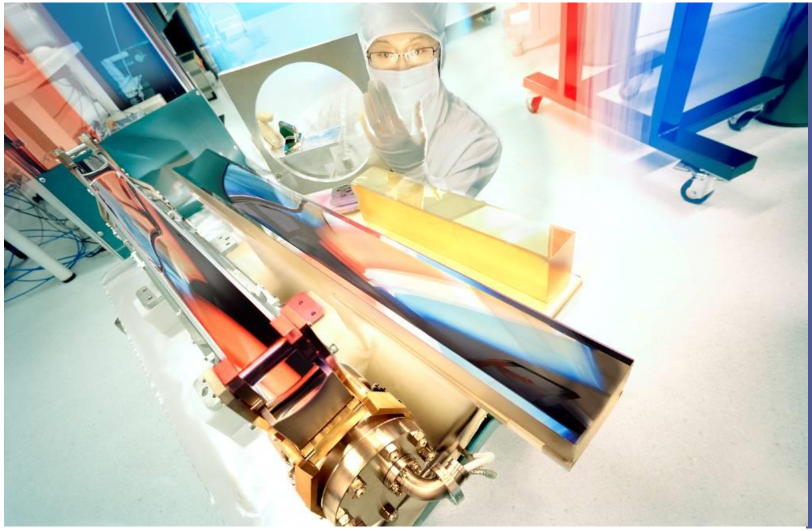
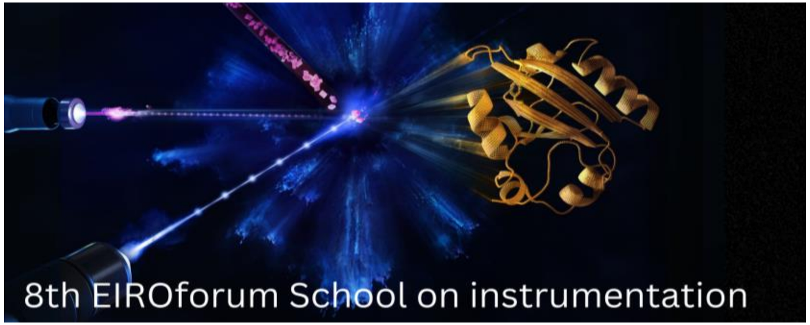


X-ray optics at accelerator-based light sources

R. Barrett

ESRF X-ray Optics Group

barrett@esrf.fr



OUTLINE

- Characteristics of Synchrotron Radiation Sources
- Why do we need X-ray optics?
- X-ray Mirrors
- Crystal monochromators
- Focusing optics

