R&D Plans for Nanofocusing Optics

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X-ray Science Division
On behalf of the Optics Group

July 16, 2012

Outlines

- Kirkpatrick-Baez Mirrors (focus to 50 nm)
- Zone Plates (focus to 20 nm at 25 keV)
- Multilayer Laue Lens (Focus to 10 nm at 25 keV)
- Summary

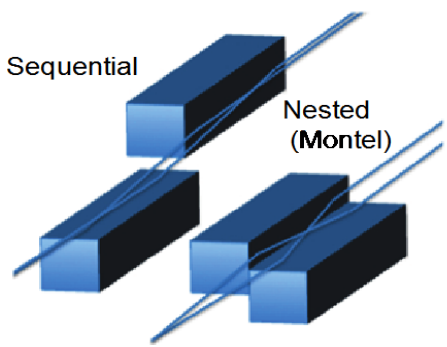
Focusing Optics Requirements and Options

Resolution of a delivered x-ray focusing optics

- Thresholds (Performance Deliverable): 20 nm at 25 keV
- Objective : 10 nm at 25 keV
- ISN and S3DD beamlines: 50 nm in both mono- and polychromatic focusing
- ISN: 20 nm focus

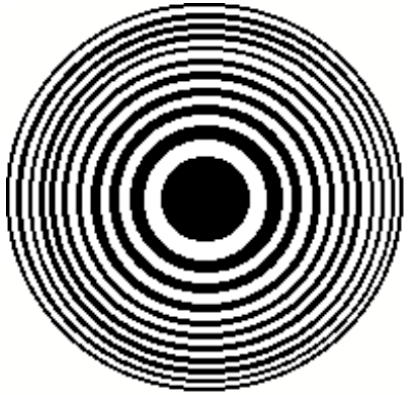
Reflective Optics

- **Kirkpatrick-Baez Mirrors**
  - **K-B mirrors: 50 nm**
  - **ML K-B optics: <25 nm**

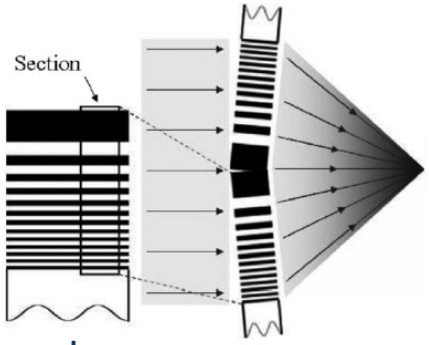


Diffractive Optics

- **Fresnel Zone Plates (FZPs)**
  - **Focus to 20 nm**



- **Multilayer Laue Lenses (MLLs)**
  - **Focus to 10 nm**

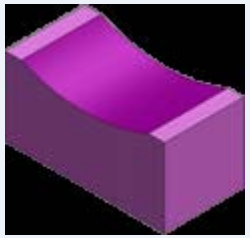


APS has expertise in all of these to carry out related R&D work

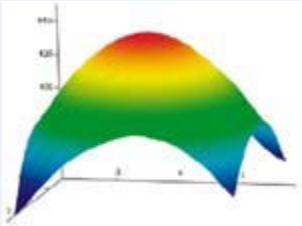
Teams & collaborators

- **Zone Plates**
  - Joan Vila Comamala (OPT)
  - Collaboration with APS/IMG (Steve Wang) and MiC (Jörg Maser) groups
  - Collaboration with BNL/CXRO
- **MLL**
  - Bing Shi (OPT)
  - Chian Liu (OPT)
  - Al Macrander (OPT)
  - Jörg Maser (MIC)
  - Collaboration with NSLS-II, CNM and APS/MIC Group
- **KB mirrors**
  - Chian Liu (OPT)
  - Bing Shi (OPT)
  - Jun Qian (OPT/metrology)
  - Lahsen Assoufid (OPT)
  - Collaboration with ESRF
- **Beamline Testing of optics (1-BM and 32-ID or 8-ID?)**
  - Al Macrander (OPT)
  - Joan Vila Comamala (OPT)
  - Lahsen Assoufid (OPT)
  - Jörg Maser (OPT)

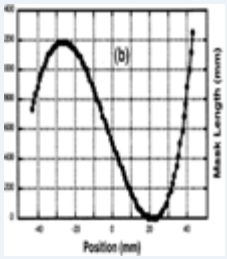
Fabrication of KB mirrors by profile-coating*



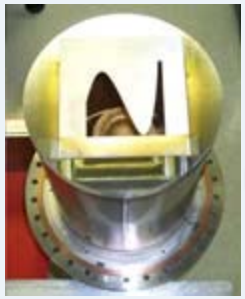
Substrate Metrology



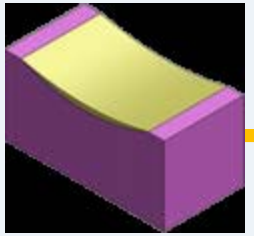
Profile of sputtering rate



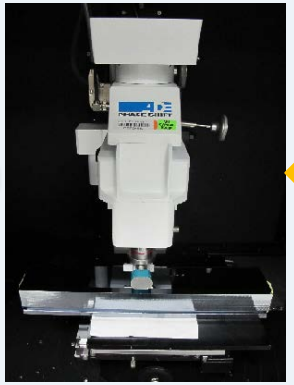
Coating profile



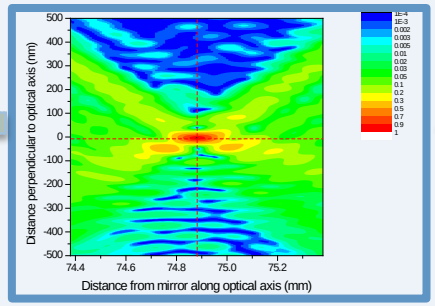
Make a customized mask for sputtering



Mirror coating



Metrology

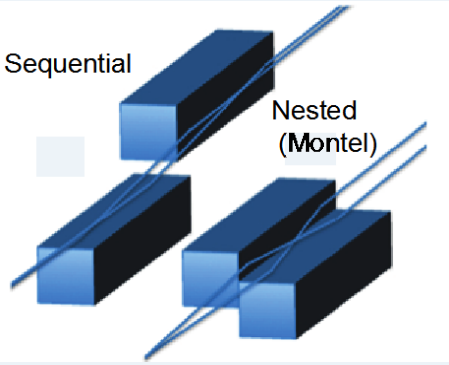


Simulations

Done?

