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X-Ray Microfocusing Optics

Barry Lai X-Ray Science Division Advanced Photon Source

Outline

Introduction

General considerations

Reflective optics

Diffractive optics

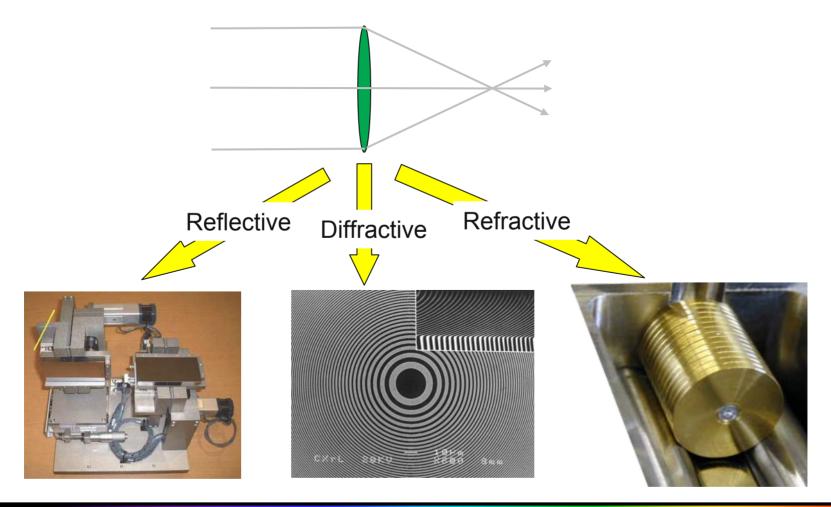
Refractive optics

Future prospects



Introduction

- Optics that focus x-rays to a spot size \leq 10 micron





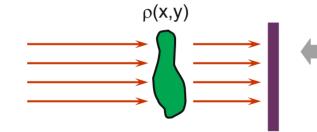
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When to use microfocusing optics:

- For x-ray microscopy
 - Most samples are heterogeneous, even down to nm scale
- Increased flux density
 - Gain ~ 10^6 is possible, hence higher sensitivity
- Enable smaller samples or new sample environment



X-ray Microscopy



Direct imaging (radiography), with magnification in visible light *

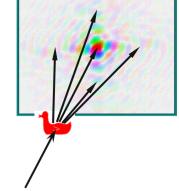
Transmission microscope (TXM)

depends only on total flux, but not brilliance / coherence

Scanning microscope (SXM)

depends only on coherent flux, directly benefits from reduced emittance

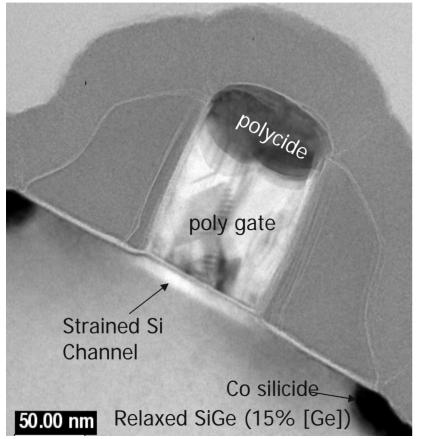
Coherent diffraction microscopy*

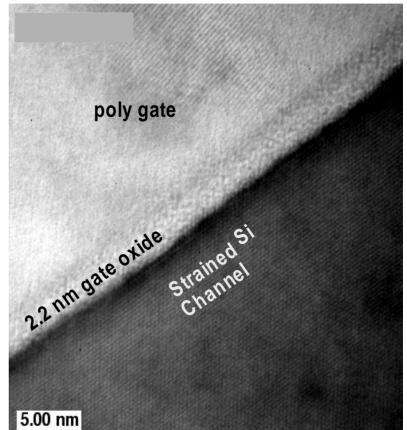




Holography*

Cross-sectional TEM of Strained-Si NMOSFET





- Quality of epitaxial layers maintained during CMOS process steps
- Gate oxide with smooth interface formed by thermal oxidation

=> today's manufacturer's are already able to produce nm scale structures. To probe such small structures meaningfully nead x-ray beam of the same order of magnitude. Slide courtesy Cev Noyan, with modification



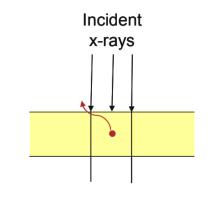
Ken Rim

-IBM

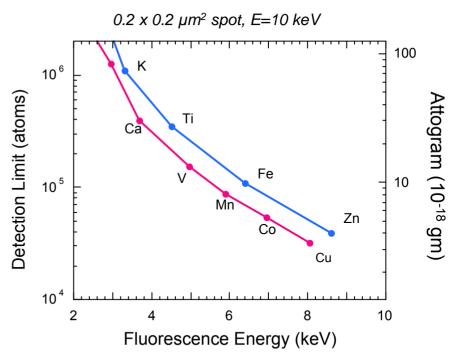
Microfocusing increases flux density

Focusing increases the signal/background ratio

- For current probes with submicron spot, attogram (10⁻¹⁸ gm) of materials can be detected in fluorescence mode
- With a 5-nm probe, sensitivity of zeptogram (10⁻²¹ gm) or a few atoms is possible



Detection Limit with 1 sec. Dwell Time





Microbeam for protein crystallography



- Very small crystals: reduce scatter from non-crystalline material
- Selective exposure of small crystal volumes:
 - very asymmetric shape of crystal (e.g. needle): reduce scatter from non-crystalline material
 - very small, well ordered domains
- Reducing radiation damage in exposed volume:
 - photo-electrons travel several μm (~6μm for 18 keV initial energy)
 - large fraction of damaging energy is not deposited in the illuminated volume for micrometer size beams
 - energy deposit per distance traveled is not uniform, very high at end of travel
- · Photo-electrons ejected predominantly in direction of electric field vector



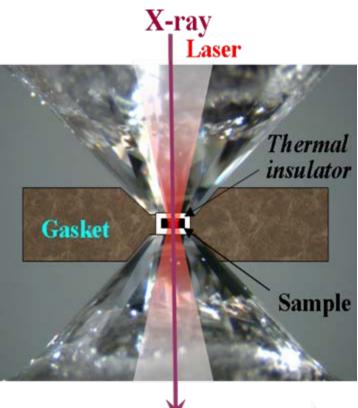
New High-Pressure Frontiers with higher spatial resolution

- Diamond Anvil Cell uses focused microbeams at high energy (>30 keV) to probe highest pressure/temperature region
- High pressure: with better focused beams, can use small anvil tips, and greatly extend the accessible pressure from 350GPa -> TPa
 - New areas for discovery of materials and phenomena
- High temperature: with smaller probes, can limit the heating area to diffraction limit of laser, thus extend max temperature from 6,000K to 12,400K
- Open up new opportunities for studies of materials under core conditions (P,T)
- Improve ability to understand structures of Giant Planets

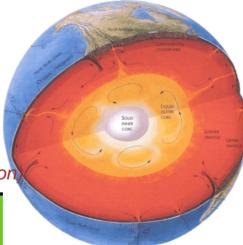
Slide adapted from Steve Sutton (UoC) & David Mao (Carnegie Institute of Washington)



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X-ray detector

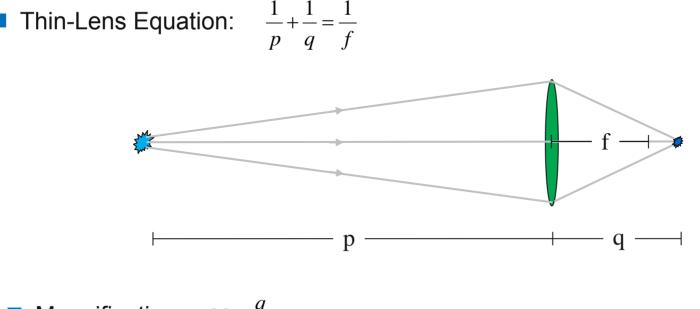


General considerations

- Magnification
- Numerical Aperture
- Resolution
- Depth of Focus
- Chromatic aberration



Geometrical Optics



- Magnification: $M = \frac{q}{p}$
- Microfocusing optics produce a demagnified image of the source (M < 1). Imaging optics produce a magnified image of the sample (M > 1).
- Some optics can work as both, others only for microfocusing (M < 1).



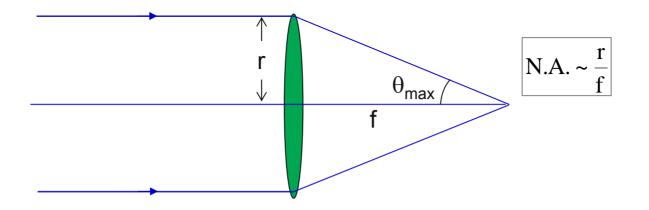
Demagnification

- For synchrotron micro/nanoprobes, $M \sim 10^{-2} 10^{-4}$
- If decreasing focal length becomes difficult, long beamlines (p) will help



Numerical Aperture

■ N.A. = n sin θ_{max} is a measure of the light gathering power $\left(N.A. = \frac{1}{2(f/\#)}\right)$

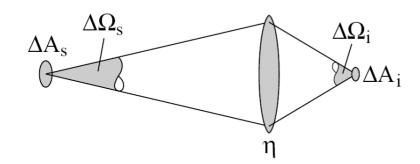


Intimately related to the performance of the optics (focused flux, diffractionlimited resolution, depth of focus, etc.)