ESRF – The European Synchrotron

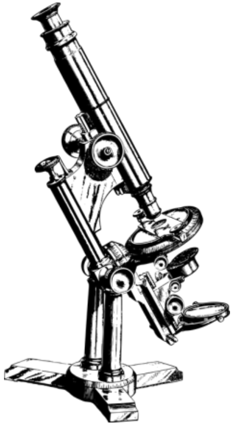
ESRF – THE EUROPEAN SYNCHROTRON

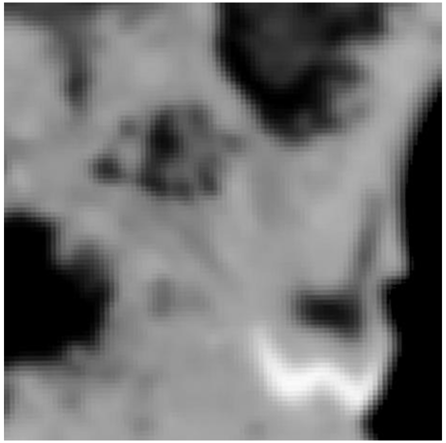
Super microscope

producing X-rays

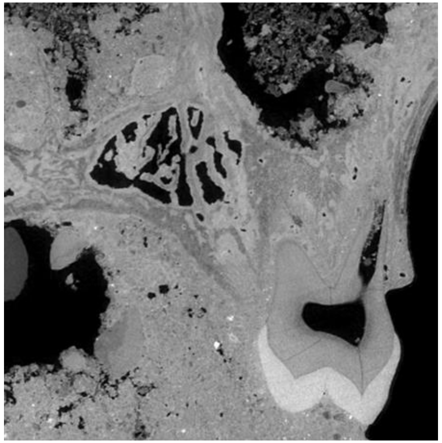
10 trillion times brighter

than in hospitals

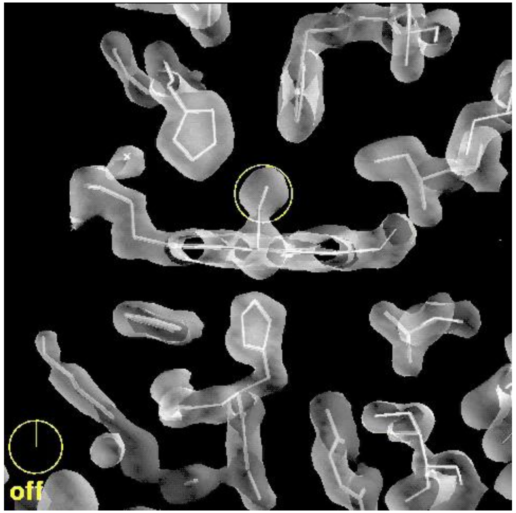




In hospital



At the ESRF

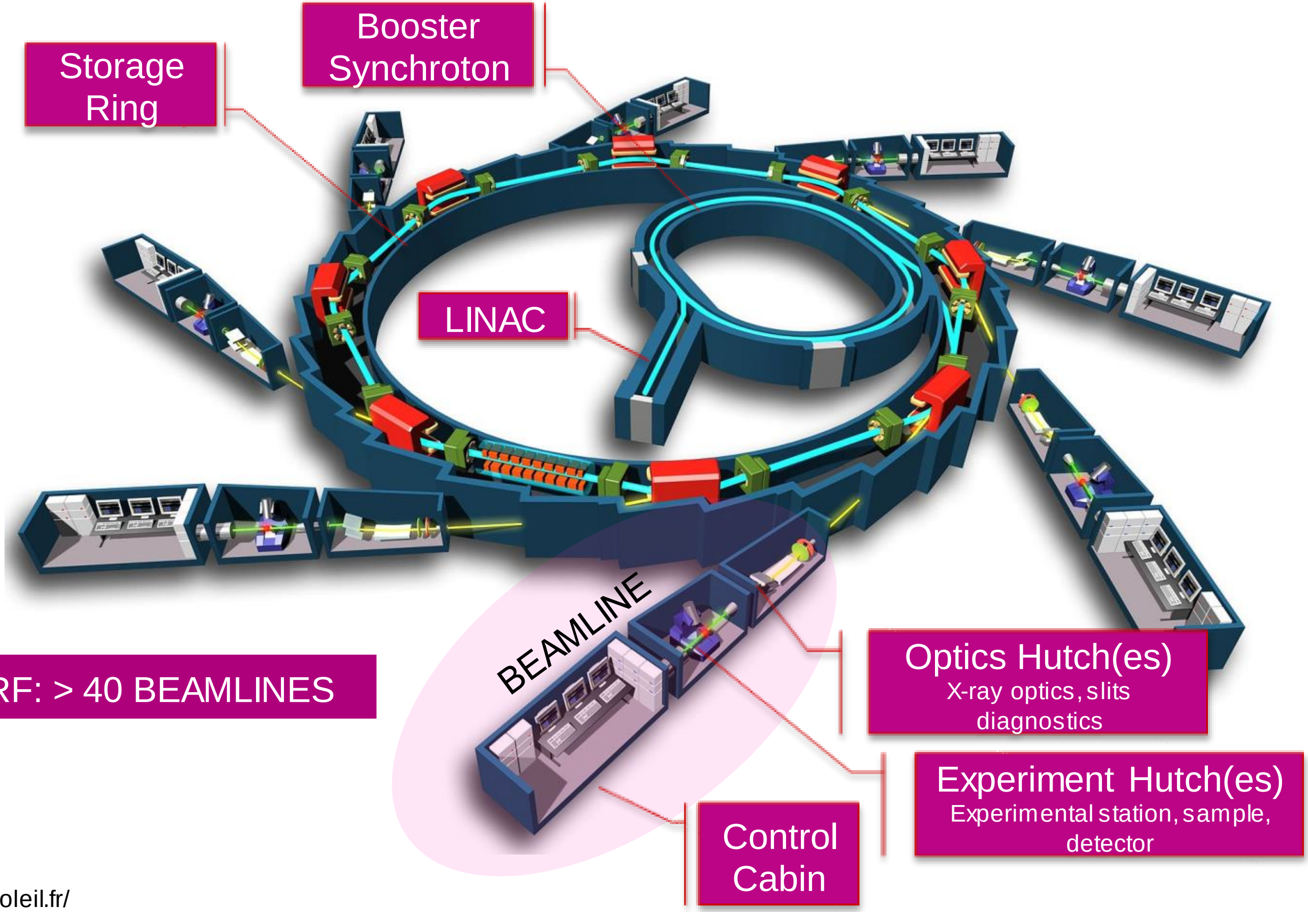


Schotte et al., 2003, Science 300

better resolution

- spatial
- spectral
- time

SCHEMATIC OF A SYNCHROTRON RADIATION (SR) LIGHT SOURCE



Storage Ring

Booster Synchroton

LINAC

BEAMLINE

Optics Hutch(es)
X-ray optics, slits diagnostics

Experiment Hutch(es)
Experimental station, sample, detector

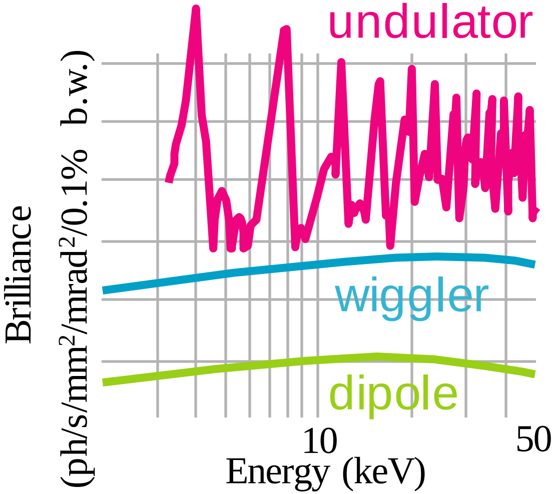
Control Cabin

ESRF: > 40 BEAMLINES

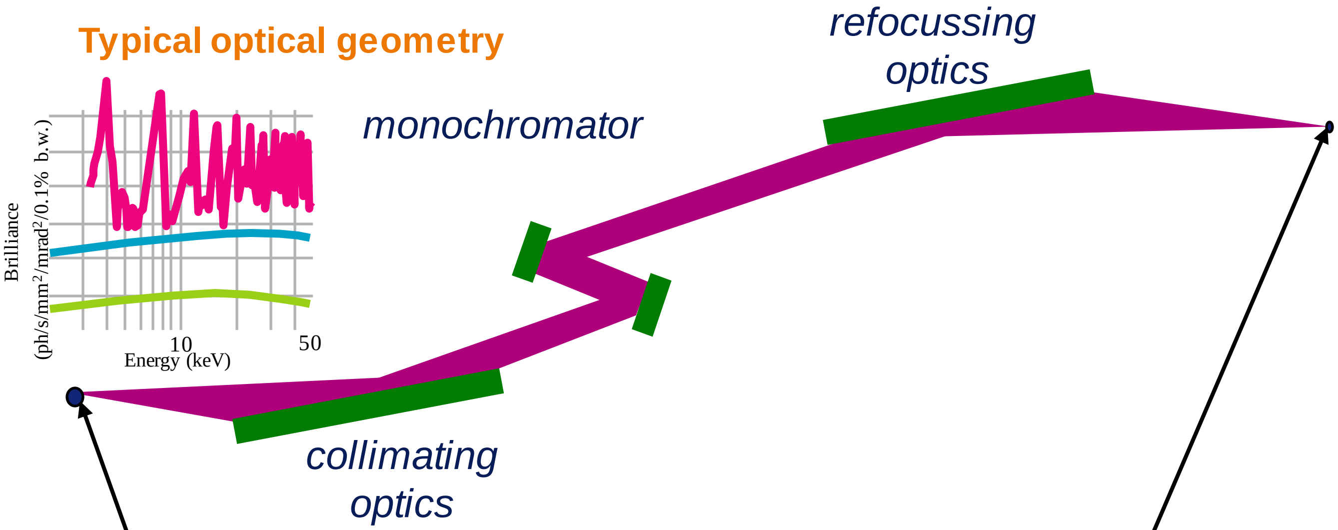
http://www.synchrotron-soleil.fr/

X-RAY BEAMS AT 3RD AND 4TH GENERATION SR SOURCES

- Beam size
  - Unfocused: few mm to few cm (source is weakly divergent)
  - Focused beam: < 50 nm to ~10’s µm
- Energy range/tunability
  - 0.1 eV < E < 0.5 MeV (at ESRF mostly 3-100 keV ≈ 4-0.125 Å)
- Energy bandwidth ($\Delta E/E$):
  - 10^{-2} to 10^{-8} at sample, typically ΔE ~ few eV @ 20keV
- Polarized radiation
  - 100% linear or circular or elliptical
- Pulsed radiation
  - Typically 50 ps pulses every ns
- High degree of coherence
- Photon Flux
  - Brilliance: 10^{22} ph/sec/mrad^2/mm^2/0.1%bw (10^{11} higher than conventional sources) ⇒ photon flux (@ $\Delta E/E = 10^{-4}$): 10^{9}-10^{14} ph/s
  - Extremely variable photon rates on detectors (< 1 ph/s to full beam flux)
- Power
  - Several kW total power, several 100 W/mm^2 power density (white beam)



SYNCHROTRON BEAMLINES


Typical optical geometry

OPTICS

?

