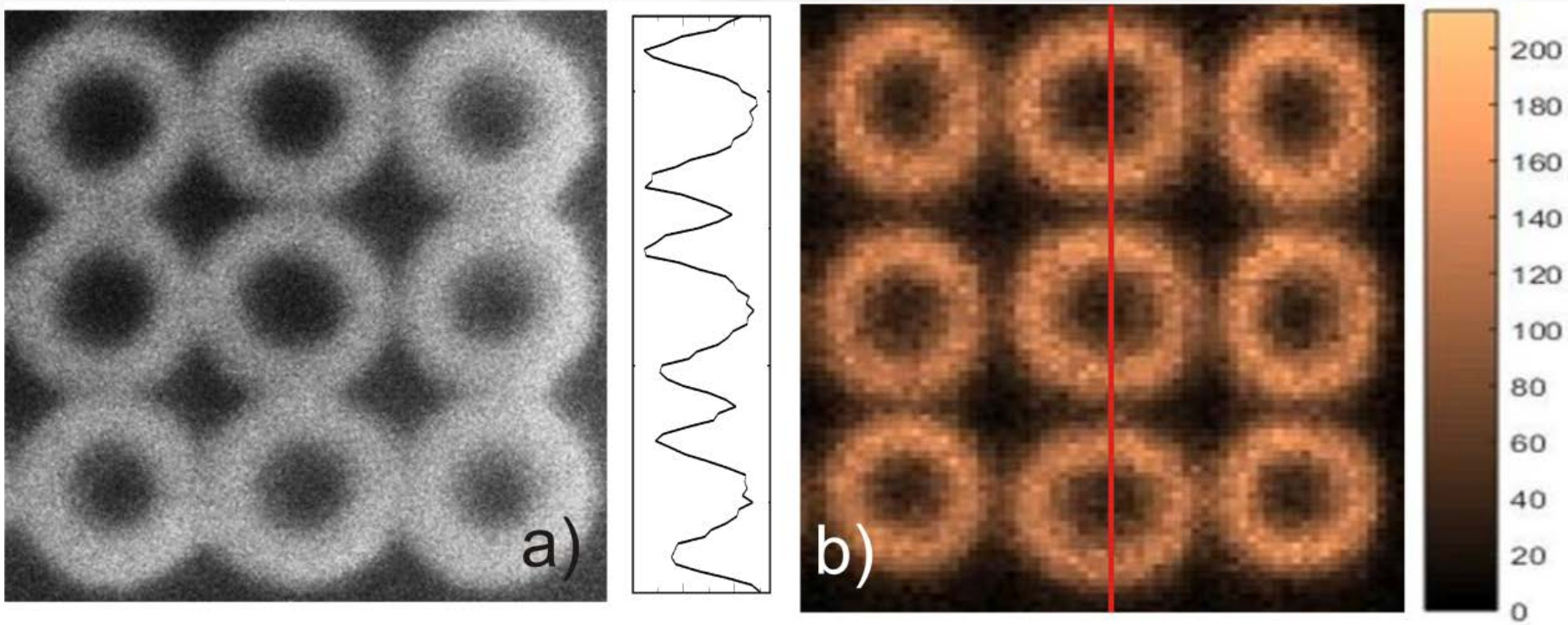


XRF, Pt 'dot'
T = 98.5 K

XRF, Cr 'dot'
T = 950 K

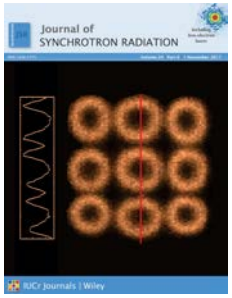


Early Commissioning Results



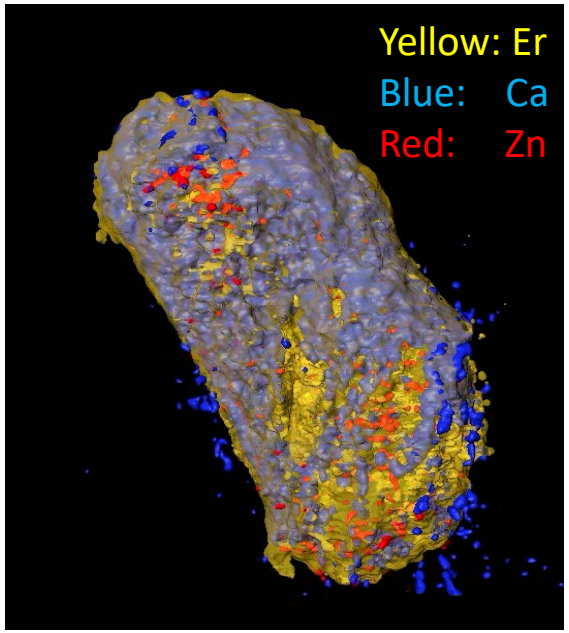
SEM image of the Pt test pattern. Circles are 80 nm in diameter and 20 nm line width. The height of the rings is 200 nm. XRF image (platinum L-edge) of the same test pattern, x-ray energy 12 keV, exposure time 0.2 s, 5 nm per pixel. Spatial resolution is ~ 12x13 nm^2

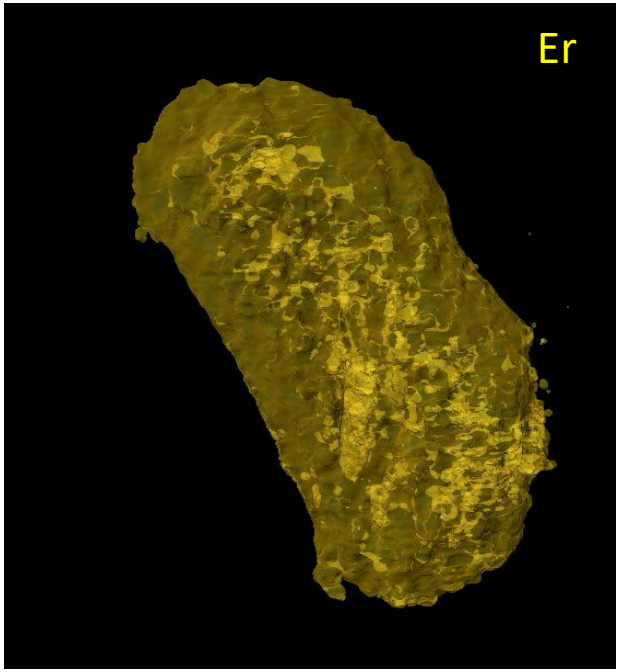
'Design and performance of an X-ray scanning microscope at the Hard X-ray Nanoprobe beamline of NSLS-II', E. Nazaretski, H. Yan, K. Lauer, N. Bouet, X. Huang, W. Xu, J. Zhou, D. Shu, Y. Hwu, and Y. S. Chu *Journal of Synchrotron Radiation* **24**, 1113 (2017)