Liouville's Theorem

Phase space density is conserved in a perfect optical system



Perfect optical system: $\Delta A_s \cdot \Delta \Omega_s = \Delta A_i \cdot \Delta \Omega_i$; $\eta = 100\%$

Microfocusing optics inevitably will increase the angular spread

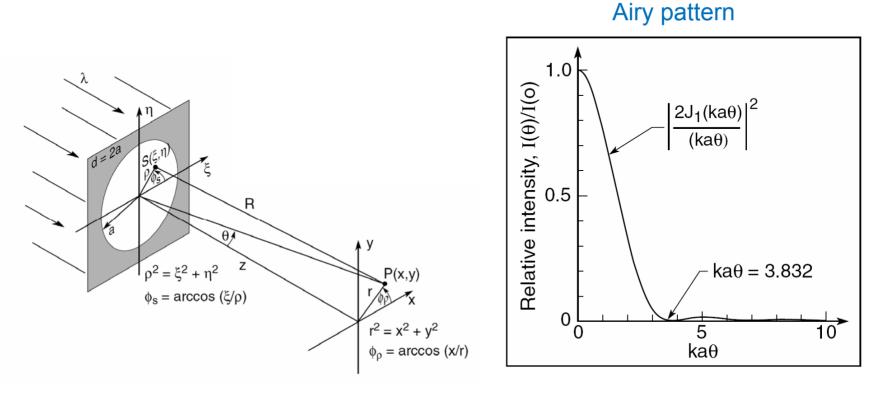
Brightness is the radiated power per unit area per solid angle at the source

$$B = \frac{P}{\Delta A_s \cdot \Delta \Omega_s}$$

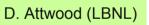
At the focus, available flux ~ B * δ^2 * NA² where δ is the spot size, hence the importance of high brightness source and large N.A. optics



Diffraction from a Circular Aperture



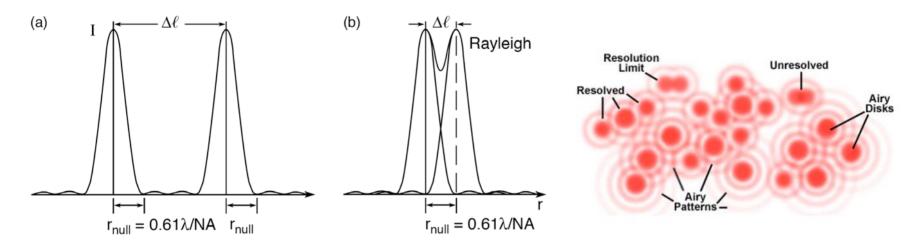
$$ka\theta = 3.822$$
$$\frac{2\pi}{\lambda}a\frac{r_{null}}{z} = 3.822$$
$$r_{null} \approx 0.61\frac{f}{a}\lambda = 0.61\frac{\lambda}{NA}$$





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Rayleigh's Criterion for Resolving Two Point Images



- Point sources are spatially coherent
- Mutually incoherent
- Intensities add
- Rayleigh criterion (26.5% dip)

Conclusion: With spatially coherent illumination, objects are "just resolvable" when

$$\operatorname{Res}|_{\operatorname{coh}} = \frac{0.61 \,\lambda}{\mathrm{NA}}$$

<= Diffraction limited resolution

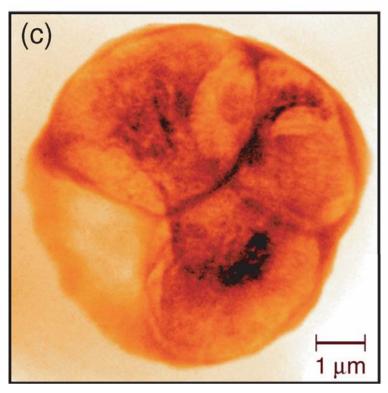
D. Attwood (LBNL)



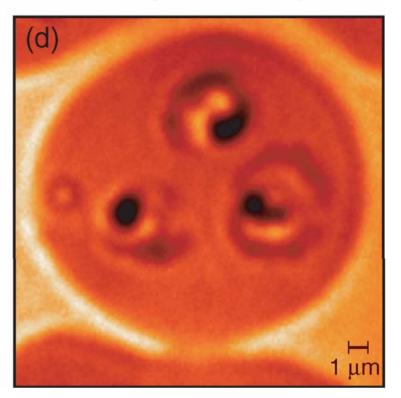
Resolution improves with smaller λ

Malaria-infected red blood cell

X-ray microscopy



Visible light microscopy



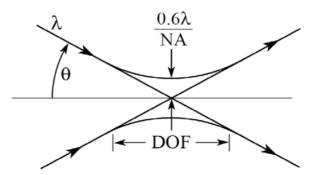
C. Magowan, W. Meyer-Ilse, and J. Brown (LBNL)



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Depth of Focus

DOF =
$$\pm \frac{\lambda}{2(NA)^2} = \pm \frac{1.34\delta^2}{\lambda}$$



DOF determines the sample thickness in 2D imaging and the maximum sample volume in 3D tomography

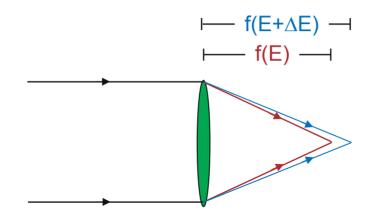
Soft X-rays Hard X-rays								
Wavelength, $\lambda(A)$	23.8 A		4.52 A (Rh)		2.29 A (Cr)		1.54 A (Cu)	
Energy, E	520 eV		2.7 KeV (L)		5.4 KeV (Ka)		8.0 KeV (Ka)	
Resolution, δ, nm	30	60	30	60	30	60	30	60
DOF (µm)	±1	±4	±5	±21	±10	±42	±16	±63
Si Transmission (%) at 0.5*DOF thickness	12%	0.02 %	20%	0.18%	63%	15%	80%	41%

W. Yun (Xradia)



Chromatic aberration

- Does focal length depends on λ?
 - Reflective optics: achromatic, can focus white beam, higher flux
 - Diffractive optics: f ~ E
 - Refractive optics: f ~ E²



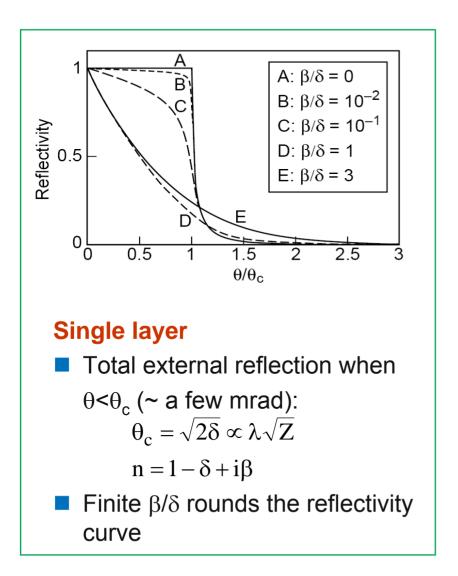


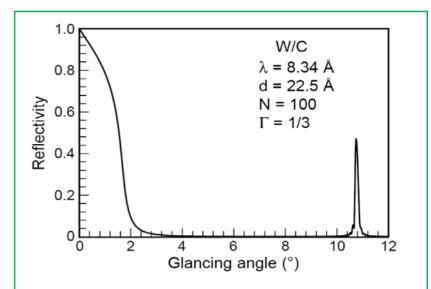
Reflective Optics

- Schwarzschild objective
- Wolter microscope
- Capillary optics
- Kirkpatrick-Baez mirrors



Reflectivity of single and multi-layer





Multilayers

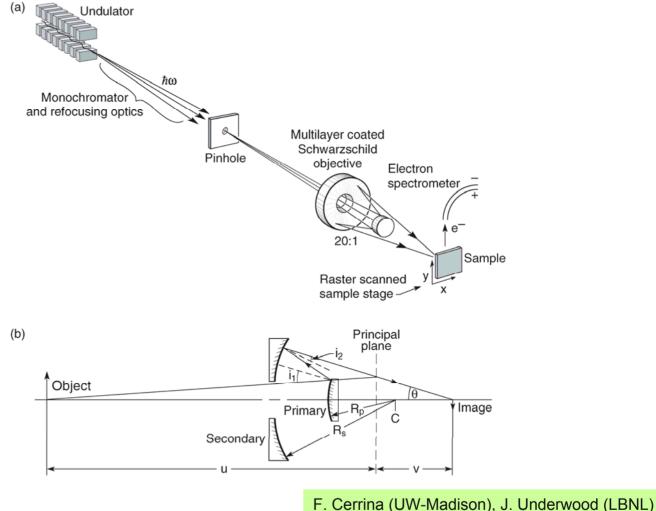
- Large θ means shorter mirror or larger acceptance
- ■Spectral bandwidth ~ a few %.