No

Yes

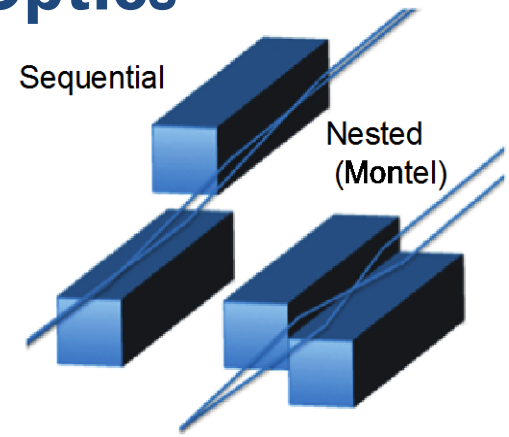


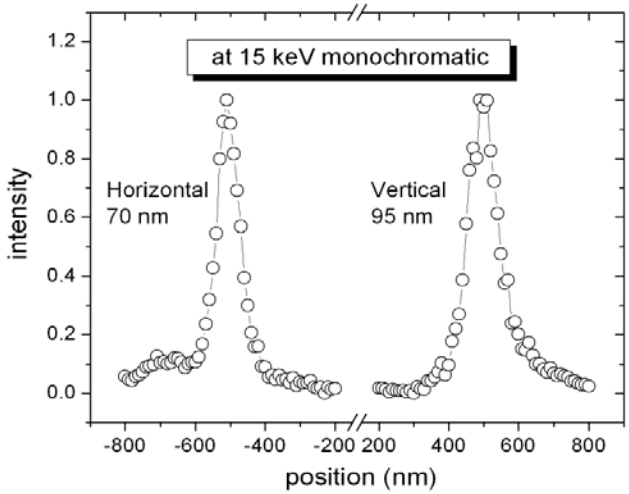
KB mirror arrangements

*C. Liu, L. Assoufid, R. Conley, A. T. Macrander, G. E. Ice, and J. Z. Tischler, Opt. Eng. 42, (2003) 3622

Development of Reflective Focusing Optics

- **Goals:**
  - 100 nm x 100 nm for routine operation
  - <50 nm pink beam focusing for ISN and S3DD beamlines
  - Develop complete K-B mirror assemblies (i.e., mirrors+mounting); this will help standardize APS optics and thus reduce cost of maintenance and time
- **Technical approach**
  - Fabrication by thin film profile-coating
  - Conventional sequential K-B mirror arrangement
  - Montel nested K-B mirror arrangement
- **APS achievement to date (single layer K-Bs)**
  - Sequential arrangement:
    - <100 nm x 100 nm [W. Liu et al. Rev. Sci. Instrum. (2005). 76]
  - Montel nested arrangement:
    - 151 nm x 145 nm [1]
    - Demonstrated 70 nm x 95 nm (unpublished) [2]



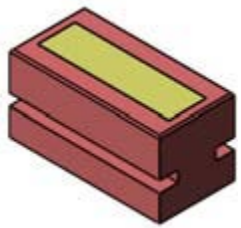


[1] W. Liu, G. E. Ice, L. Assoufid, C. Liu, B. Shi, R. Khachatryan, J. Qian, P. Zschack, J. Z. Tischler and J.-Y. Choi, J. Synchrotron Rad. (2011). 18

[2] W. Liu, G. E. Ice, L. Assoufid, C. Liu, B. Shi, R. Khachatryan, J. Qian, P. Zschack, J. Z. Tischler (unpublished)

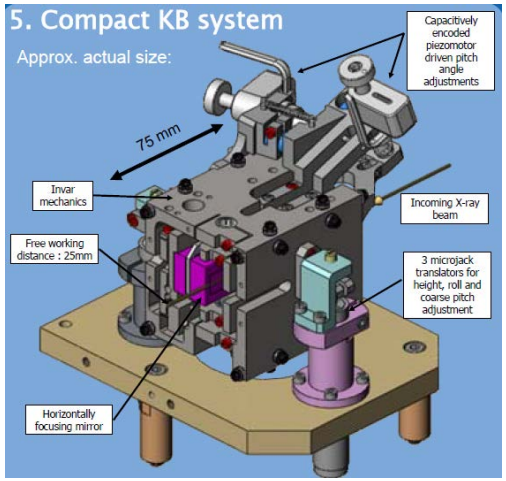
Collaboration with ESRF: APS Profile-coated Pair of KB Mirrors for ID-13-Preliminary results (6/26/12)*

- **Mirrors:** Si spherical substrates profile-coated with Pt at the APS
- **Goal:** focus to 100 nm (V) x 100 nm (H)
- **First tests achieved by ESRF team @ ID6:** 102 nm (V) x 108 nm (H) @ 12.5 keV
- **Current Limitation:** adjustment of astigmatism



VFM 40 mm x 20 mm

HFM 25 mm x 20 mm



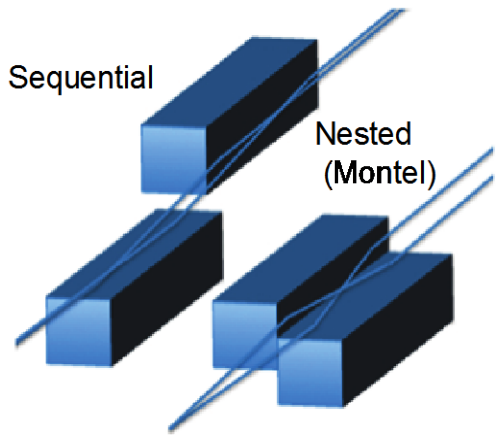
Optical characteristics of mirrors determined at ESRF (relative to best fit ellipse)	Vertical Focusing (VF)	Horizontal Focusing (HF)
Entrance arm, p [m]	98	98
Exit arm, q [mm]	81.887	38.168
Incidence angle, [mrad]	3.868	3.675
Accepted aperture [μm]	135	78
Measured slope error [μrad rms]	0.27	0.25
Measured shape error [nm rms]	0.23	0.19
Microroughness [Å rms] (2.5/5/50x objective)	1.9/1.7/1.8	2.1/1.3/1.0