Tomography of a biological cell

- Ca and Zn outline the shape of the eColi cell
- Er tags certain proteins and formes a shell structure on the surface of the cell
- Tomography also shows that some of Er has diffused into the inner part of the cell

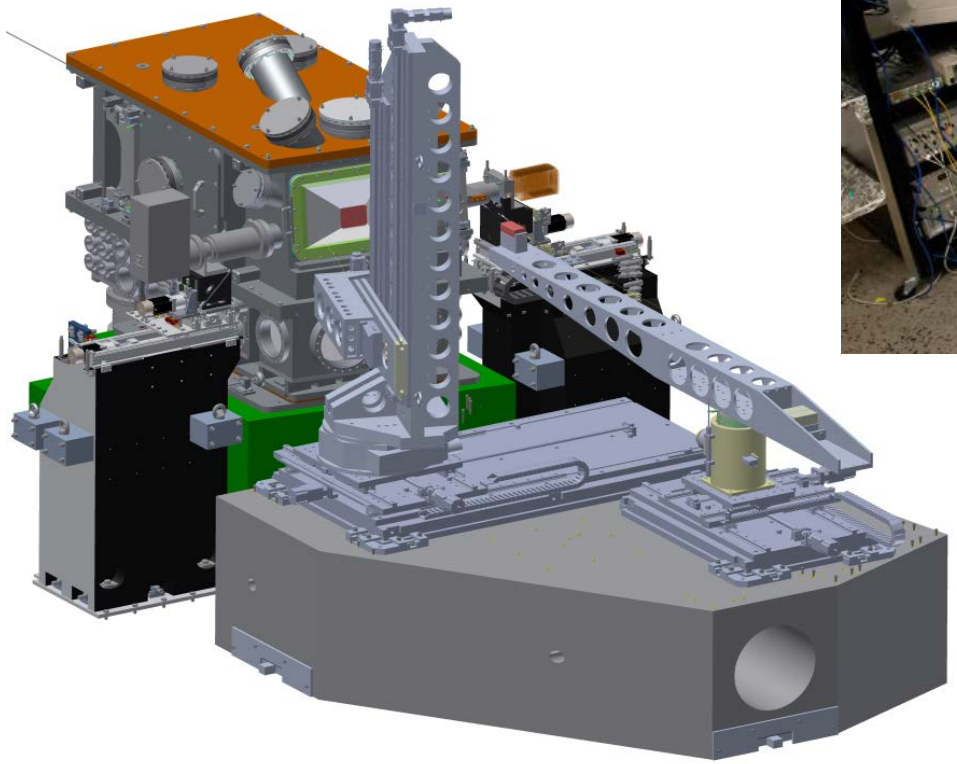


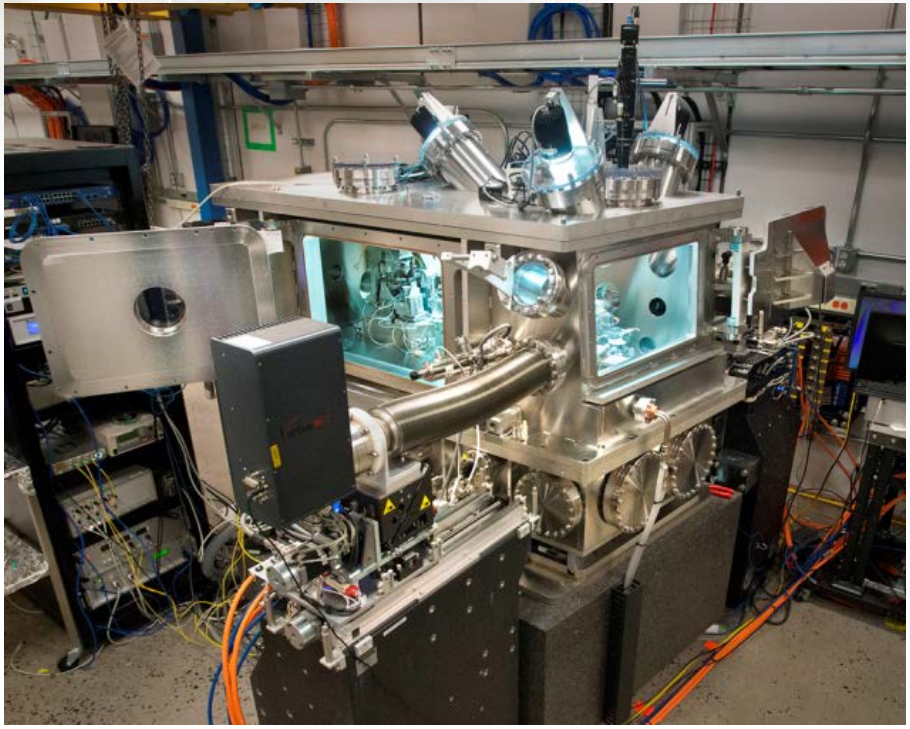


T Victor et al, X-ray Fluorescence Nanotomography of Single Bacteria with a Sub-15 nm Beam, *Sci. Rep.*, **8**, 13415 (2018).

Present status of HXN

- In user operation since 2015
- Allows scanning fluorescence, diffraction, ptychography, differential phase contrast, tomography measurements





- Routine sub-15 nm resolution using MLL and 30 nm using ZP

Microscopy TODAY 2016 Innovation Award

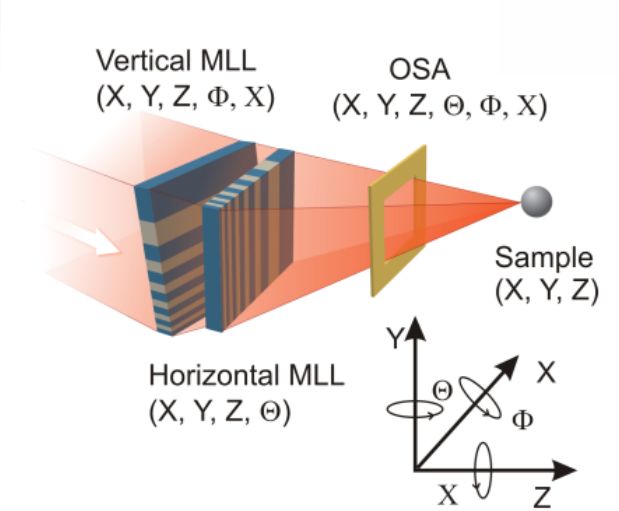
R&D 100 WINNER

Point Focusing of MLLs and Challenges

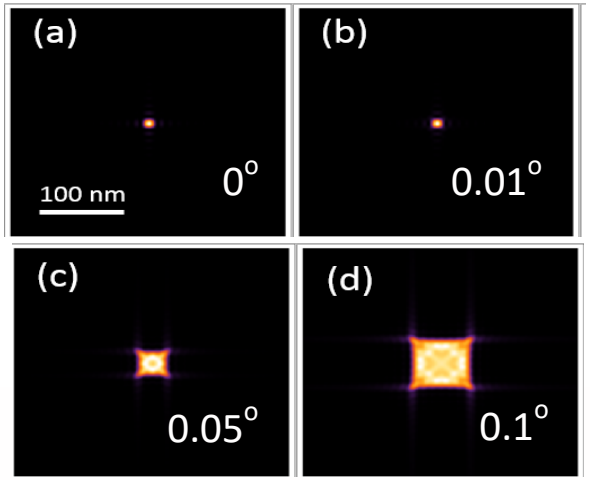
- **For point focusing:**
  - A pair of MLLs needs to be aligned orthogonally with respect to each other.