Cannot focus white beam



Schwarzschild Objective

- Near normal incidence with multilayer coating (126 eV)
- N.A. > 0.1
- Imaging microscope

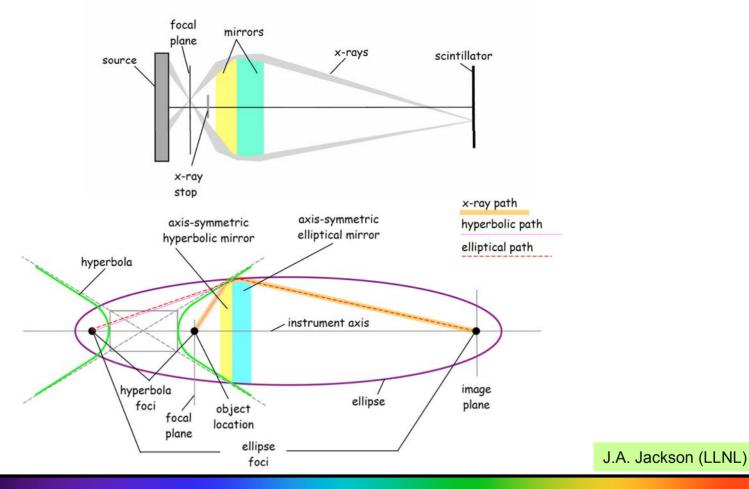




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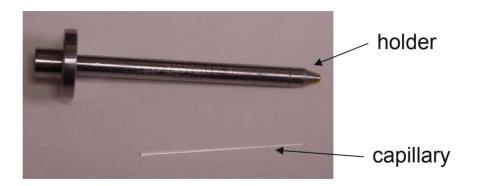
Wolter Type I Microscope

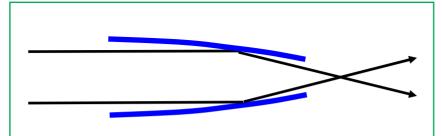
- Use 2 coaxial conical mirrors with hyperbolic and elliptical profile
- Imaging microscope
- Difficult to polish for the right figures and roughness





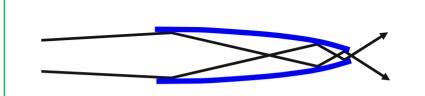
Glass capillary optics





One-bounce capillary

- Large working distance (cm)
- Compact: may fit into space too small for K-B
- Nearly 100% transmission
- N.A. ~ 2-4 mrad ($\leq 2\theta_c$)
- Difficult to make submicron spot



Multi-bounce condensing capillary

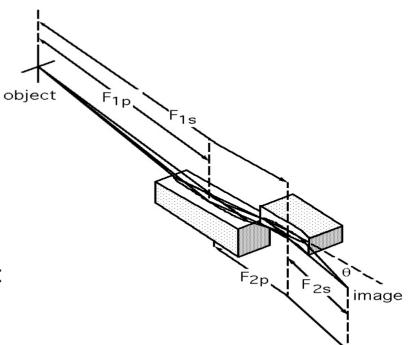
- Easy to make with small opening (submicron)
- Short working distance (100 μm)
- Low transmission

D. Bilderback (Cornell)



Kirkpatrick-Baez mirrors pair

- A horizontal and a vertical mirror arranged to have a common focus
- Achromatic: can focus white beam
- Different focal lengths and demagnifications: can be used to produce ~ round focal spot

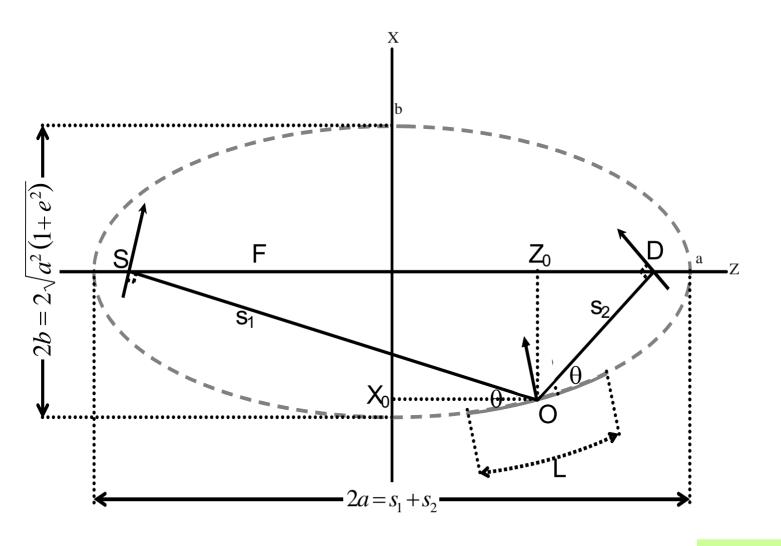


Very popular for focusing in the 1-10 μ m regime: relatively easy to make, longer mirrors can be used for higher flux

For submicron focusing, mirrors with precise elliptical profile are required (figure error < 1 μ rad)



Elliptical x-ray mirrors



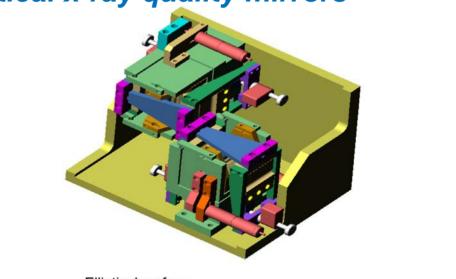
A. Macrander (APS)



Three methods used for elliptical x-ray quality mirrors

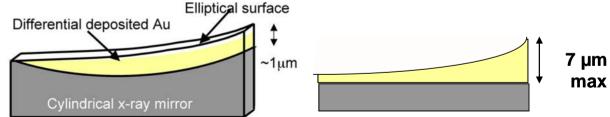


- ESRF



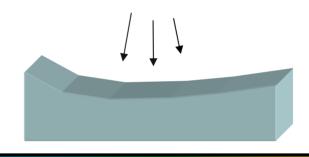


- APS (C. Liu)



Differential polishing

- Osaka/Spring8
- APS/Tinsley

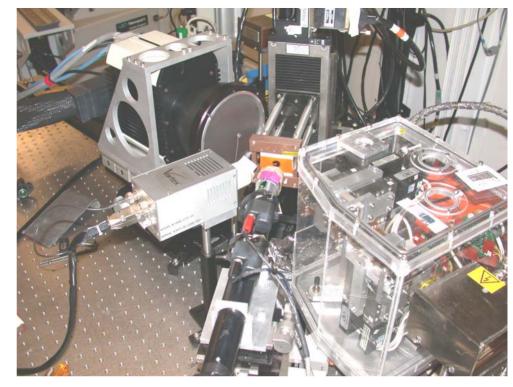


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G. Ice (ORNL)

K-B mirrors are very popular for micron scale focusing

- At the APS, K-B microprobes with 100 and 200-mm long bent mirrors are common:
 - MR-CAT 10-ID
 - (J. Kropf, K. Kemner)
 - GSECARS 13-ID-C
 - (S. Sutton, M. Rivers)
 - BioCAT 18-ID
 - (T. Irving, R. Barrea)
 - PNC/XOR 20-ID
 - (S. Heald, D. Brewe)
- Monochromatic flux ~ 10¹¹–10¹² ph/sec



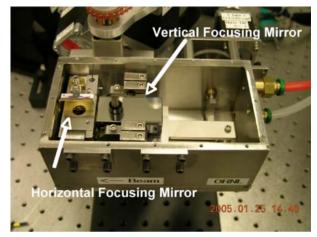




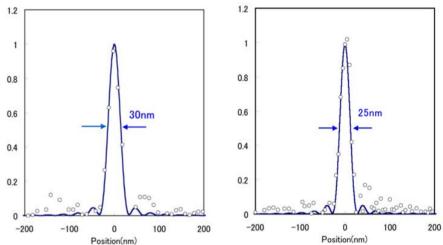
KB mirror systems for nanofocusing

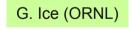
- APS/ORNL collaboration KB optics
 Poly/mono Beams 85 x 95 nm
- ESRF 45 nm
- Osaka/Spring-8 ~ 25 nm x30 nm
- Simple KB system diffraction limit ~17 nm

APS/ORNL 34-ID



Osaka mirrors





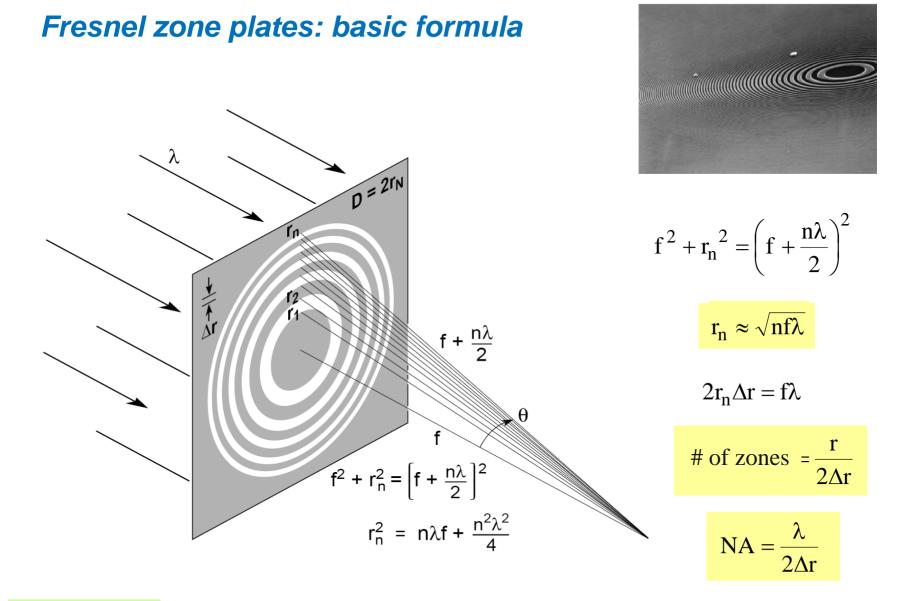


Diffractive Optics

Fresnel zone plates (FZP)

Multilayer Laue Lens (MLL)