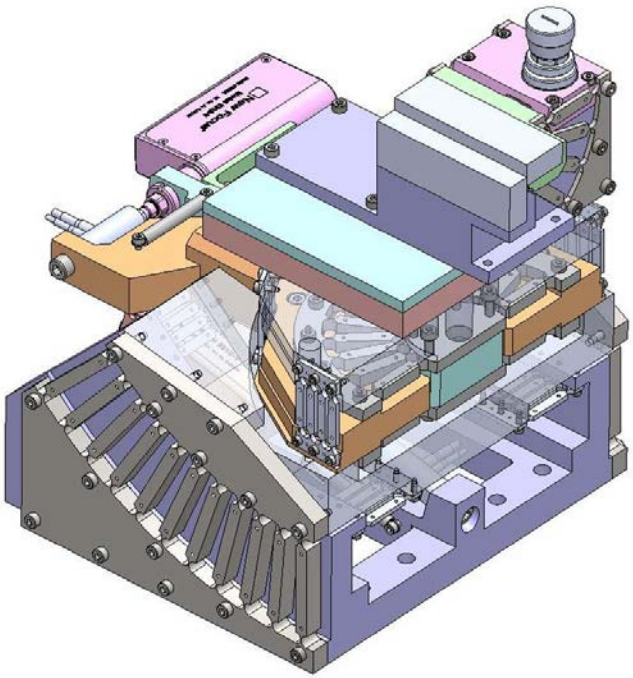
*R. Barrett, P. Cloetens, B. Shi, A. Vivo, J. Qian,R. Baker, L. Assoufid , Poster SRI2012

Further tests will be carried out in Fall 2012.

Need for standardized compact mounting system for K-B mirrors

- **Option #1**: Develop a modular system to accommodate either sequential or Montel arrangement.



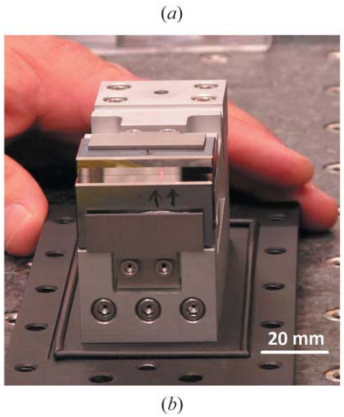


- **Option #2**: Develop a mount for Montel arrangement and acquire/exchange with ESRF their design for sequential K-B mirrors

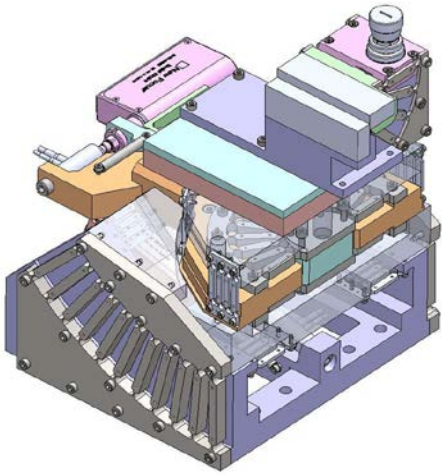
- ESRF design for sequential K-B mirrors

Courtesy: R. Barrett

- Improve ORNL-APS Design for Montel nested K-B mirrors



W. Liu et al. J. Synchrotron Rad. (2011). 18

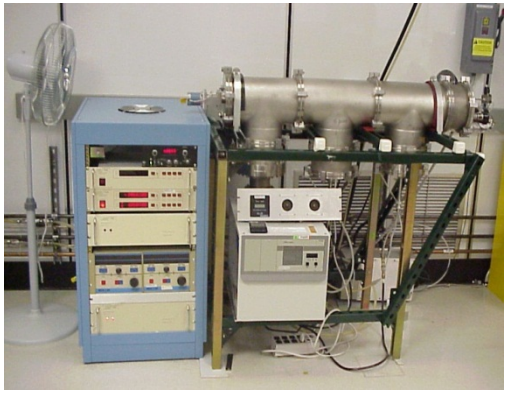


Courtesy: D. Shu

Reflective Focusing Optics: infrastructure improvement

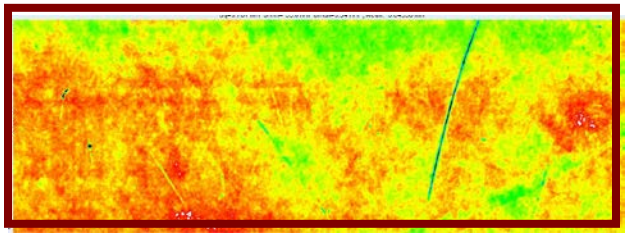
Achieving K-B mirrors with 50-nm focus requires improved deposition chamber and metrology

- **Deposition tool improvement :**
  - Existing system capacity is limited by the poor quality of its translation stage and the size of the coating chamber.
  - Modest approach: Replace the linear stage of the existing system
  - Comprehensive approach: acquire a new system that is suitable for both single and multilayers K-B mirrors as well as for multilayer Laue Lenses fabrication



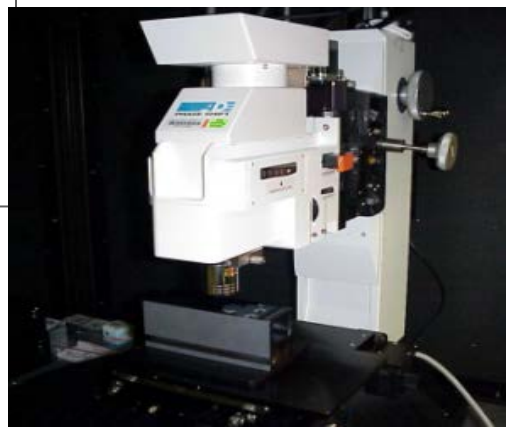
The APS small deposition system dedicated to KB mirrors fabrication

- **Metrology improvement :**
  - 2-D surface profile measurements are essential. This is typically done by subapertures stitching interferometry (See figure below)
  - Existing tool accuracy is limited by system's aberrations to 3 nm P-V
  - Require accuracy <1 nm P-V



Reconstructed 2-D partial surface topography of an elliptical mirror clearly reveals fabrication defects.