- **Alignment Challenges:**
  - A total of eight degrees of rotational and translational motions
  - 10 nm point focus: angular misalignment < 0.01°
  - Control the separation distance between two lenses
  - Long-term stability

- **Requires an extremely complex and stable instrument.**



Yan & Chu, J. Synchr. Rad, **20**, 89 (2012)

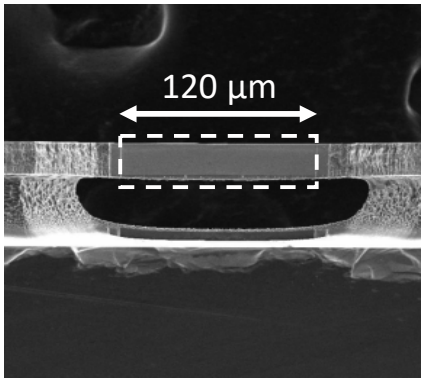


H. Yan et al., Optics Express, **25** (21), 25234-25242 (2017)

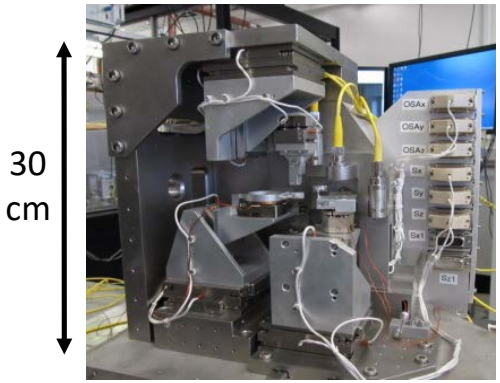
Complex Alignment Procedure and Instrument

- **Current System:**
  - A total of eight degrees of rotational and translational motions
  - A significant mismatch between optics dimensions and microscopy system
  - <u>The complexity of the instrument hinders its applications on other beamlines.</u>

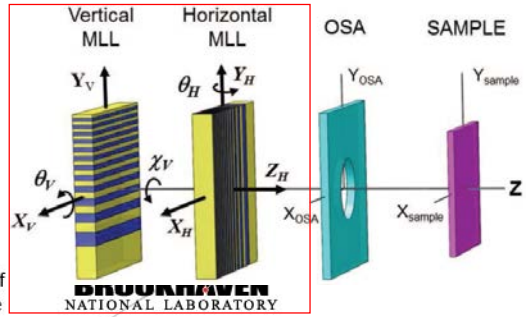
- ***MLL***

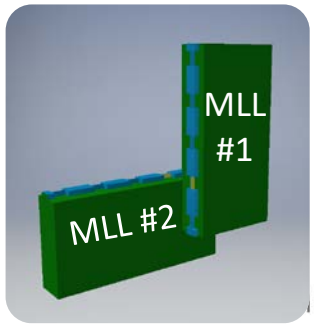


- ***MLL Microscope***



- **<u>To simplify alignment: monolithic 2D MLL optics</u>**





Development of monolithic 2D Multilayer Laue Lens nanofocusing optics

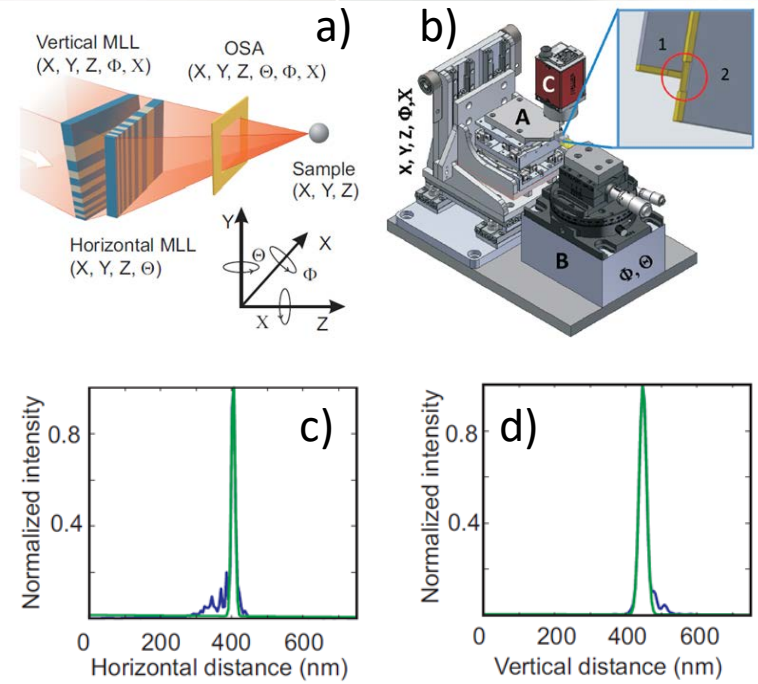
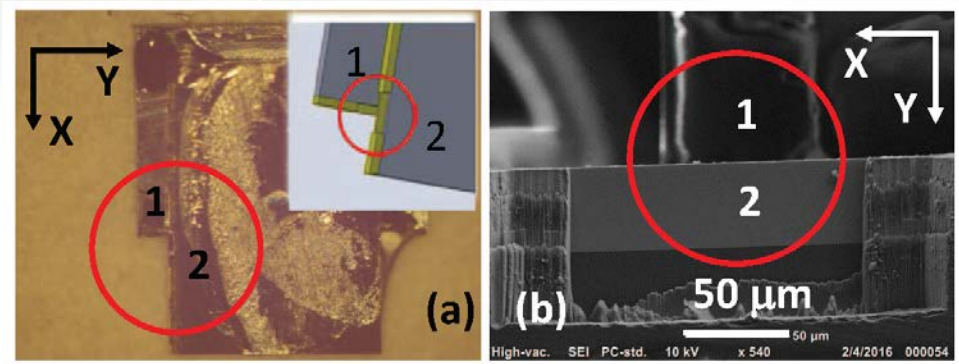