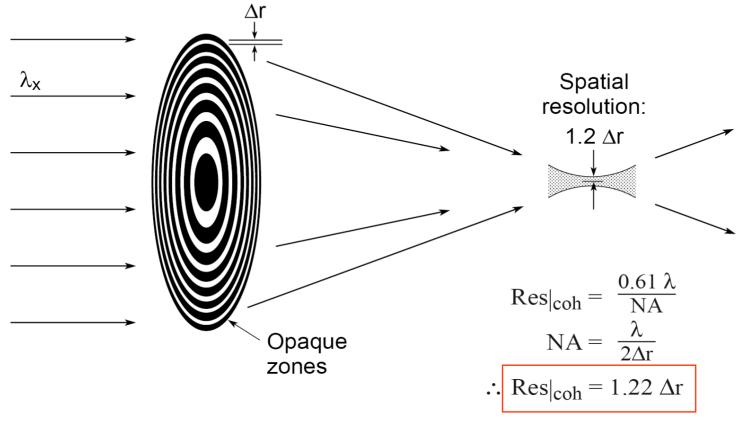




D. Attwood (LBNL)



Diffraction limited resolution

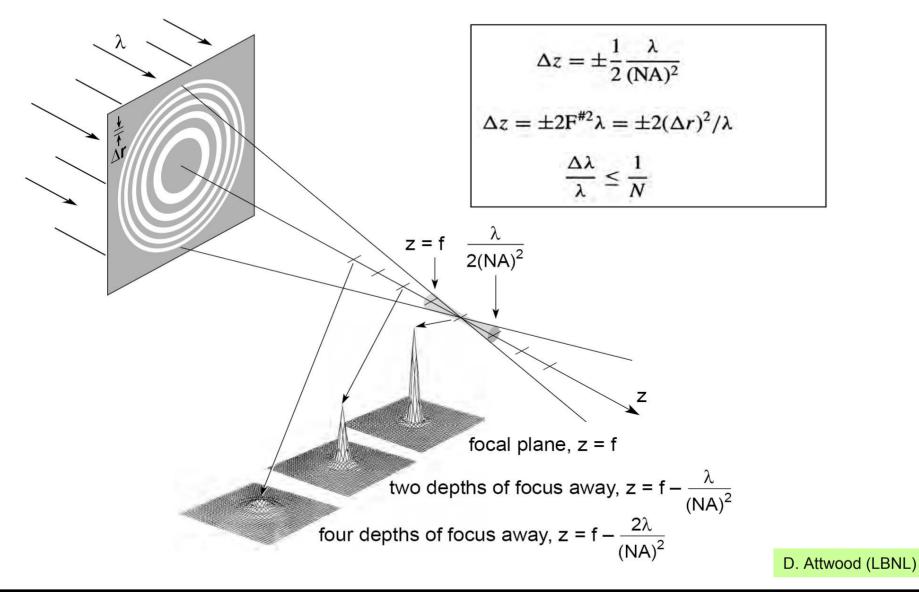


Coherent illumination

D. Attwood (LBNL)

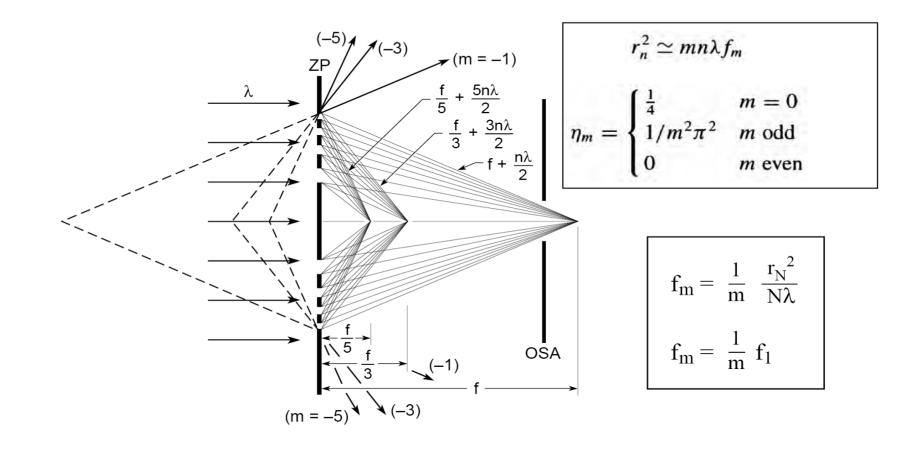


Depth of focus and spectral bandwidth





Higher orders and negative orders



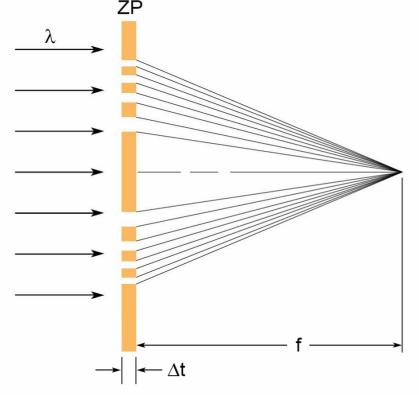
D. Attwood (LBNL)



Efficiency: Phase vs Amplitude Zone Plates

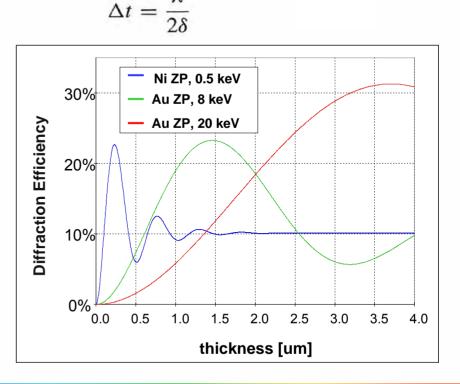
Efficiency of an amplitude ZP with opaque zones ~ 10%

Efficiency of a phase ZP with π -phase shift ~ 40%



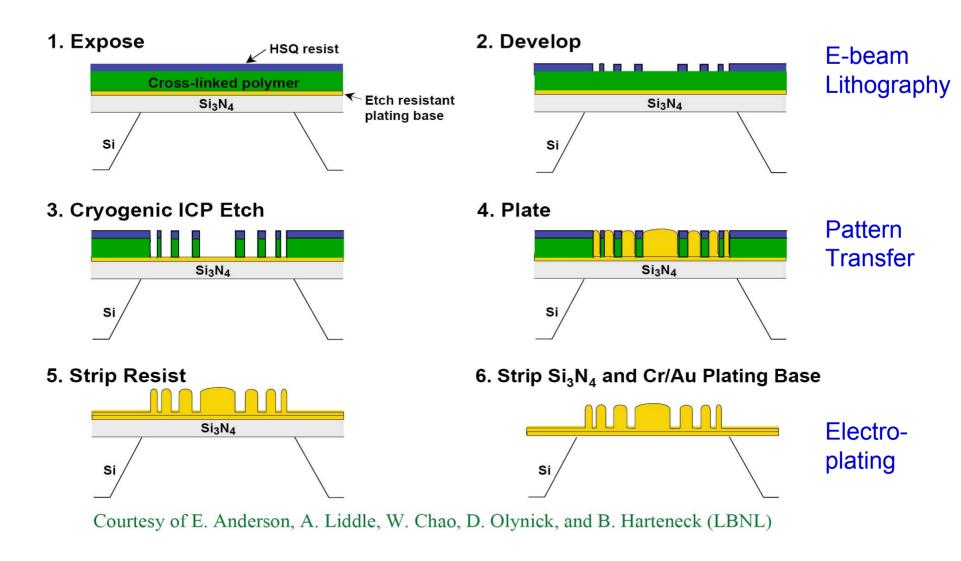
$$\Delta \phi = \left(\frac{2\pi\delta}{\lambda}\right) \Delta t$$

For a π -phase shift





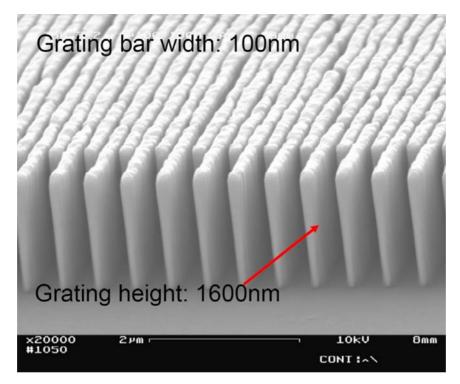
Fabrication of FZP





Recent Hard X-ray Zone Plates

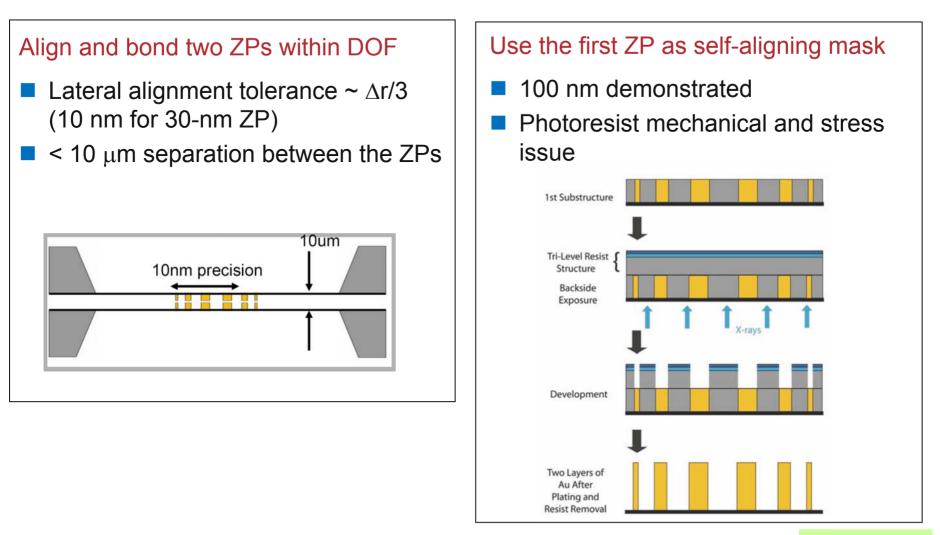
- ▲r = 32 nm, 450 nm thick, AR = 14
- ▲r = 24 nm, 300 nm thick, AR = 12.5
- To achieve good efficiency, aspect ratio needs to be increased (e.g. needs 1.5 µm thick for optimal efficiency at 8 keV)



W. Yun (Xradia)



Other means to increase the aspect ratio



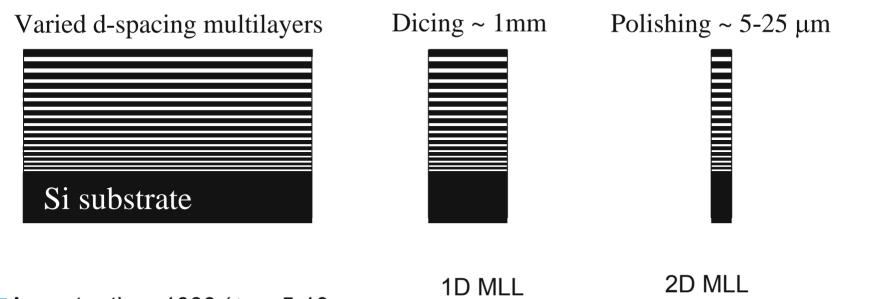
W. Yun (Xradia)



Even with stacking, aspect ratio > 100 is probably beyond lithographic zone plates!