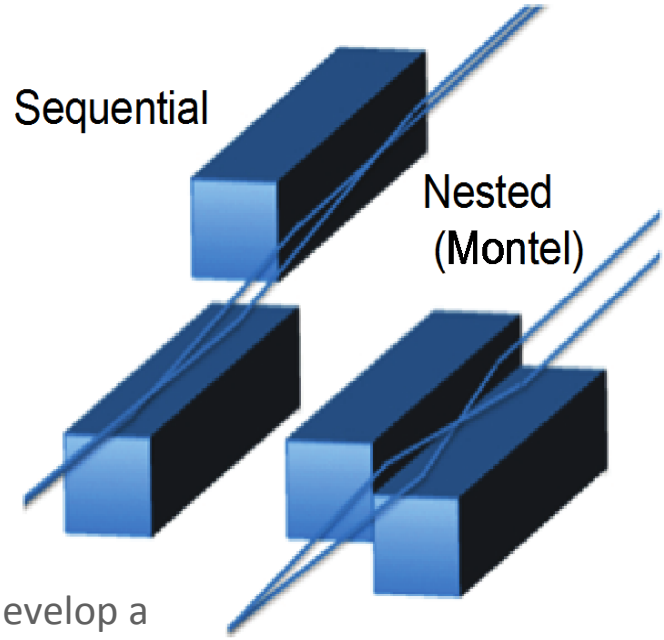
L. Assoufid et al. SPIE Proc. (2007) 6704.



APS Microstitching system

Reflective Focusing Optics: Efforts and resources

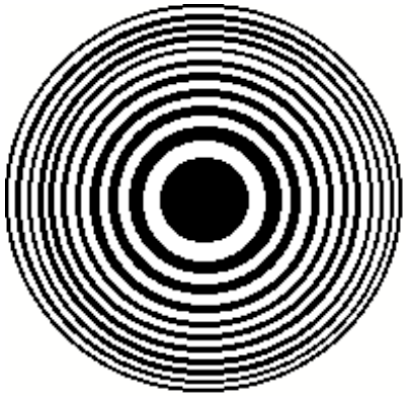
- **Efforts: Existing staff**
  - 0.5 FTE/year deposition staff for 3 years (Bing Shi, Chian Liu)
  - 0.1 FTE/year metrology for 3 years (J. Qian, L. Assoufid)
  - 0.1 FTE-year beamline scientist for testing (J. vila-Comamala, A. Macrander, L. Assoufid)



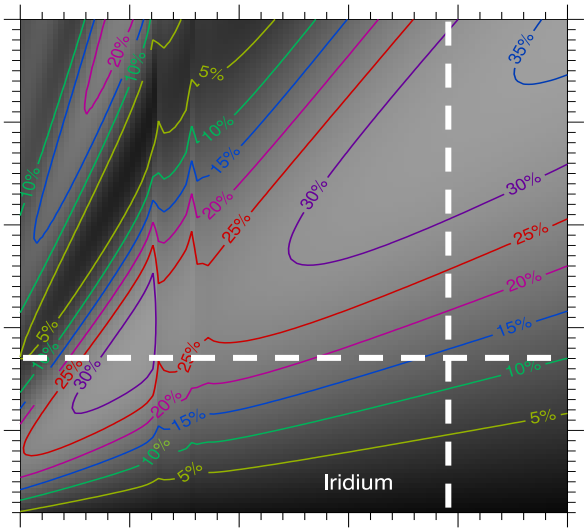
- **Additional effort**
  - 0.5 FTE/year of engineering over 2 years for 3 years to develop a standardized K-B mirror mounting system
  - Control software: APS Beamline Control & Data Acquisition staff

Zone plate optics provide a viable path to achieve 20 nm at 25 keV

- **FZPs are Essential for APS micro/nano-probes**
  - Nanotomography program at the 32-ID transmission x-ray microscope
  - Compact, simple to mount and align
- **Challenges**
  - At present: 20-50 nm resolution in the 0.2-15 keV range.
  - Commerically available ZP have very low efficiency (a few %) at E>10 keV and 20 nm)
  - Short lifetime
- **Technical approach**
  - Top down nanofabrication (e-beam, etch, plating or ALD)
  - Stacking



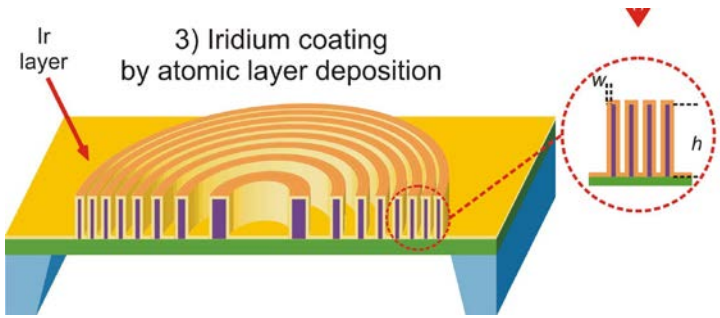
Fresnel Zone Plate



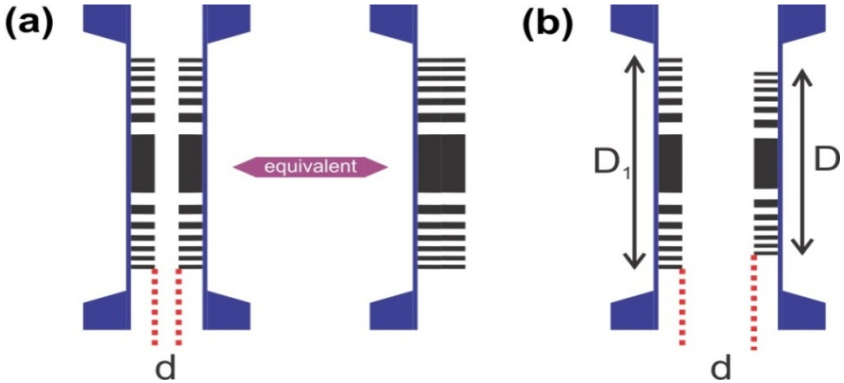
Efficiency of an Iridium zone plate as a function of energy and thickness.