Figure (a) Schematic of the MLL microscope. (b) Computer Aided Design (CAD) model of the alignment system developed for MLL bonding. (c-d) Wavefront reconstructions at horizontal and vertical focal planes

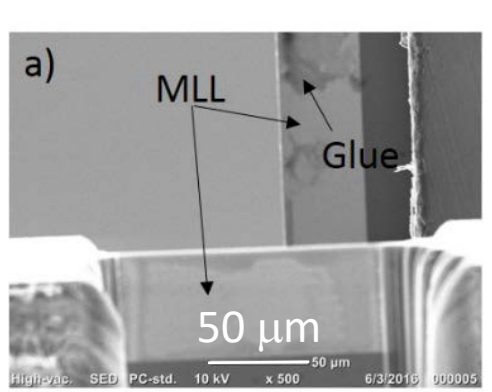


a) Optical microscope image of two bonded MLL's. The overlapping area is marked with a circle. b) SEM image of two bonded lenses

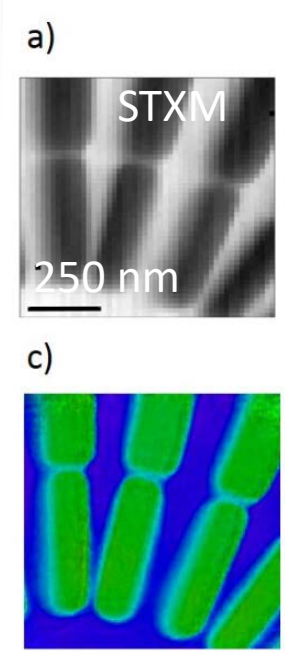
- Focus sizes are 12 nm and 24 nm at horizontal and vertical focal planes, respectively.
- Horizontal and vertical focal planes are separated by 221 µm, consistent with interferometer measurement.

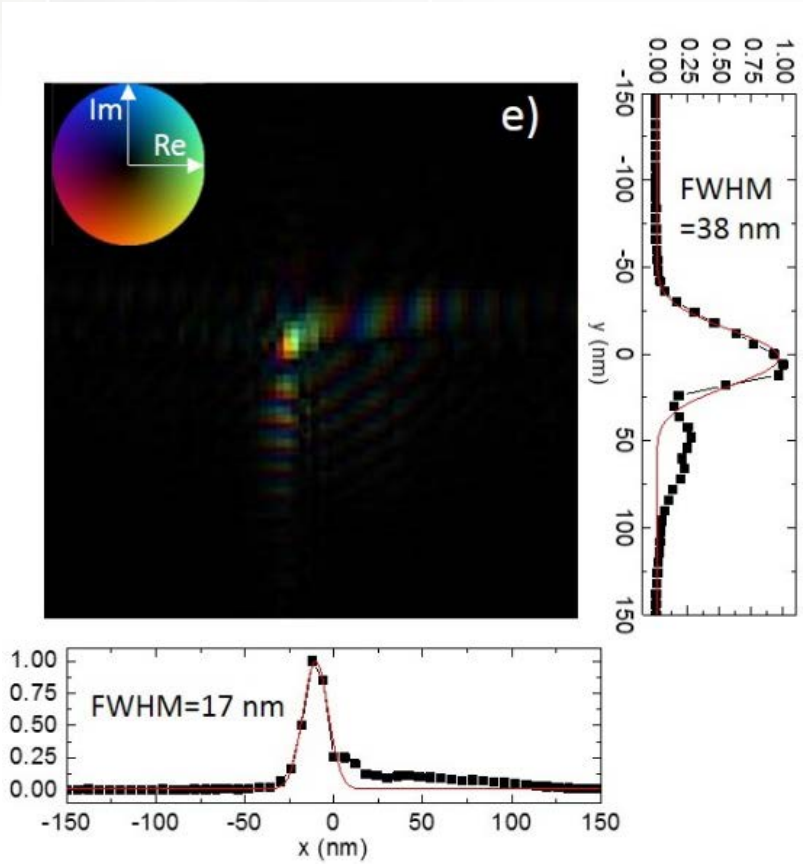
'Development and characterization of monolithic multilayer Laue lens nanofocusing optics' E. Nazaretski, W. Xu, N. Bouet, J. Zhou, H. Yan, X. Huang and Y S. Chu, *Applied Physics Letters* **108**, 261102 (2016)