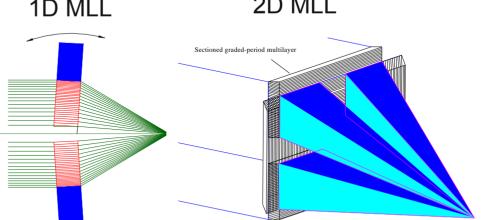


Multilayer Laue Lens: novel approach for high aspect ratio



Aspect ratio > 1000 ($\Delta r = 5-10$ nm, 10 μ m thick) demonstrated

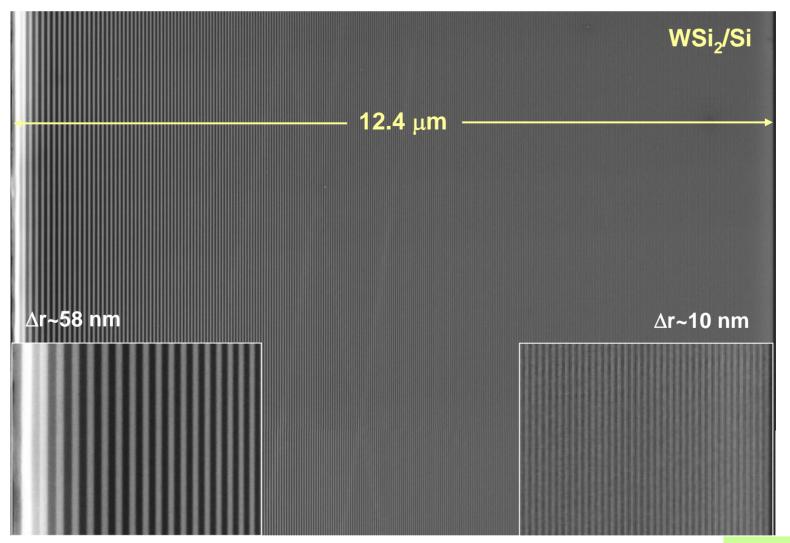
Engineering challenge of aligning and assembling 2 or 4 MLLs to produce a single optics



A. Macrander (APS)



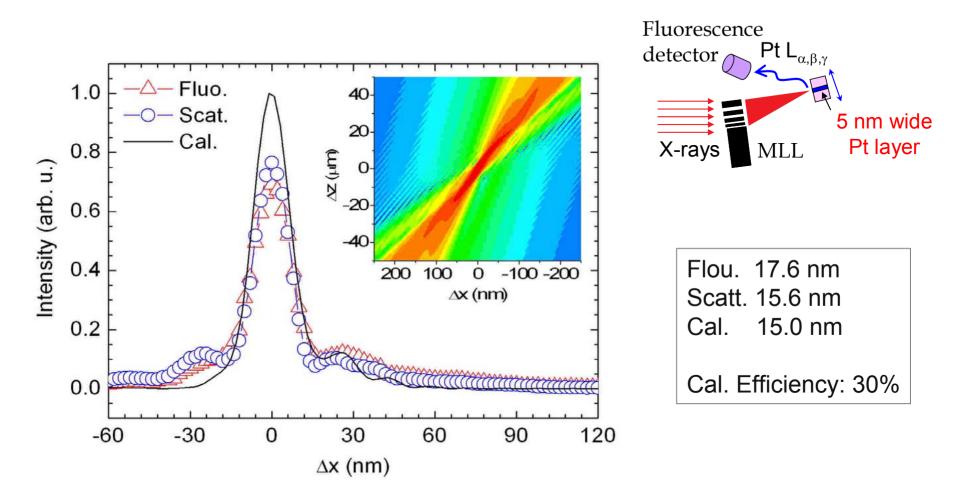
SEM image of an MLL



A. Macrander (APS)



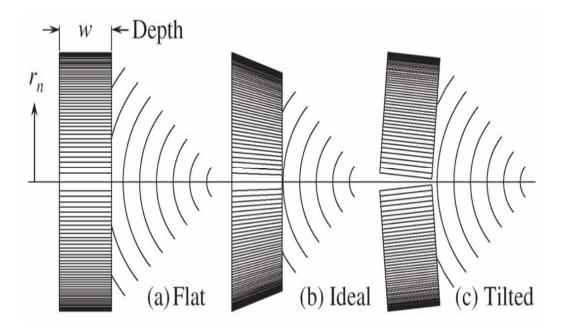
Best measured line focus of MLL



H.C. Kang, H. Yan, et al., submitted



Thin vs Thick Zone Plate



When aspect ratio increases, effects from dynamical diffraction vs kinematic scattering need to be considered

Zones should be inclined locally to satisfy Bragg condition

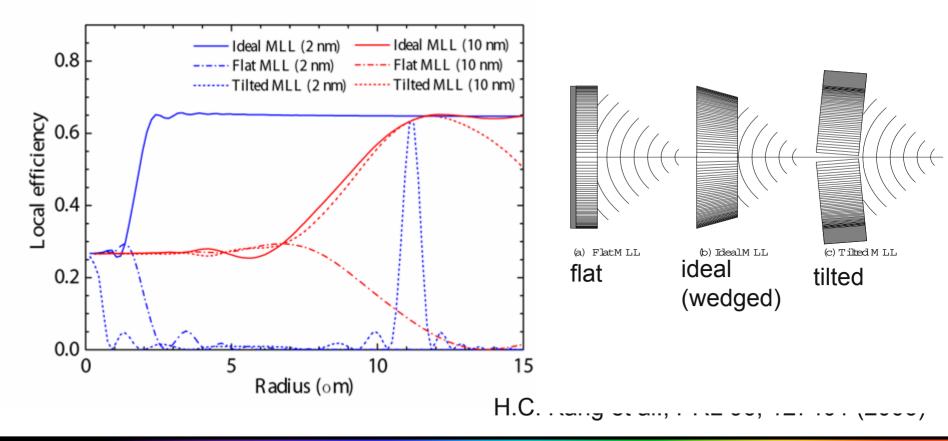
Thin to thick transition: $w = (2\Delta r)^2 / \lambda \sim DOF$



For flat structure, local efficiency decreases at large r

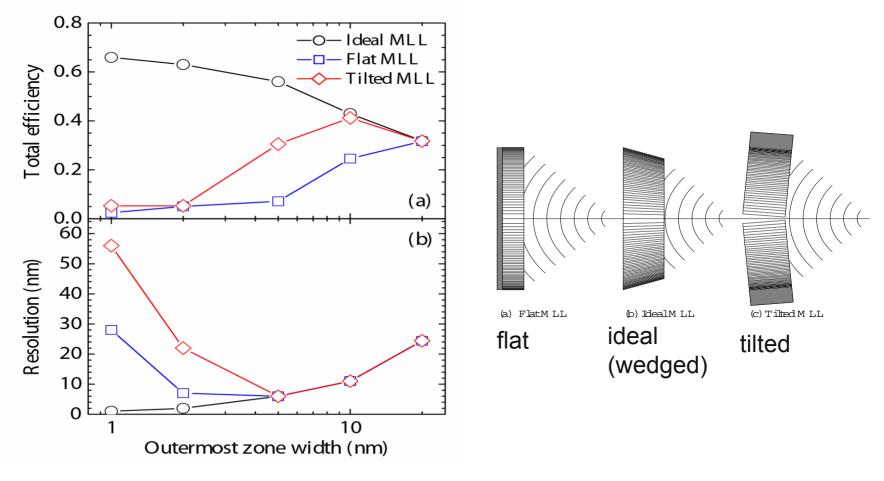
■For tilted or ideal wedged structures, efficiency actually increases beyond the thin phase ZP limit of 40%

This effect is enhanced for high resolution (small Δr)





Despite lower overall efficiency, both flat and tilted structure can achieve ~ 5 nm resolution



H.C. Kang et al., PRL 96, 127401 (2006)



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Refractive Optics

Compound refractive lens (CRL)



Refraction & Absorption

Refraction of hard x-rays in matter weak

 $n = 1 - \delta + i\beta$, $(\delta \approx 10^{-6} \text{ @20 keV})$

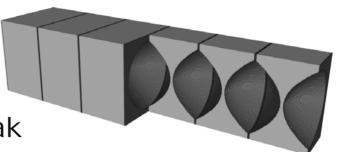
- strong curvature of lens surfaces
- stacking of many lenses behind each other

Absorption of hard x-rays in matter strong

- lenses must be made of low Z material (Be, B, C, Al, ...)
- lenses should be made as thin as possible