Source

- spectrum (ΔE/E)
- emittance (size x divergence)
- degree of spatial coherence
- brilliance (ph/s/mm^2/mrad^2/0.1%bw)
- polarisation (linear, circular, elliptic)

Sample

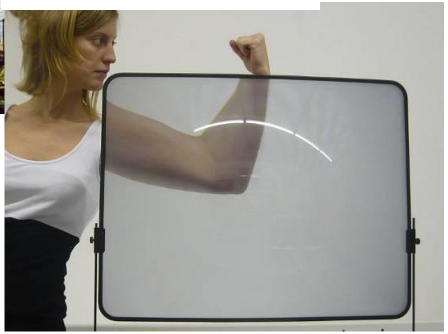
- beam size (µm)
- beam divergence (µrad)
- flux (ph/s)
- temporal coherence: ΔE/E
- spatial coherence
- polarisation

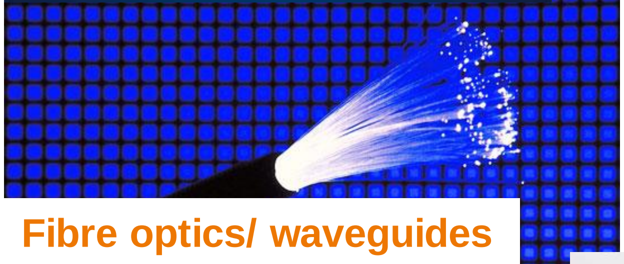
VISIBLE LIGHT OPTICS

Refractive lenses

Polarising Optics

Fresnel lenses

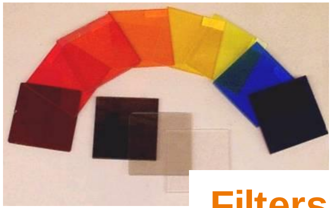




Fibre optics/ waveguides

Diffractive optics

Mirrors

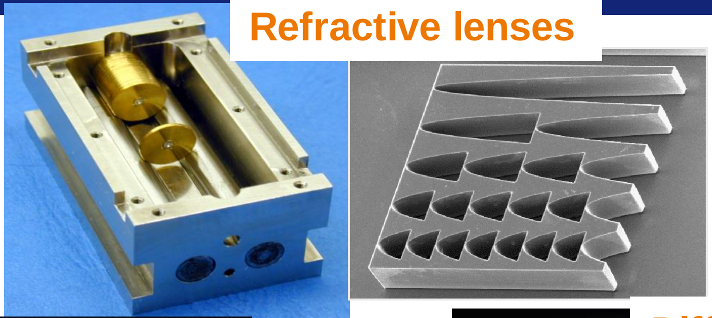


Filters

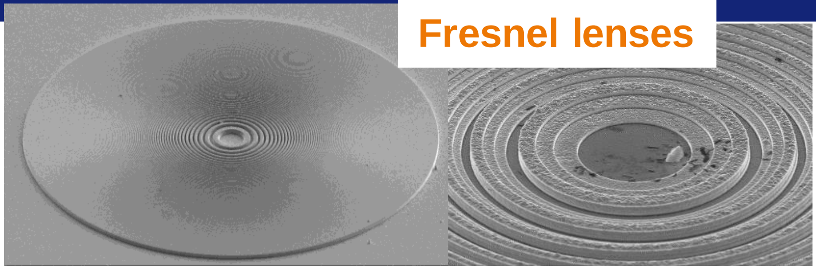