Development of monolithic 2D Multilayer Laue Lens nanofocusing optics



An SEM image of the cross-bonded MLLs





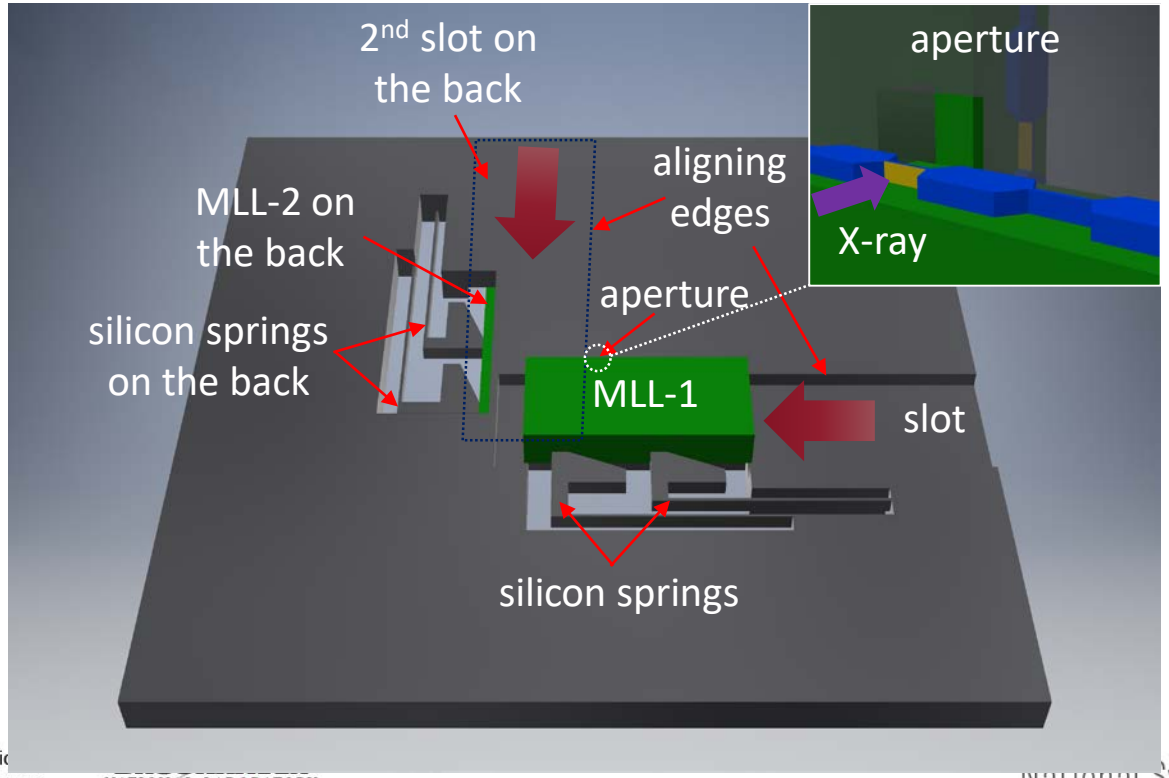
Point focus at 9.317 keV photon energy

A scanning transmission x-ray microscopy (STXM) image of a star test pattern, 50 × 50 pixels with 20 nm step size. c) Reconstructed complex object function from ptychography; its brightness and color represents the amplitude and phase, respectively (see the inset image in e). e) The reconstructed complex probe in the sample plane; a point focus is evident, though there are side lobes due to imperfections of MLLs and the bonding process. The projected intensity profiles indicate a FWHM size of 17 nm in horizontal and 38 nm in vertical direction.

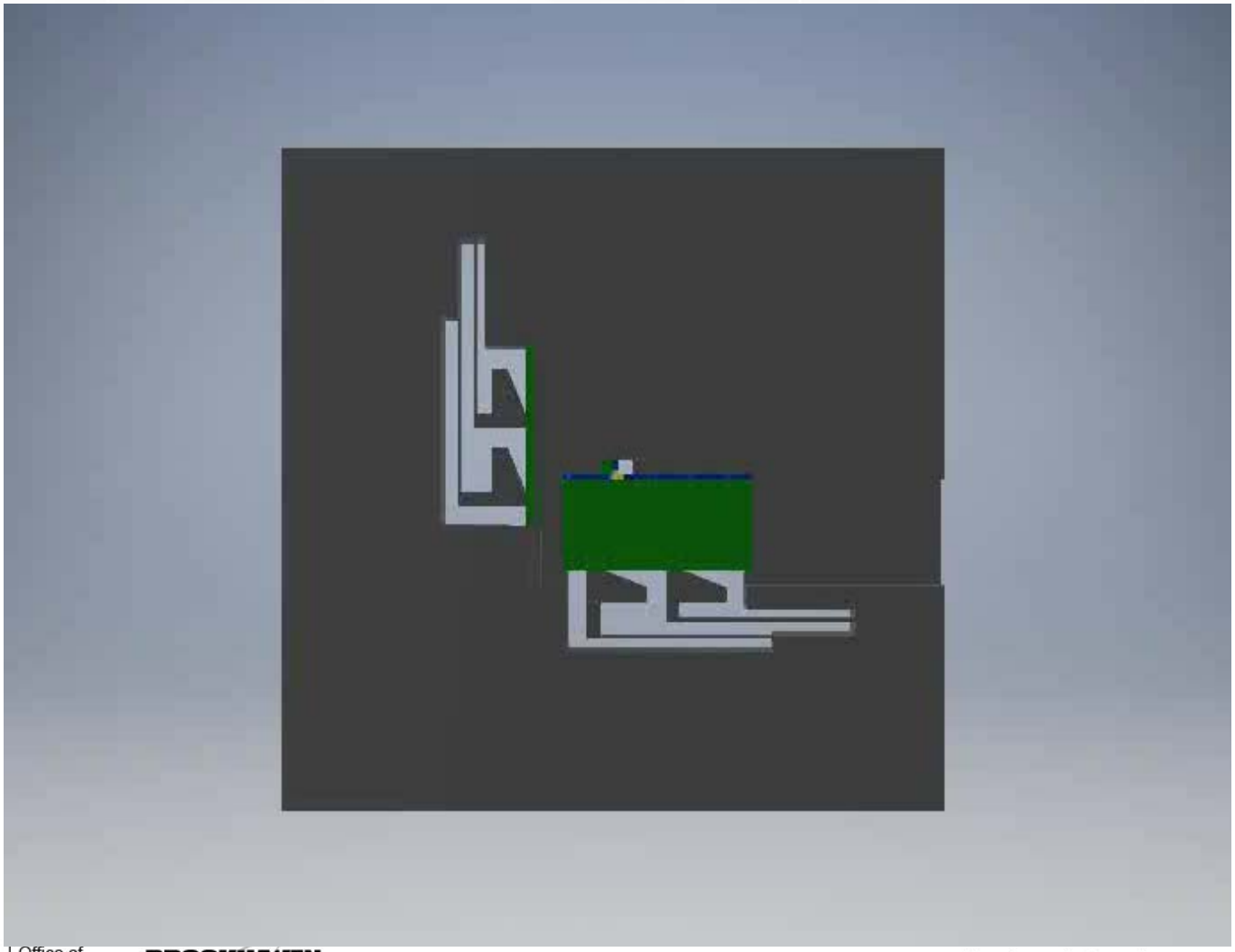
'Hard x-ray scanning imaging achieved with bonded multilayer Laue lenses' X. Huang, W. Xu, E. Nazaretski,, N. Bouet, J. Zhou, Y S. Chu and H. Yan, *Optics Express* **25**, 8698 (2017)

MEMS-based Monolithic 2D Multilayer Laue Lens Nanofocusing Optics

- Pre-package MLLs using a microfabricated silicon template, which includes a slot, aligning edge, and Si springs on each side, and an aperture at the center.
- Two MLLs are slid into the slot on each side and aligned orthogonally to each other, with respect to aligning edges and secured by Si springs.
- The separation distance between two MLLs is controlled by the gap between two slots.
- The effective areas of MLLs overlap at the aperture.