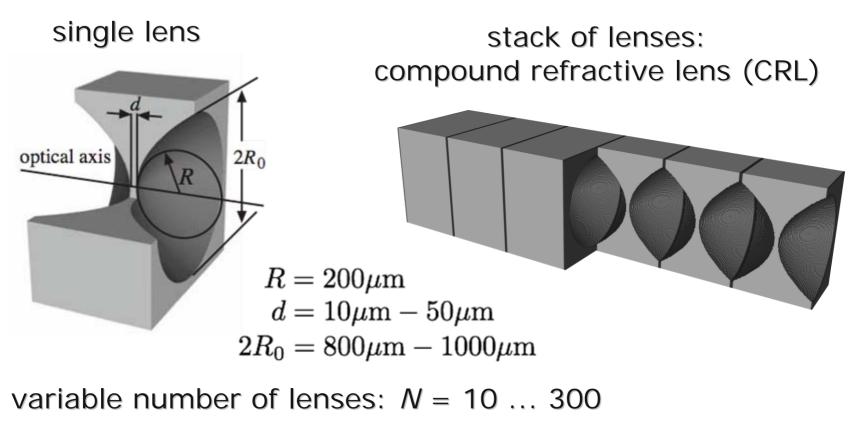
Refractive index *n* smaller than 1:

focusing lens must be concave





Parabolic Refractive X-Ray Lenses



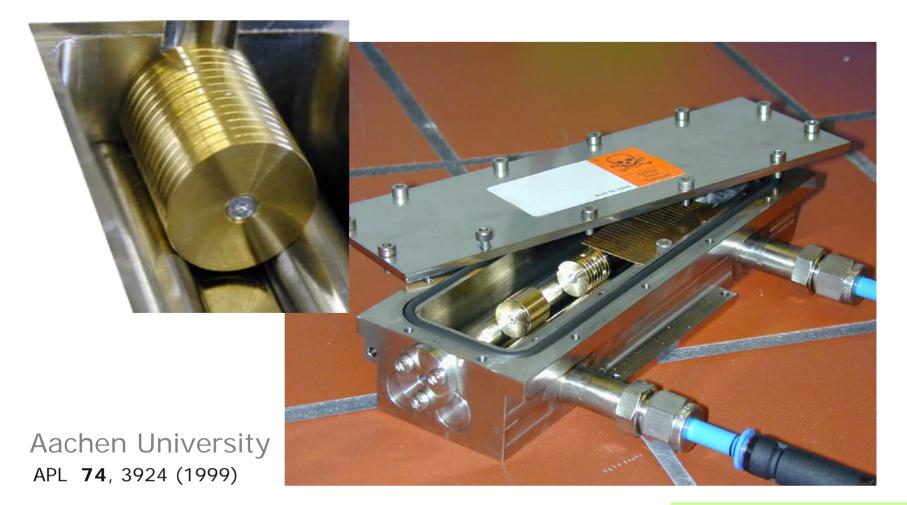
parabolic profile:

No spherical aberration

→ imaging optic

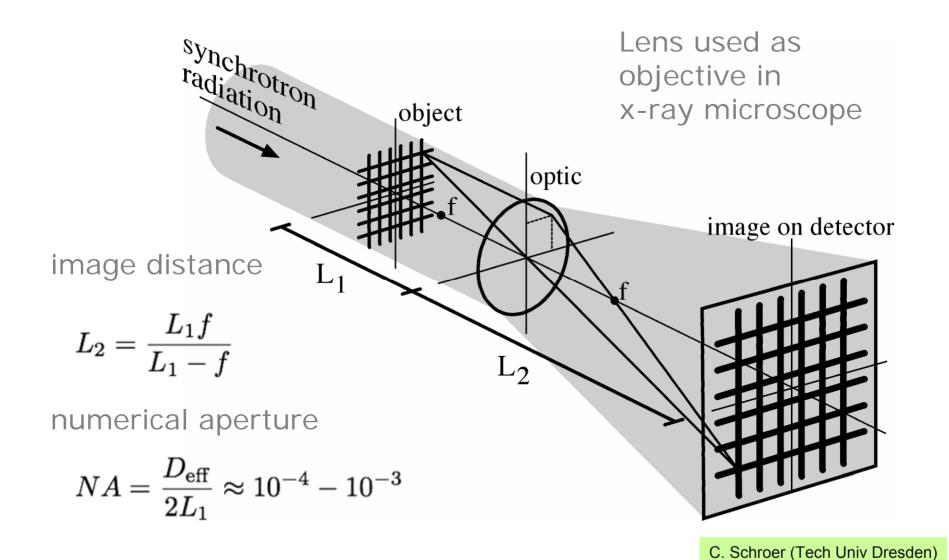


Parabolic Refractive X-Ray Lenses



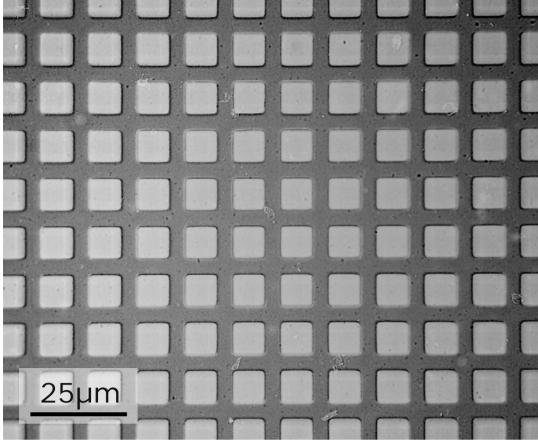


Imaging with Magnification



Undistorted (Magnified) Image

Parabolic profile of lenses is crucial to good image quality

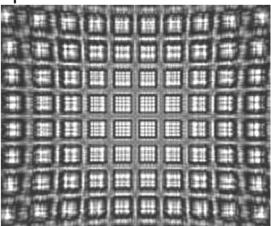


Ni-mesh (2000mesh)

parameters:

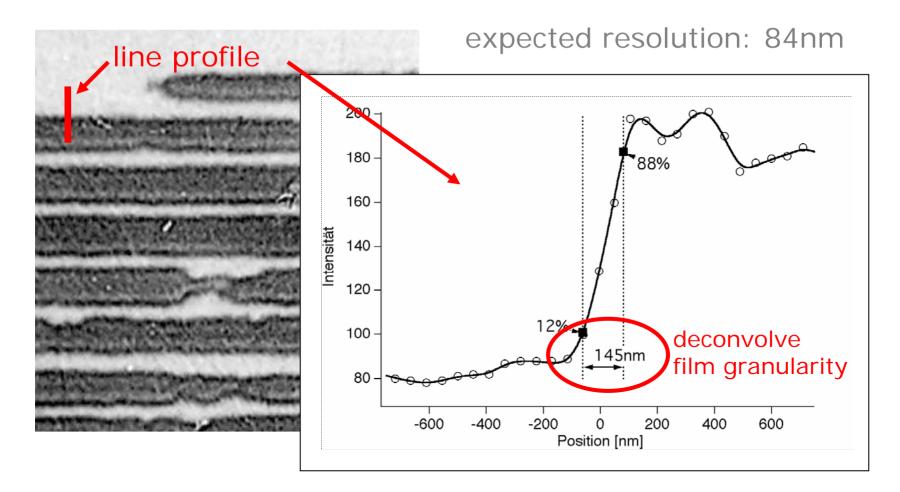
- *E* = 12keV
- N = 91 (Be)
- *f* = 495mm,
- *m* = 10x

simulation: spherical lens





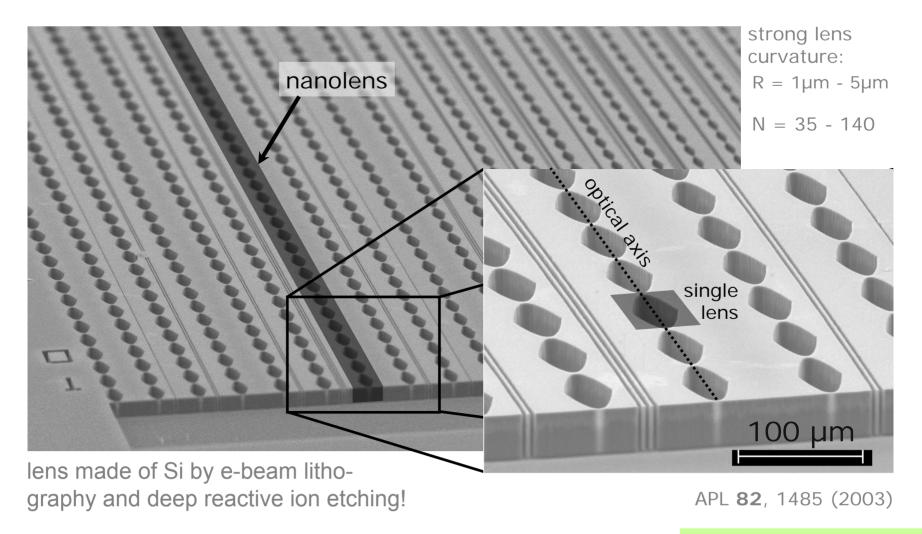
Full-Field Imaging: Resolution



resolution of Optic: 105nm ± 30nm

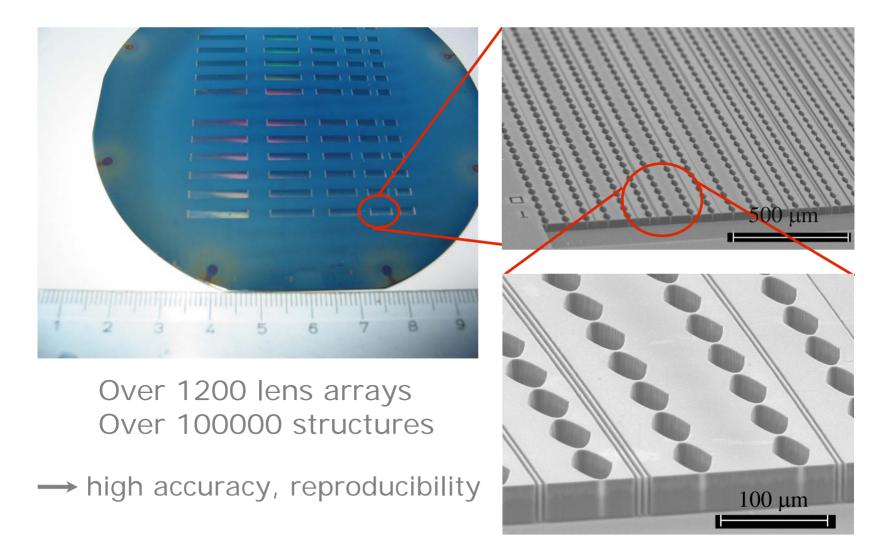


1-Dimensional Nanofocusing Lenses (NFLs)