+ interferometers, …

X-RAY OPTICS

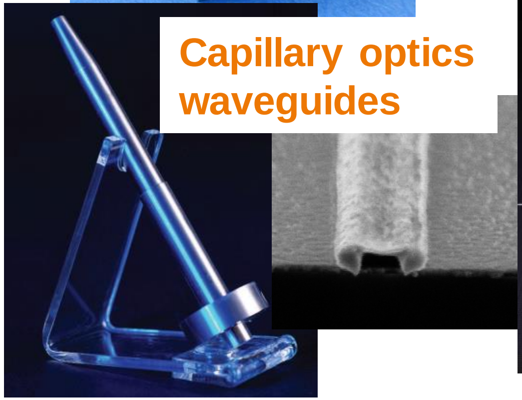
Refractive lenses



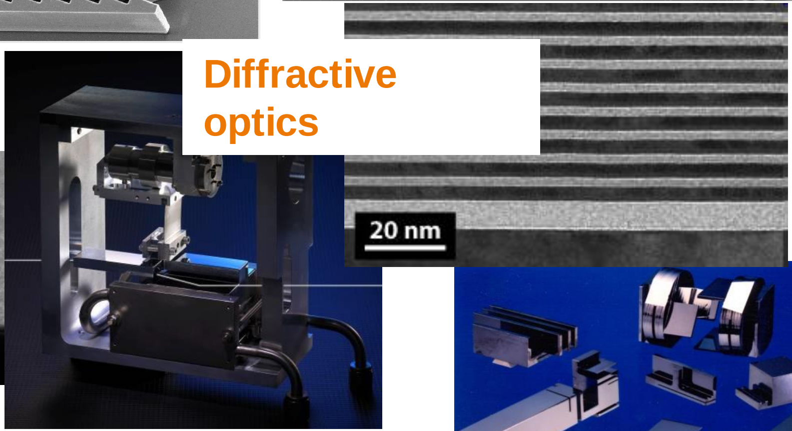
Fresnel lenses

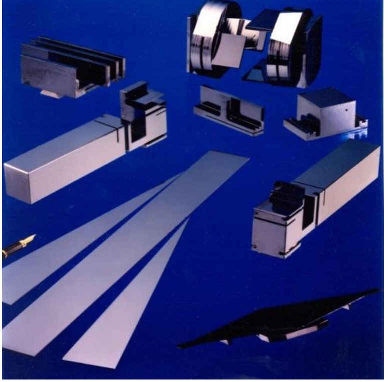


Capillary optics waveguides



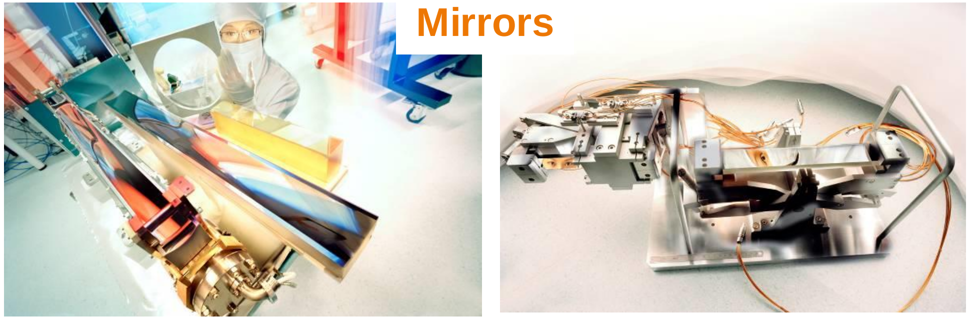
Diffractive optics





Filters

Mirrors



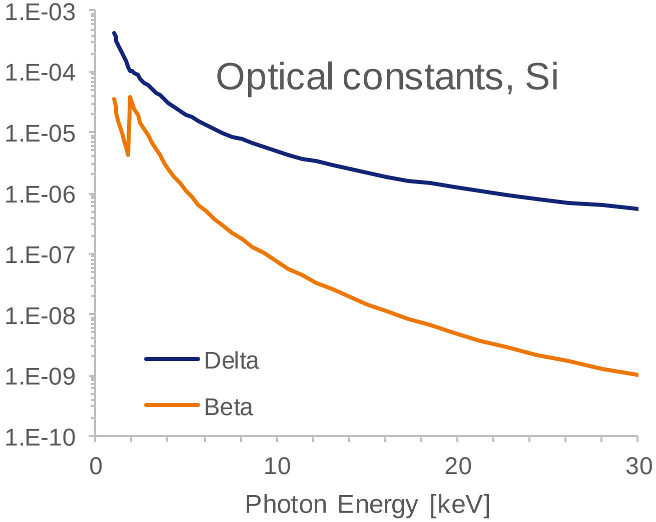
+ polarising optics, interferometers, …

REFRACTIVE INDEX FOR X-RAYS: N < 1

The refractive index for X-rays for wavelength, λ, is usually written as:

$$n = 1 - \delta - i\beta \text{ where } \delta = \left(\frac{n_e r_e f_1}{2\pi}\right)\lambda^2 \text{ and } \beta = \lambda\mu/4\pi$$