Zone plate optics at the APS

- LDRD-supported research program to develop hard x-ray zone plates for 10-100 keV using: e-beam Lithography on Anodic Aluminum Oxide and Atomic Layer Deposition (AAO ALD) and zone doubling (S. Wang, J. Vila Comamala), via Center for Nanoscale Materials general user proposal.
- Nanofabrication technique transferred by J. Vila Comamala to CNM from the Paul Scherrer Institut (Switzerland)
- Joan Vila –Comamala has been hired by the Optics Group. He will continue developing the technology in collaboration with S. Wang (APS/IMG) and other groups:
  - Anodic Aluminum Oxide @ Material Science Division (H.-H. Wang) and Atomic layer deposition @ Energy System Division (Jeff W. Elam & D. Comstock)
  - National Consortium for Diffractive Optics: hard and soft x-ray zone plate "production" at Center for X-ray Optics in Berkeley, hard x-ray zone plate R&D at Argonne.

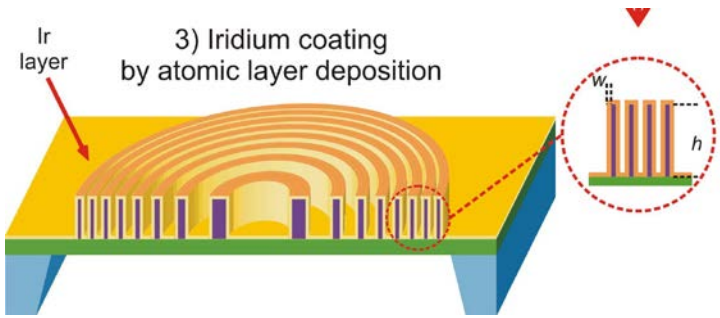


Zone doubling: 20 nm wide, 170 nm tall Ir: Jefimovs *et al.*, *Phys. Rev. Lett.* **99**, 264801 (2007). Co-author Joan Vila Comamala now at APS.



Identical FZPs can be stacked to obtain an equivalent thicker structure with a substantially increased the diffraction efficiency

- **Efforts: existing staff**
  - 1.5 FTE Physicist (J. Vila-Comamala) for over 2 years
- **Additional Efforts**
  - 0.5 FTE-year Scientific Associate for 2 years for testing
  - 0.5 FTE-year of engineering effort to develop and test a MEMS-based stacked zone plate alignment system



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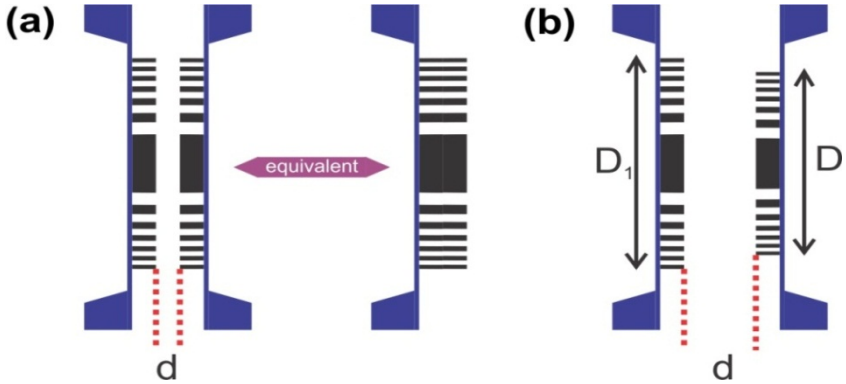
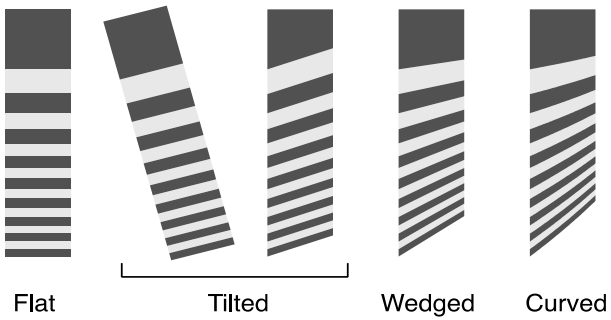


Identical FZPs can be stacked to obtain an equivalent thicker structure with a substantially increased the diffraction efficiency

Multilayer Laue Lens (MLL) provide a path to 10 nm focus

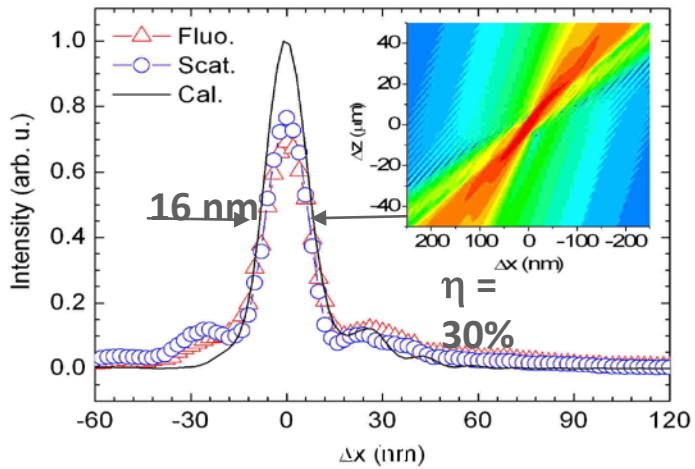
- **Technical approach**
  - Crossed multilayer-based linear zone plate structure