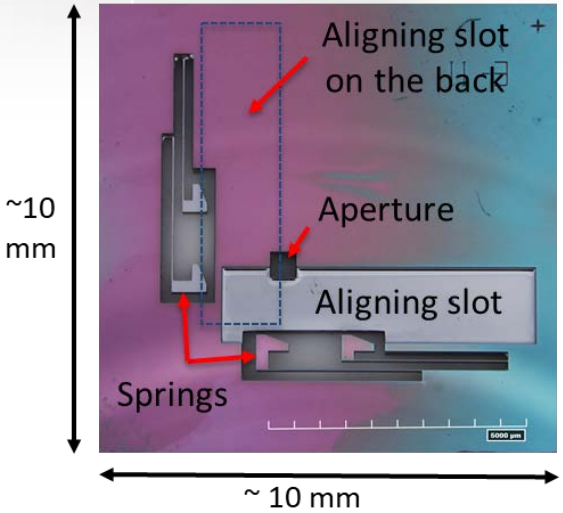


MEMS-based Monolithic 2D Multilayer Laue Lens Nanofocusing Optics

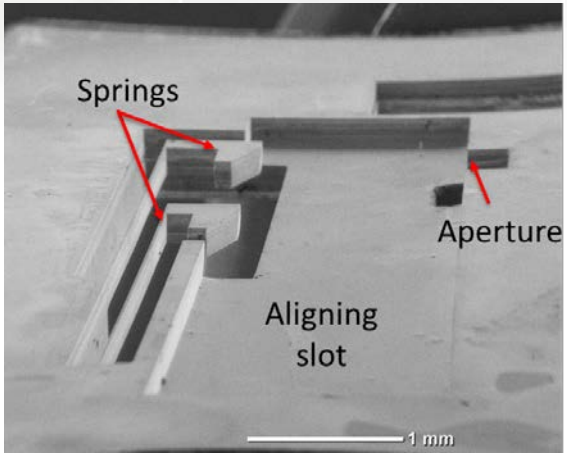


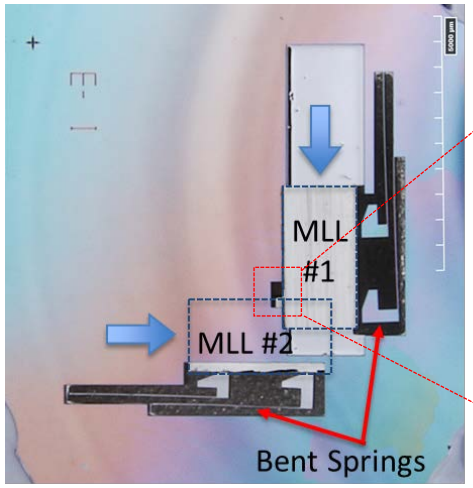
Microfabricated Templates

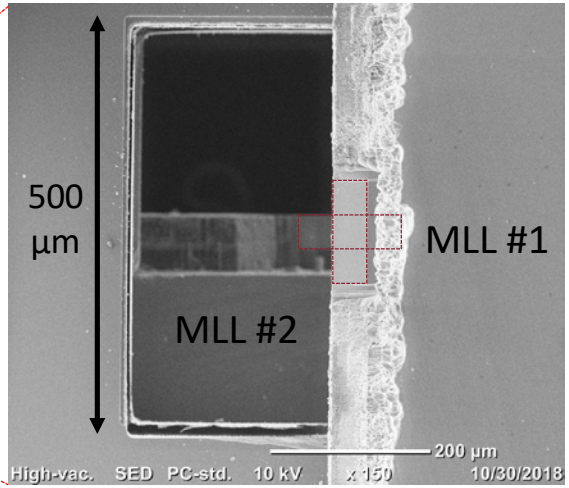
- Optical image



- SEM image

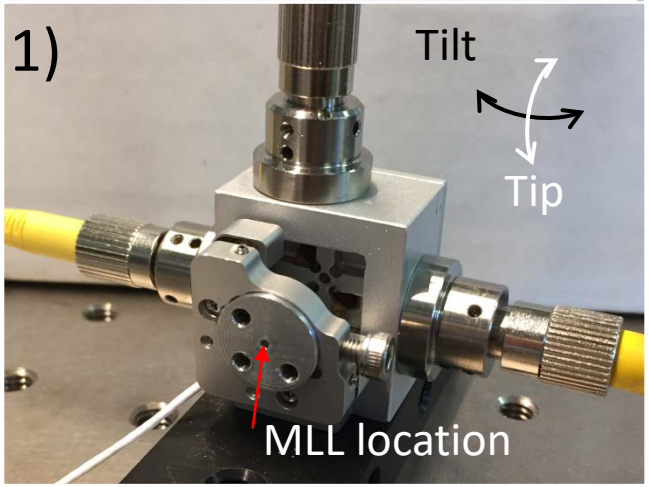


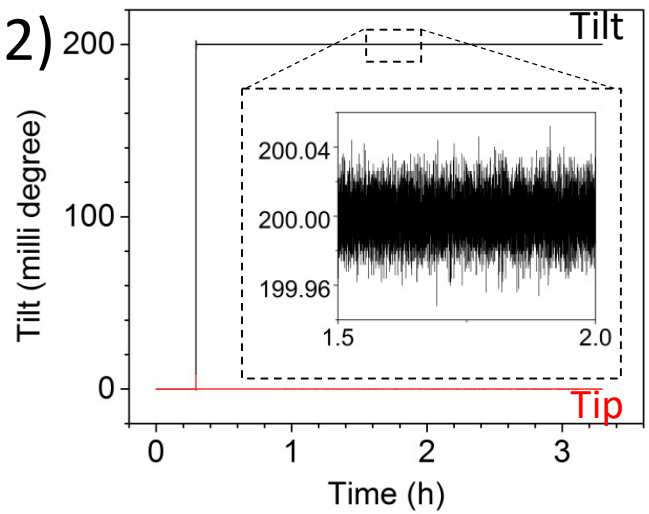


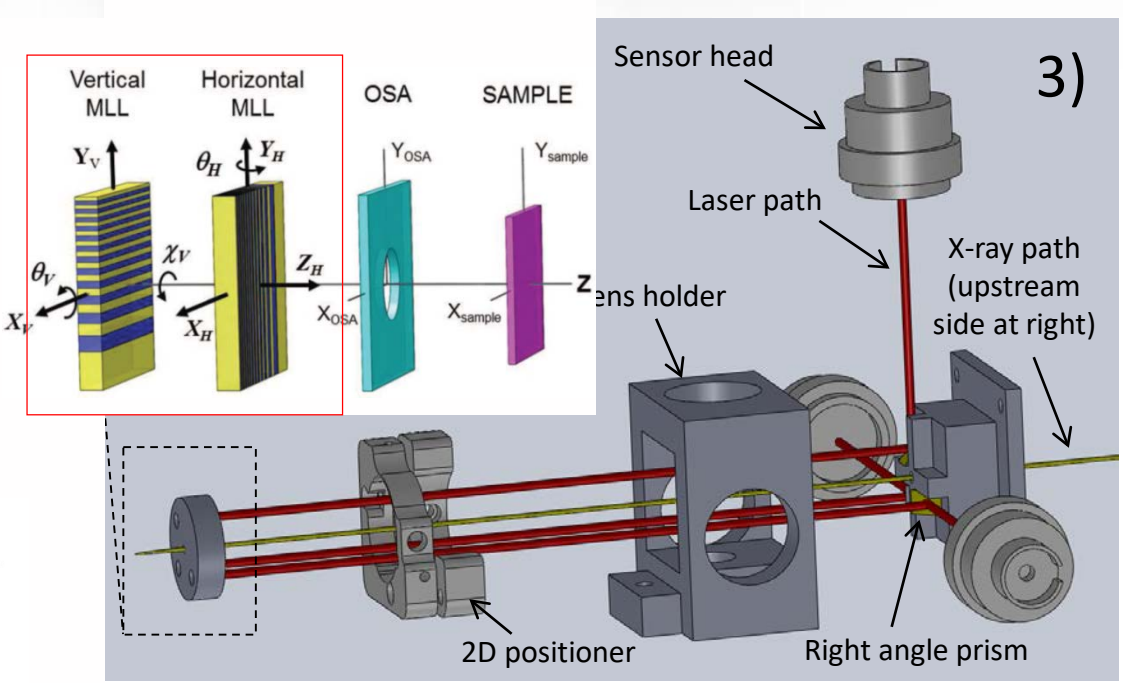


- Two MLLs are packed into the template.
- Effective areas of the lenses overlap through the aperture.