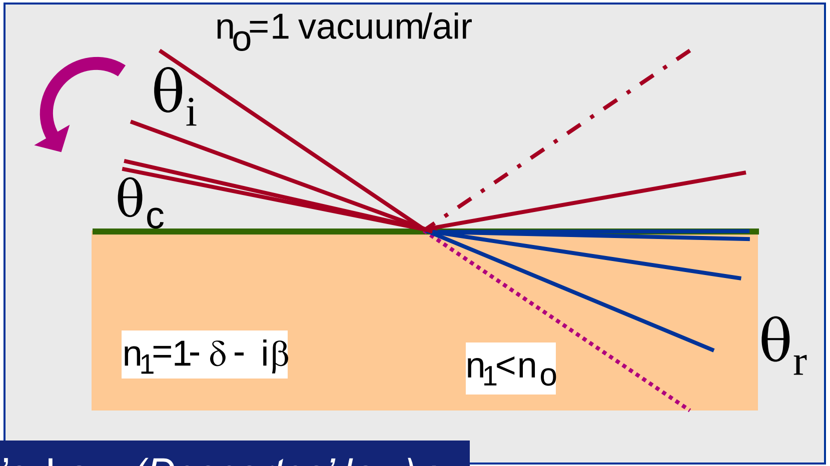
r_e is classical electron radius (2.82 x 10^{-15} m), n_e is electron density, f_1 is the real part of the atomic scattering factor ($f_0 = f_1 + if_2$), μ is linear absorption coefficient ($I = I_0 e^{-\mu t}$)



So refractive index for X-rays is less than, but close to, 1

- weak refraction c.f. visible light
- consequences for type of optics used for X-rays

TOTAL EXTERNAL REFLECTION OF X-RAYS



Snell's Law *(Descartes' law)* :

$$n_o \cos\theta_i = n_1 \cos\theta_r$$

for δ << 1 and β << δ

$$\boxed{\theta_c \approx \sqrt{2\delta} \propto \lambda\sqrt{Z}}$$

θ_c, critical angle for total external reflection.

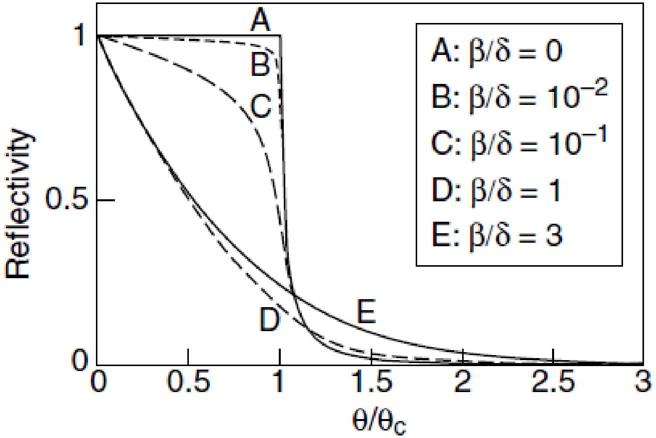
See also: http://www.coe.berkeley.edu/AST/sxreuv/

θ_c (E=10keV)	• Silicon	3 mrad
	• Nickel	6 mrad
	• Gold	9 mrad

X-ray mirrors operate in grazing incidence:

- asymmetric apertures
- long to capture X-ray source emission

Reflectivity (calculated from Fresnel equations)

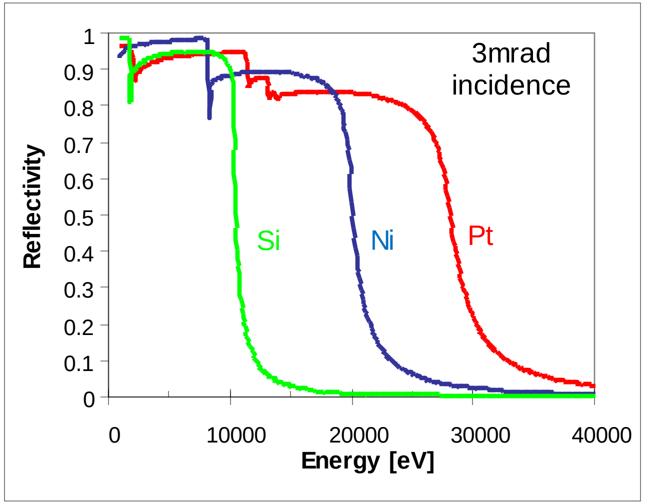


X-RAY MIRRORS: LOW PASS ENERGY FILTERS

X-rays of **fixed energy** will be totally externally reflected at angles below $\boldsymbol{\theta_c}$

Since $\theta_c \approx \sqrt{2\delta} = \lambda\sqrt{\frac{n_e r_e f_1}{\pi}}$ is inversely dependent upon the energy, we can calculate a **critical energy,** $\boldsymbol{E_c}$, below which X-rays at a **fixed angle** will be totally externally reflected.