- **Achievements to date**



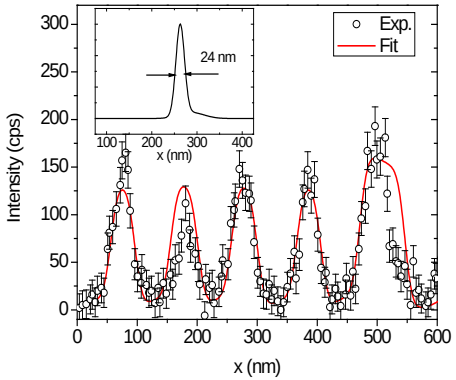
Different types of MLL structures

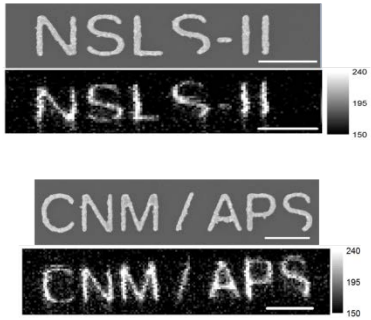
1-D focus: 16 nm (half MLL) **@ 19.5 keV**



Kang et al, APL 92, 221114 (2008)

2-D focus: 25 x 40 nm^2 (2D, crossed MLL) **@ 20 keV**



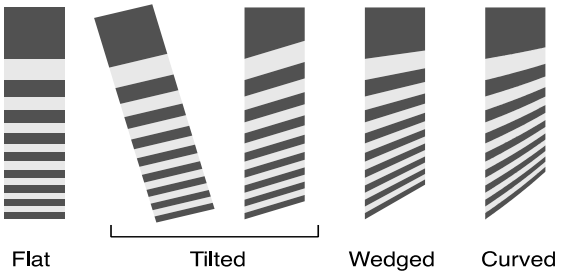


2-D imaging with crossed MLL (ANL/BNL collaboration)

Yan et al, H. Yan et al, Opt. Exp. 2011

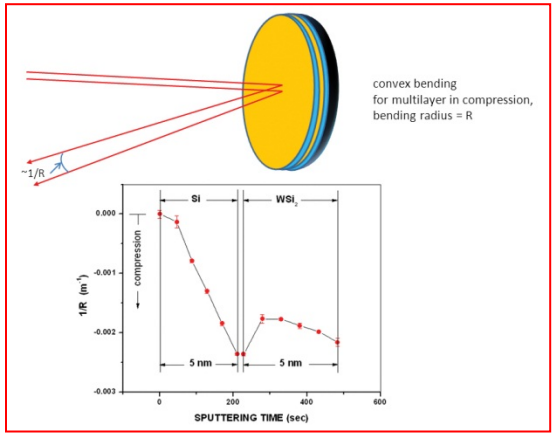
Multilayer Laue Lens (MLL) provide a path to 10 nm focus

- **Challenges**
  - MLL with thickness > 75 um will be required
  - 10,000 of layers will be required
    - Grown-in stress
    - advanced metrology
    - Maximum thickness that can be grown at APS is 20 um



Different types of MLL structures

- **Efforts and resources:**
  - **Options #1: In-house development**
    - 0.5 FTE-year deposition staff for 3 years
    - 1 FTE-year for postdoc for 3 years
    - 0.5 FTE-year of engineering + $100K in costs to develop and test a MEMS-based MLL alignment system
    - $70K Deposition system upgrade for half-diameter MLL (~$700-800K for full MLL; the system will enable us to fabricate graded ML K-B mirrors-a promising approach to sub 10 nm focus.)
  - **Options #2: Collaboration with NSLS-II:**
    - Joint R&D (Ray Conley visited the APS July 11, 2012 to discuss common R&D tasks)
    - Fabrication at NSLS-II (Ray Conley) in exchange for ZPs **(requires a formal agreement)**



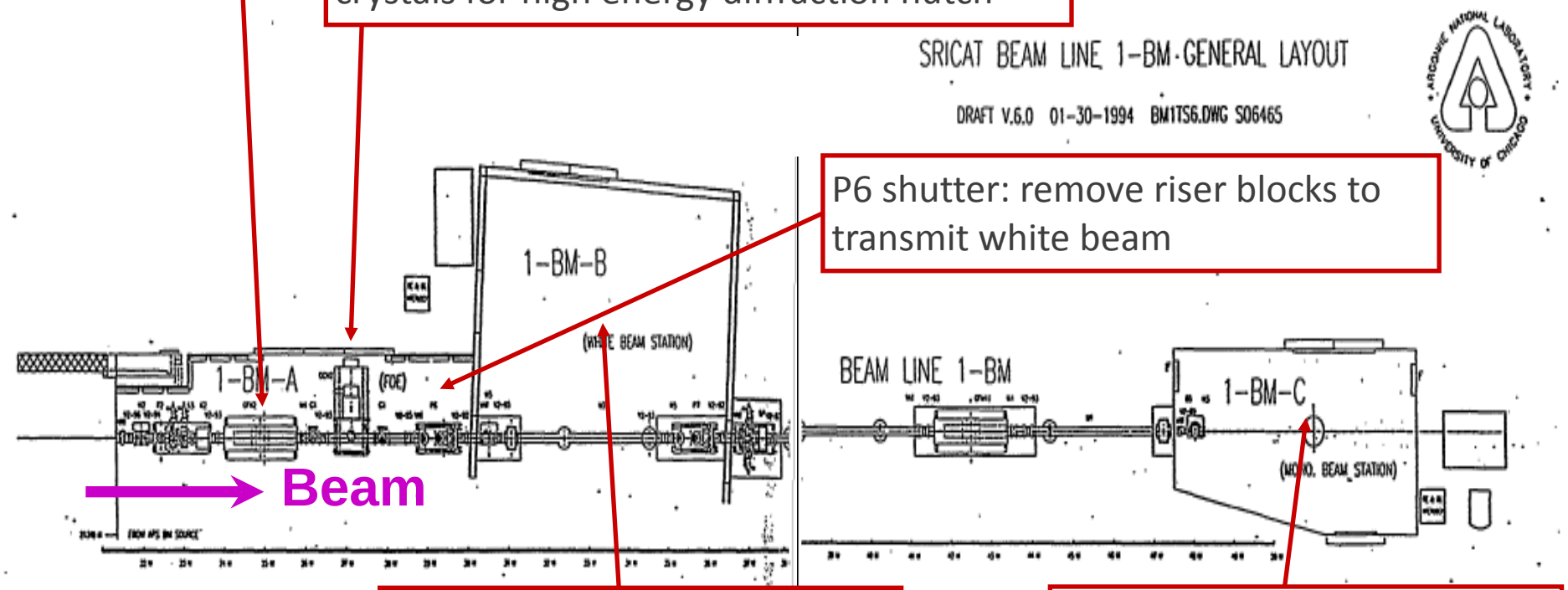
Grown-in stress in MLLs: K. MacArthur, et al., Appl. Phys. Lett. 99 (2011)