2D tip/tilt stage for the next generation MLL microscope



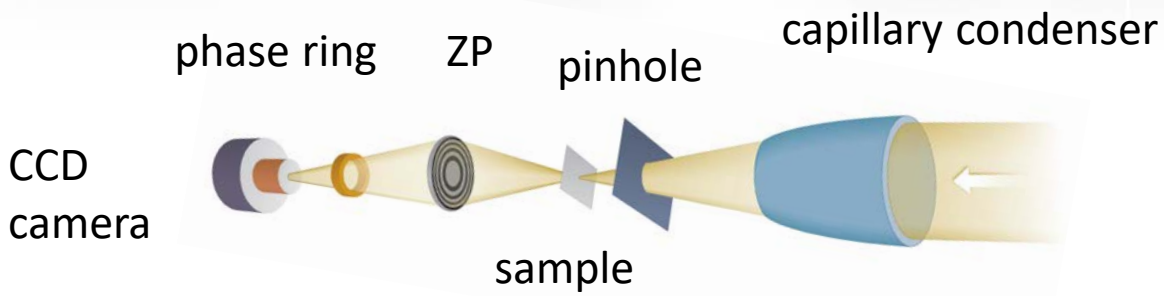


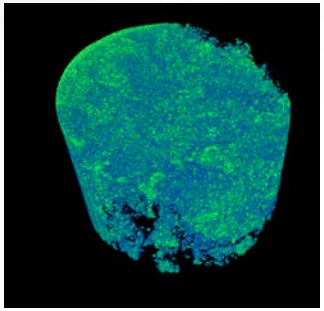


- Better than 100 μ° resolution and over 4° working range in both directions
- Linear dimension ~30 mm
- Negligible thermal dissipation

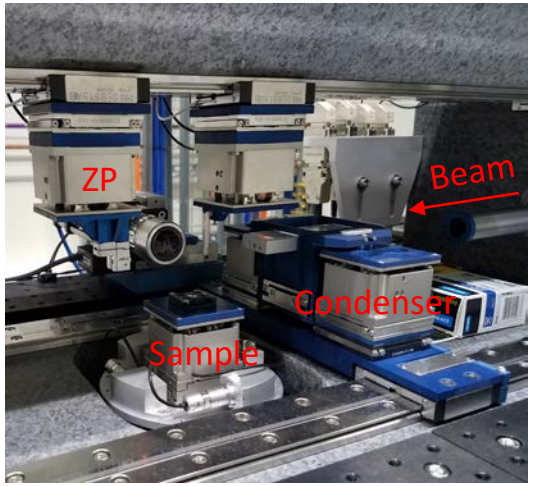
'High resolution tip-tilt positioning system for a next generation MLL-based x-ray microscope', W. Xu *et al., Meas. Sci. Technol.* **28,** 127001 (2017)

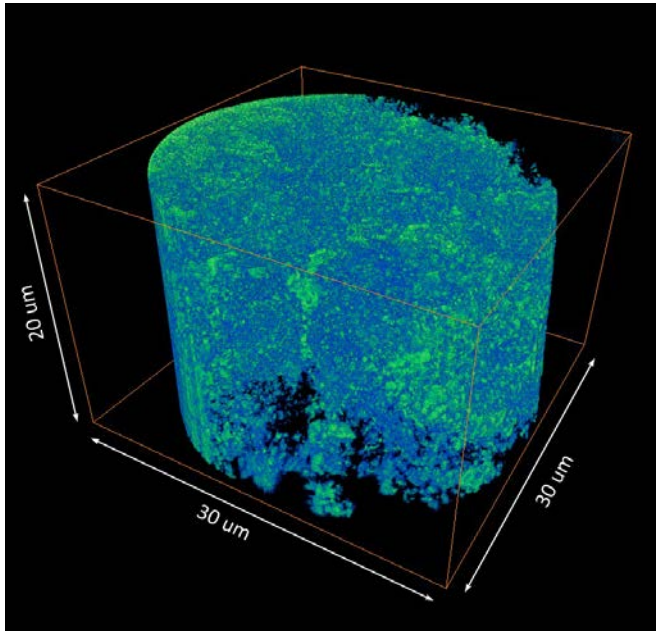
Full-field TXM instrument at the FXI beamline





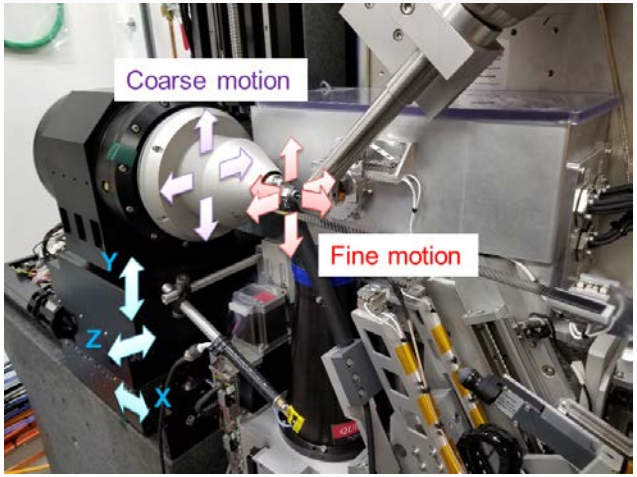
Porous Cu, pixel size 30 nm, acquisition time < 5 min