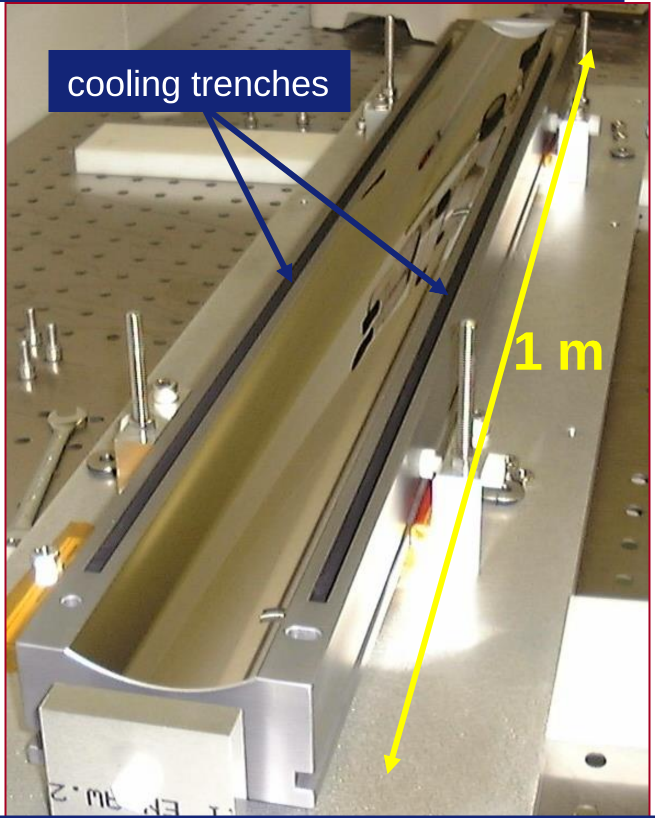
‘real’ materials



- X-rays above the critical energy will be absorbed, scattered or transmitted through the mirror
- X-ray mirrors are often illuminated with a polychromatic beam and used to filter the high-energies from the beam
- Most mirrors are manufactured from Si
- By reducing the grazing angle, E_c can be increased
- E_c can also be increased by applying a coating on the mirror with higher density

DUE TO SMALL θ_c X-RAY MIRRORS TEND TO BE LONG

Pt-coated single-crystal Si mirror

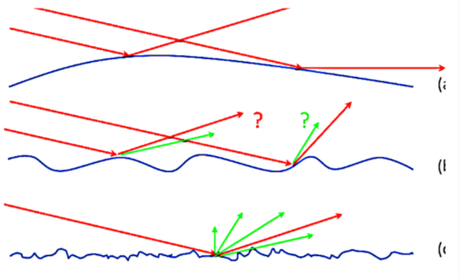


Mirrors are often curved to provide focusing
Grazing incidence → asymmetric curvature