Optics and Detectors Test Beamline If at 1-BM

(A. Macrander, L. Assoufid, X. Huang, C. Jacobsen)

Collimating mirror: remove, and replace with 1) fast shutter and 2) flat Bragg double-crystal monochromator for test hutch

PSL monochromator: equip with Laue-Laue crystals for high energy diffraction hutch



P6 shutter: remove riser blocks to transmit white beam

Optics and detector test hutch

High-energy diffraction hutch

Figure from Srajer, Rodricks, Assoufid, and Mills, APS LS-35

Plans and Milestones: Nanofocusing optics

Optics	Operations / 2-3 years	R&D / 2-3 years	Options
Kirkpatrick-Baez (KB) Mirrors	• Develop capability to routinely deploy 100 nm KB mirror, including mounts • Develop 100 nm graded multilayer mirrors with wide bandpass (>10%)	• Develop 50 nm KB optics • Develop modular mounting options supporting both sequential and Montel configurations	• Develop 50 nm Montel optics • Develop <20 nm graded multilayer KB optics • Adaptive optics R&D (deformable pre-mirror) towards 10 nm
Fresnel Zone Plates	• Routinely produce ZPs down to drN=20 nm, 0.4 um thick • Develop double ZP stacking (conventional)	• Develop ZP optics down to drN=16 nm • Develop capability for deep stacking (multiple ZPs) using MEMS (lateral alignment), for energies up to 25 keV	• Add MEMS-based axial adjustment to maximize efficiency and extend useable energy range
Multilayer Laue Lenses	• Develop MEMS-based positioning of 20 nm MLLs	• Leverage MLL development at BNL towards 10 nm • Develop multidimensional MEMS mounting (tip, tilt, lateral positioning)	• Develop in-house MLL fabrication targeted to <10 nm at 25 keV, at least for short focal lengths.

*Not achievable with the current APS beamlines

Thank you!

Extra slide

Comparison of Nanofocusing optics: Fresnel zone plates (FZP), Multilayer Laue Lens (MLL) and Multilayer Kirkpatrick-Baez (KB) mirrors

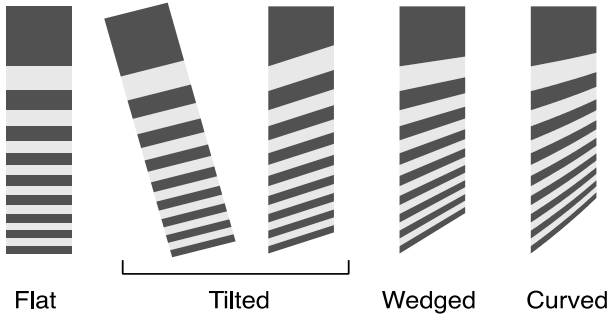
Type of Optic	FZP	MLL	Reflective K-B Optics	
			Single Layer mirrors	Multilayer mirrors
Typical energy (keV)	15	19.5/20	15	20
Present spot size	25 nm*	16 nm (1D)* 25 x 40 nm^2 (2D)*	30 x 50 nm^2** 70 x 95 nm^2*	7 nm (1D) **
Fabrication limit	~10 nm	~2 nm	~25 nm	~4 nm
Chromaticity	$f \sim 1/\lambda$	$f \sim 1/\lambda$	Achromatic	~
Demonstrated efficiency	6% @ 10 keV	30% (1D), 17% (2D)	Unspecified	Unspecified
Acceptance (normal-incidence-equivalent diameter demonstrated)	~250 μm diam	~40 μm	~350 μm**	~650 μm

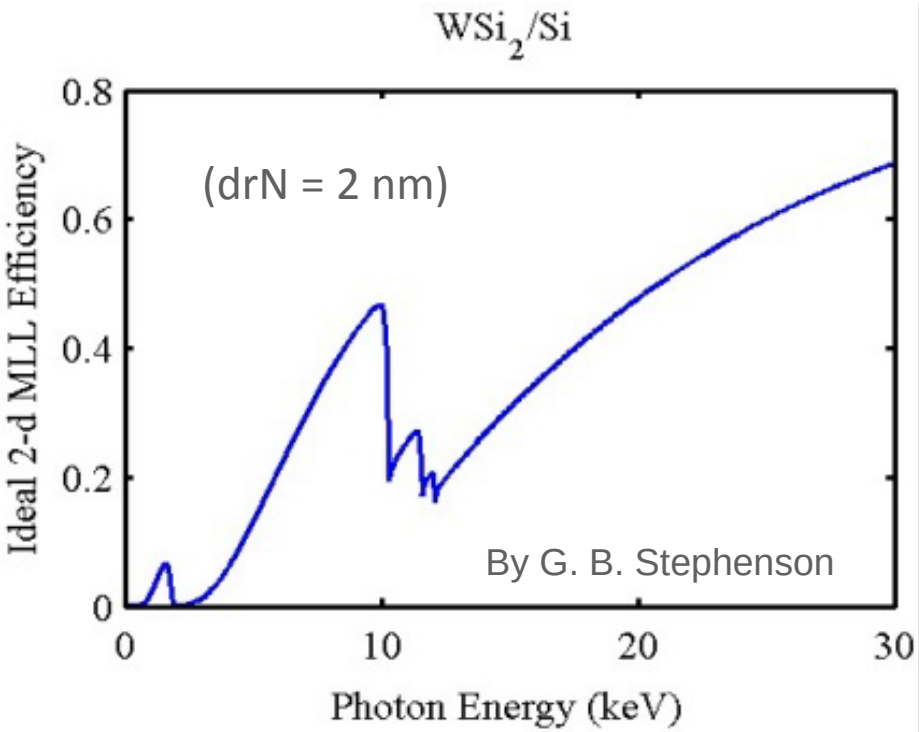
*Achieved at APS (MLL in collaboration with NSLS-II; KB Mirrors are of Montel type)
**Achieved by the Osaka/Spting-8 team (Not achievable with the current APS beamlines)