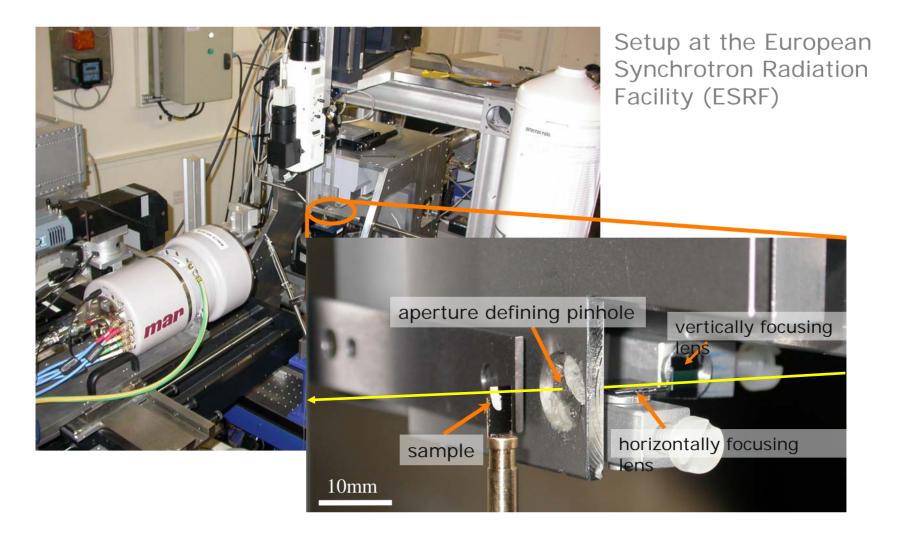


Fabrication of Si Nanofocusing Lenses





Crossed Nanofocusing Lenses





Focusing with NFLs

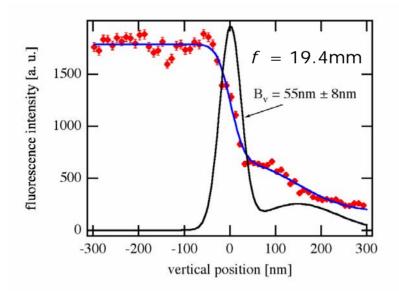
Si lens: $E = 21 \text{keV}, L_1 = 47 \text{m}$

source:

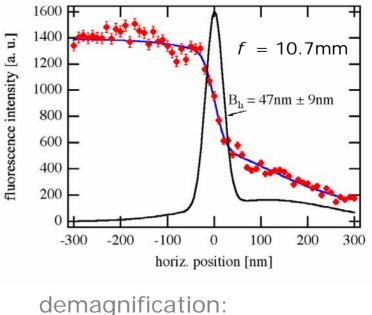
ID13 low- β invac. undulator

source size: 150 x $60\mu m^2$

vertical focus: 55nm



horizontal focus: 47nm



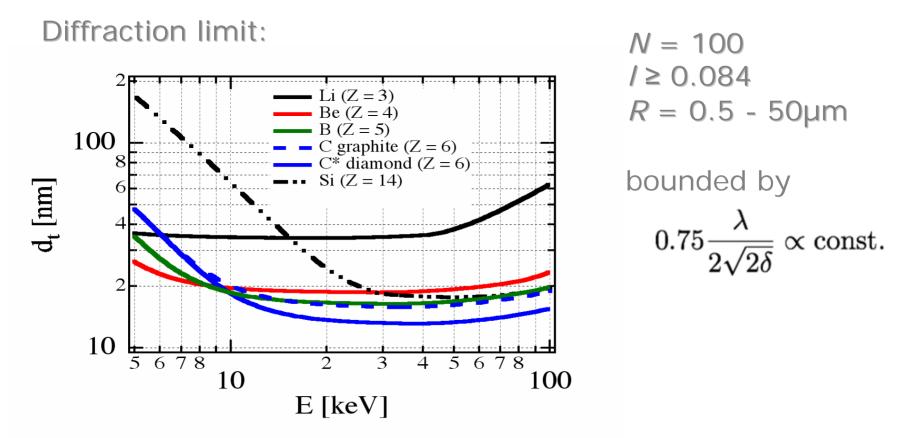
~ 2400 x 4400

flux: 1.7 · 108ph/s

APL 87, 124103 (2005)



Effective Aperture and Diffraction Limit



Best materials: high density and low Z



Summary: best resolution achieved currently

K-B mirrors : 25 x 30 nm

- H. Mimura et al., APL 90, 051903 (2007);
- S. Matsuyama et al., RSI 77, 103102(2006).

FZP: 30 nm (third order focus)
 G-C. Yin et al., APL 89, 221122 (2006).

MLL: 17 nm line focus H.C. Kang, H. Yan, et al., submitted.

CRL: 47 x 55 nm

C. G. Schroer et al, APL 87, 124103 (2005).

Waveguides: 25 X 47 nm

A. Jarre et al., PRL 94, 074801 (2005).



Summary: other considerations

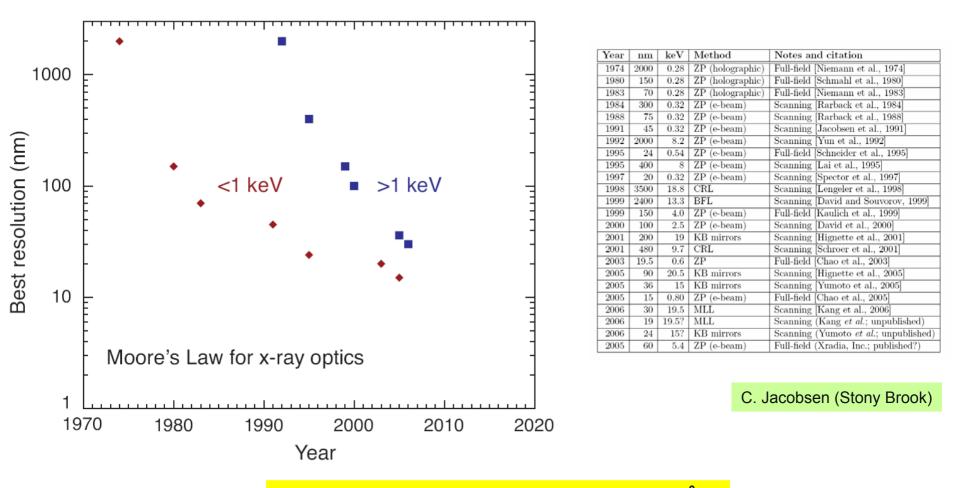
	K-B mirror	FZP/MLL	Refractive Lens
Resolution	25 x 30 nm	30/17 nm	47 x 55 nm
Flux density gain	> 500,000	> 500,000	10,000
Chromatic aberration	Achromatic	1/λ	1/λ ²
Coherence preservation	Fair	Good	Acceptable
Easy to use	Require effort	Good	Fair



Future Prospects



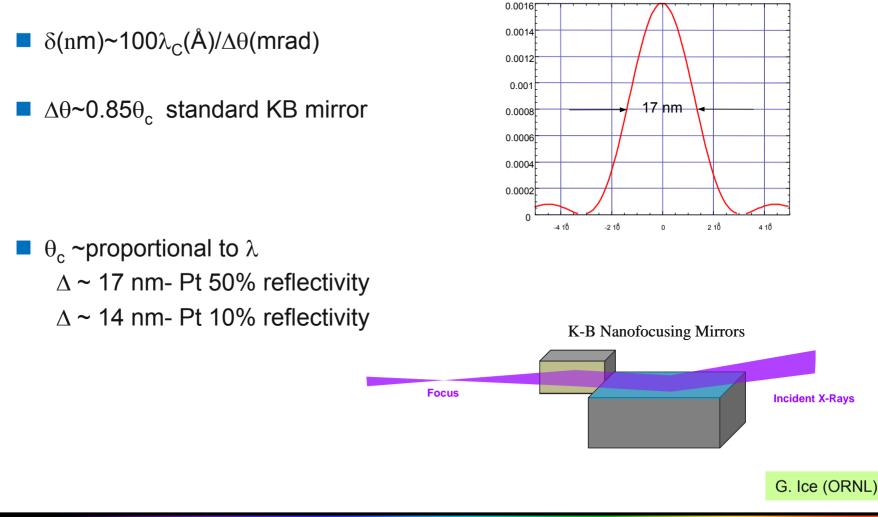
Resolution had improved dramatically



Where is the limit? 1 nm? 1 Å?