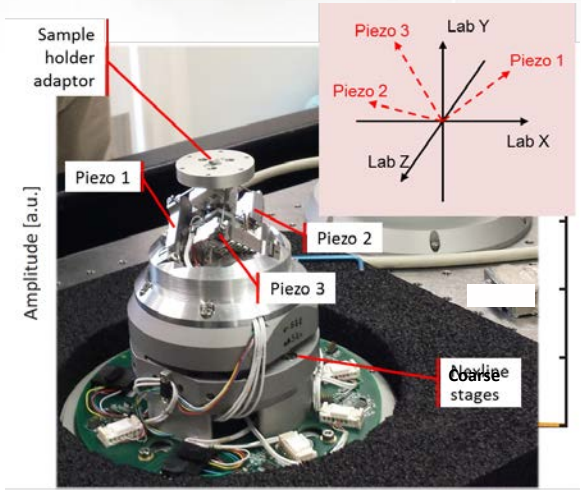


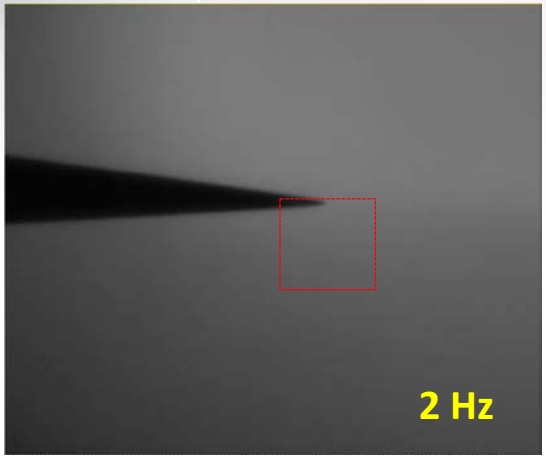


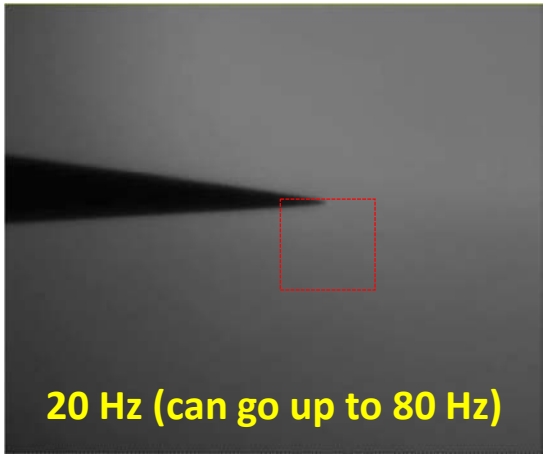
TXM development team:, S. Coburn, E. Nazaretski, W. Xu, Mingyuan Ge, Wah-Keat Lee, *Appl. Phys. Lett.*, 2018

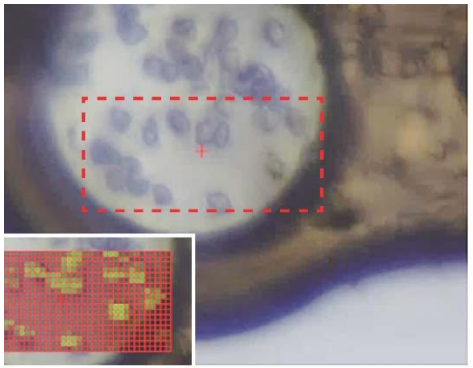
Ultra-high speed serial crystallography at full flux – FMX beamline



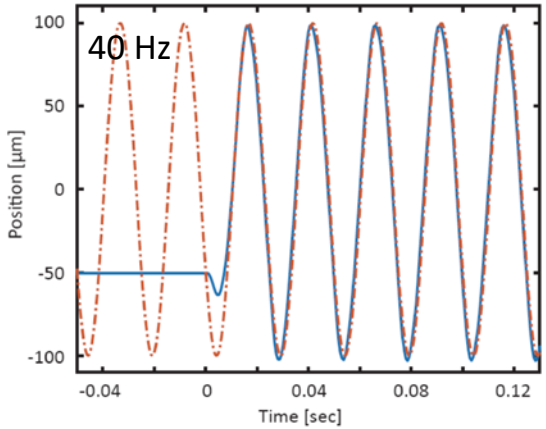








5 – 10 µm sized proteinase K crystals



'Ultra-high speed serial crystallography at full flux; FMX beamline, Y. Gao et al., J. Synch. Rad. (2018)

FXI goniometer/scanner development team: Y. Gao W. Xu, E. Nazaretski, M. Fuchs

Development team and collaborations

MLL Fabrication

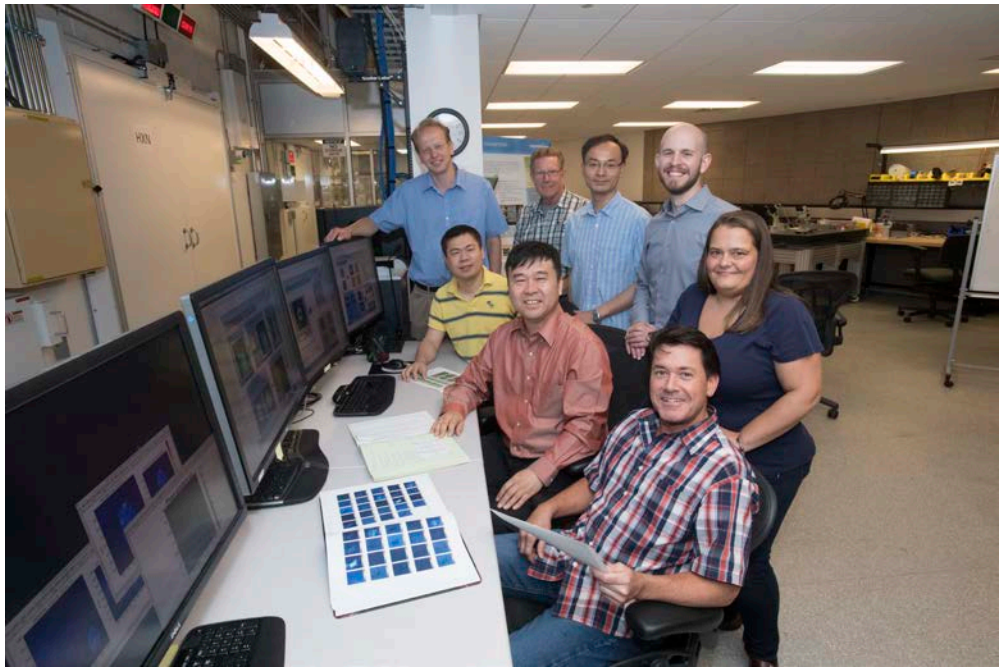
Nathalie Bouet
Juan Zhou, Matt Vescovi

HXN Beamline

Yong Chu
Hanfei Yan
Xiaojing Huang
Peter Ilinski
Mingyuan Ge
James Biancarosa
Michael Maklary
Li Li (data analysis)
and many more…

FXI Beamline

Wah-Keat Lee
Mingyuan Ge
Scott Coburn



HXN Endstation

Evgeny Nazaretski
Weihe Xu
Dennis Kuhne
Wei Xu
Ken Lauer
Andrew Desantis
Brian Mullany
and many more…

FMX Beamline

Martin Fuchs
Yuan Gao
Stewart Myers

Other BNL collaborations

Ray Conley, (APS, MLL optics), Yeukuang Hwu (Taiwan, ZP)
Deming Shu (APS, endstation development), Oleg Chubar (wavefront simulations)
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