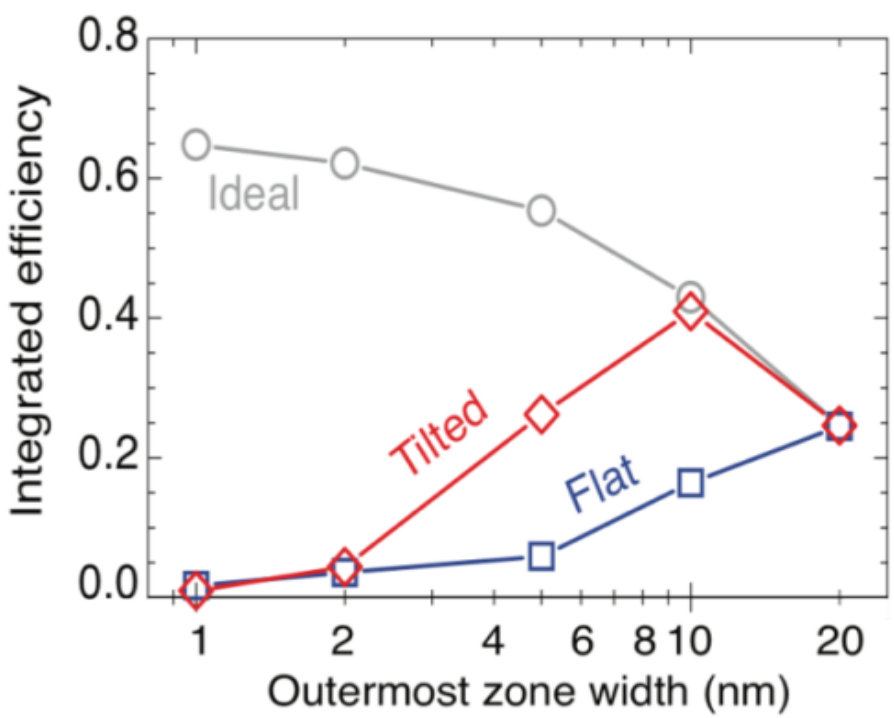
MLLs can provide very high focusing efficiency at high photon energy

Kang et al, APL 92, 221114 (2008)

Yan et al, H. Yan et al, Opt. Exp. 2011

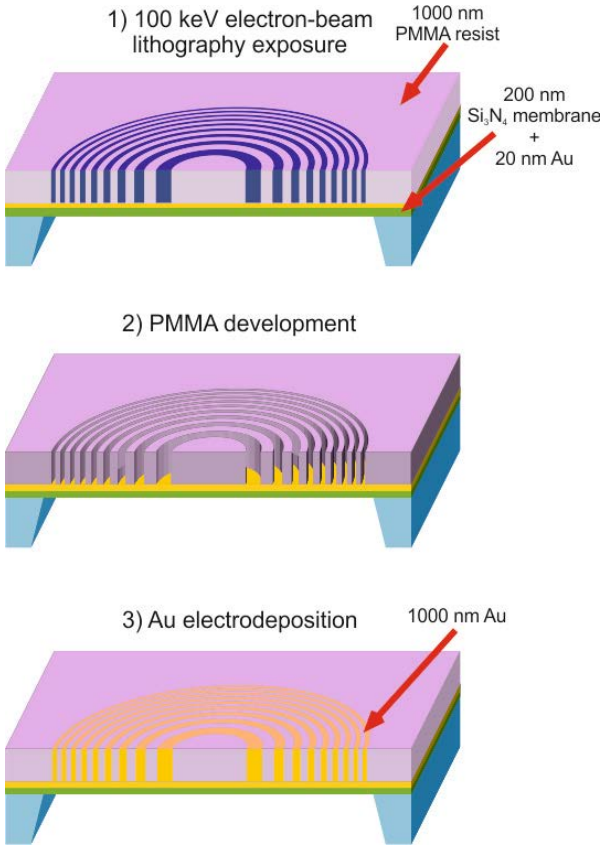




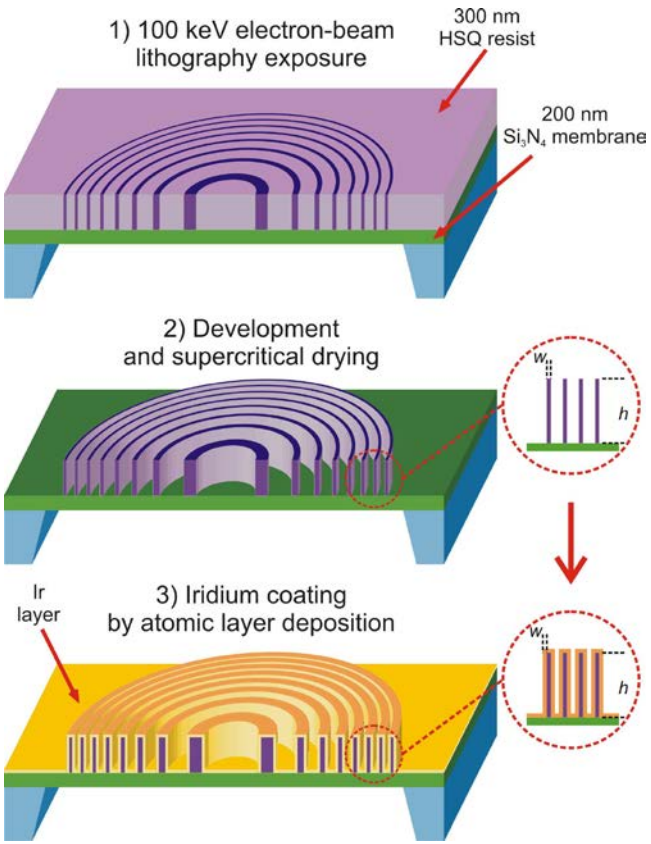


Nanofabrication technique transferred to CNM from the Paul Scherrer Institut (Switzerland)

Au electroplating



Zone-doubling technique



Atomic layer deposition
by J. W. Elam & D. Comstock (ES)