Reflective Optics: Focal size ultimately limited by θ_c

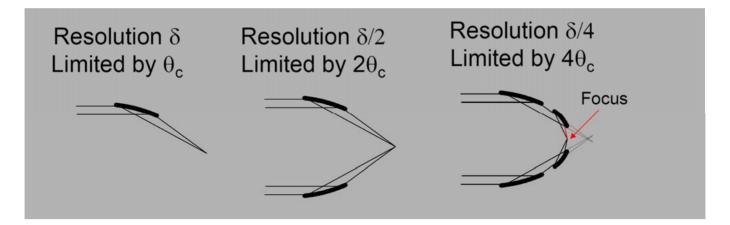


Diffraction limit



Reflective optics: radical approaches needed for sub 10 nm

- Multilayers \rightarrow 4-5 nm
 - ESRF/Osaka
 - Limited bandpass-ideal for undulator harmonic
- Coaxial/multiple reflections \rightarrow 3-4 nm



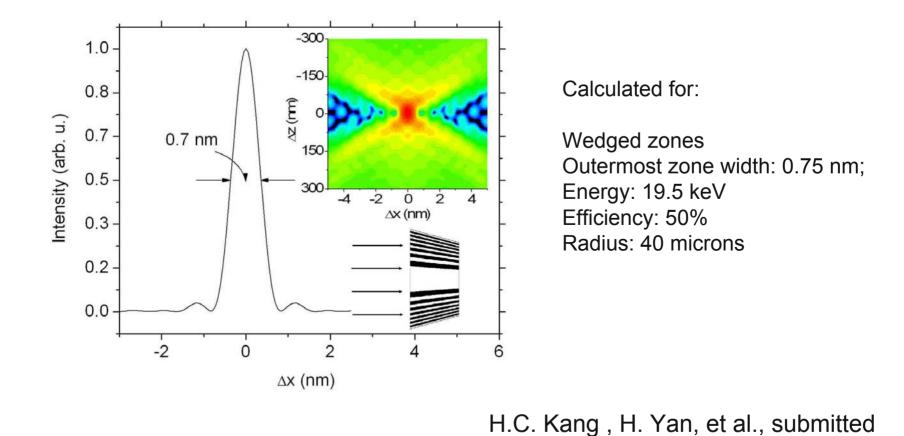
Combination of both $\rightarrow \rightarrow$ 1 nm?

G. Ice (ORNL)



MLL: Presently Feasible Outermost Zone Width

(0.75 nm layer width has been demonstrated: Y. Chu et al., RSI 73, 1485 (2002))

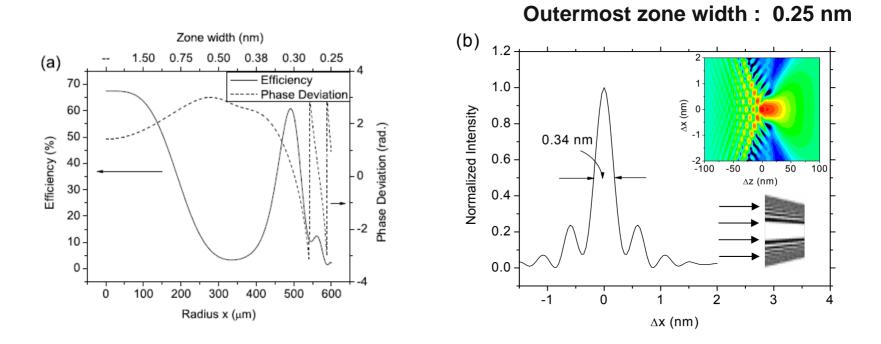




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MLL: when ∆r ~ single atomic layer

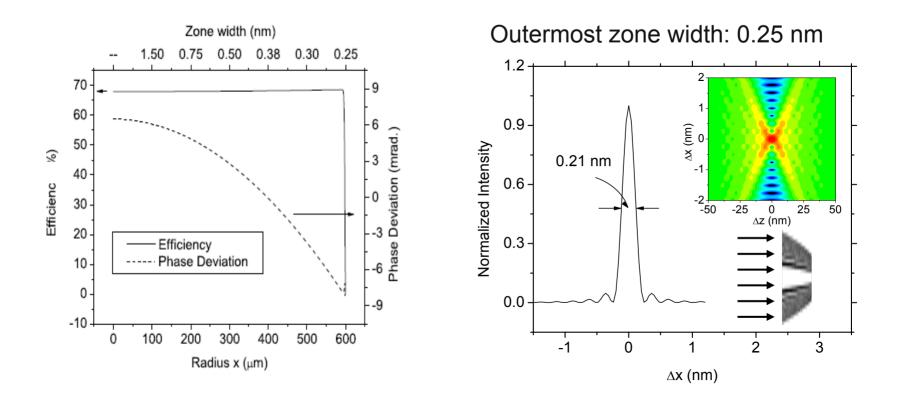
Each zone is tilted progressively to satisfy the local Bragg condition, resulting in a wedged shape.



H. Yan et al., <u>http://arxiv.org/abs/0704.3982</u>; PRB, in press.



Utimately parabolically curved interfaces are needed



H. Yan et al., <u>http://arxiv.org/abs/0704.3982</u>; PRB, in press.



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Refractive Lens: Adiabatically Focusing Lens (AFL)

Current limitation:

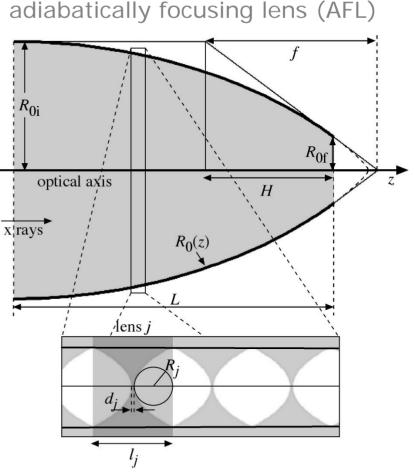
geometry of lens limits refractive power per unit length for given aperture:

 $\frac{1}{lf_s} = \frac{2\delta}{lR} \approx \frac{2\delta}{R_0^2}$

Solution:

adjust R_0 to fit the converging beam as it is focused

PRL 94, 054802 (2005)







Example AFL

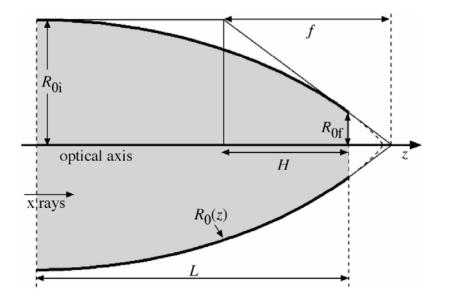
(a) **Diamond lens:** 18.9µm low atomic number Z and high density p N = 1166 individual lenses entrance aperture: 18.9µm 8 6 exit aperture: 100nm z [mm] (b) f = 2.3mm 40. r [mm] diffraction limit: 4.7nm -40 10 20 30 40 compare to NFL: $z - L[\mu m]$ (c) 40 same aperture r [mm] diffraction limit: 14.2nm -40 ⊢► 10 20 30 40 0 $z - L [\mu m]$ C. Schroer (Tech Univ Dresden)

contracting wave field inside lens



AFLs Made of Silicon

entrance aperture: $2R_{0i} = 20\mu m$ exit aperture: $2R_{0f} = 1\mu m$ energy: 10 - 20keV in 500eV steps



properties:

f = 2.7mm $d_{\rm t} = 12.6$ nm

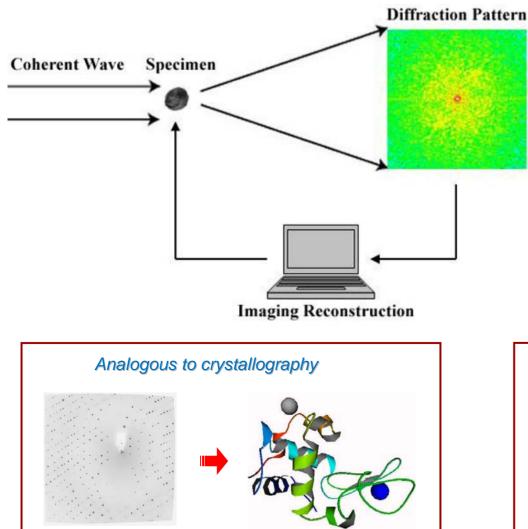
as horizontal lens in x-ray nanoprobe (e. g. ID13 ESRF):

 $L_1 = 47$ m, source size: 150µm

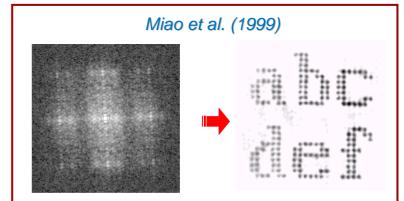
horizontal focus: 15.3nm (17400 x reduction)



(Lensless) Coherent Diffraction Imaging



- Coherent diffraction imaging is much like crystallography but applied to noncrystalline materials
- Lateral resolution can in principle approach λ, not limited by N.A. of available optics. Long depth of focus.
- Requires a fully coherent x-ray beam





Conclusions

- Microfocusing optics is an vibrant field with many parallel developments:
 - Reflective optics
 - Diffractive optics
 - Refractive optics
- Resolution had improved dramatically over the last two decades. 30-50 nm are currently available.
- Future spot size of a few nm is physically possible, but requires great engineering effort. There may be sufficient sensitivity and resolution to detect single atoms?
- However, microprobes of all length scale are required for most scientific studies. It is likely that 10 nm 10 μ m will remain the primary workhorse.