Roughness ≤ 2Å rms
Radii of curvature:
- Sagittal: 71.60 mm
- Meridional: 25 km

Slope error (RMS)
< 1.0 µrad over 900 mm

slope errors & roughness

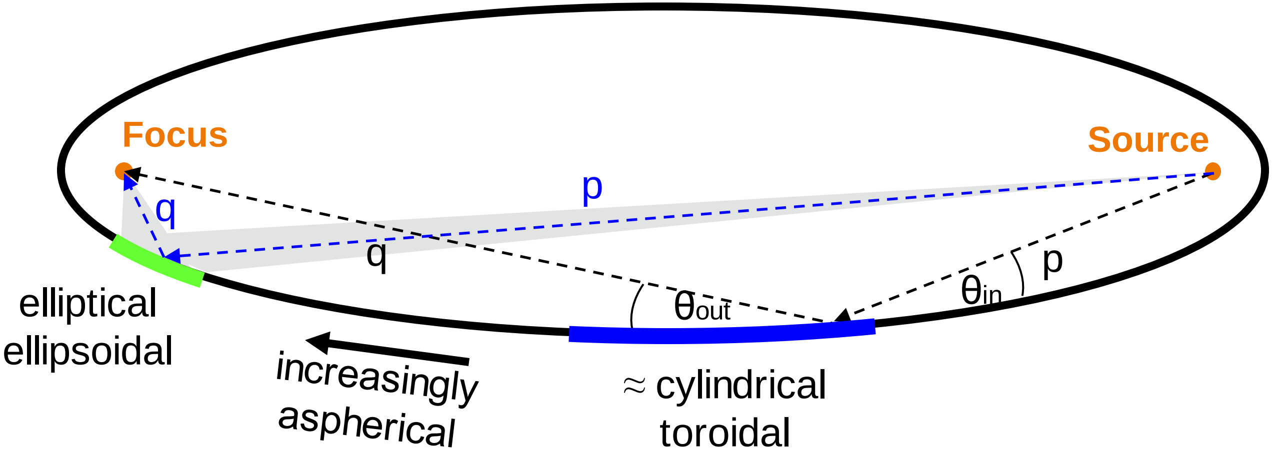


- Mirrors are prone to contamination and are usually operated under vacuum
- They can remove a considerable amount of the power (> 100’s W) of the incident beam and reduce the X-ray power on downstream optics (which may be more beam sensitive)
- Often need to be actively cooled (usually using water cooled heat exchangers)

CURVED MIRRORS FOR FOCUSING

The high reflectivities (and relatively large apertures) of mirror systems makes them attractive for focusing the X-ray beam

For point to point focusing an ellipsoidal surface is ideal – constructing the surface such that the X-ray source is at one focus of the ellipse gives a focused beam at the other. Similarly placing the source at the focus of a paraboidal mirror will produce a collimated beam after reflection



Mirror focusing is achromatic – focal length doesn't depend on energy

- If 0.3< M=q/p < 3 then generally we can use a simplified cylindrical/toroidal approximation
- For stronger demagnifications aberrations are too strong and technologically challenging aspheric surfaces are required

Local radii of curvature :

incidence angle θ

$$\left.\begin{aligned} R_m &= \frac{2}{\sin\theta_i}\left(\frac{pq}{p+q}\right) \\ R_s &= 2\sin\theta_i\left(\frac{pq}{p+q}\right) \end{aligned}\right\}$$

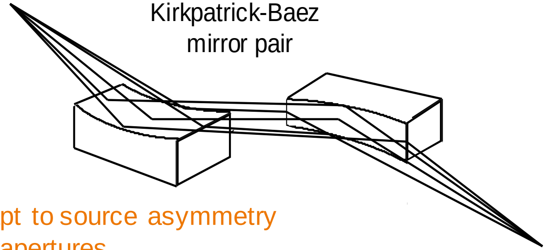
Typically: Rm ~ km (bending?)

Rs ~ cm

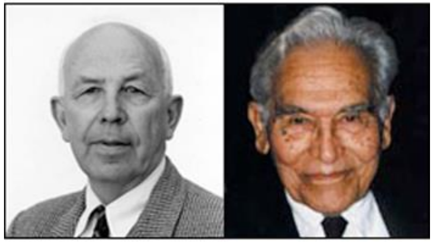
2D FOCUSING: KIRKPATRICK-BAEZ MIRROR SYSTEMS

Short radius ellipsoidal mirrors are particularly difficult to manufacture

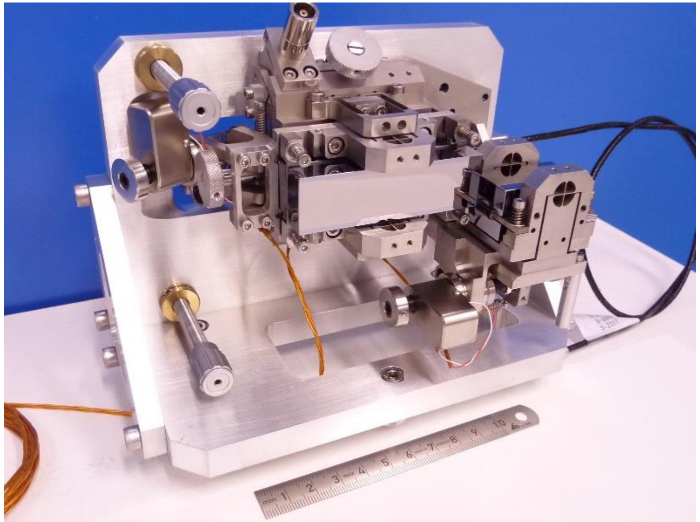
Alternative is to use 2 elliptical cylinder mirrors focusing in orthogonal planes. This was originally proposed in 1948 and is now commonly used for micro- or nano-focusing 'KB-mirrors'



- adapt to source asymmetry
  - apertures
  - demagnification
- mirrors are either dynamically bent or statically figured to create the elliptical surface



Kirkpatrick, Baez

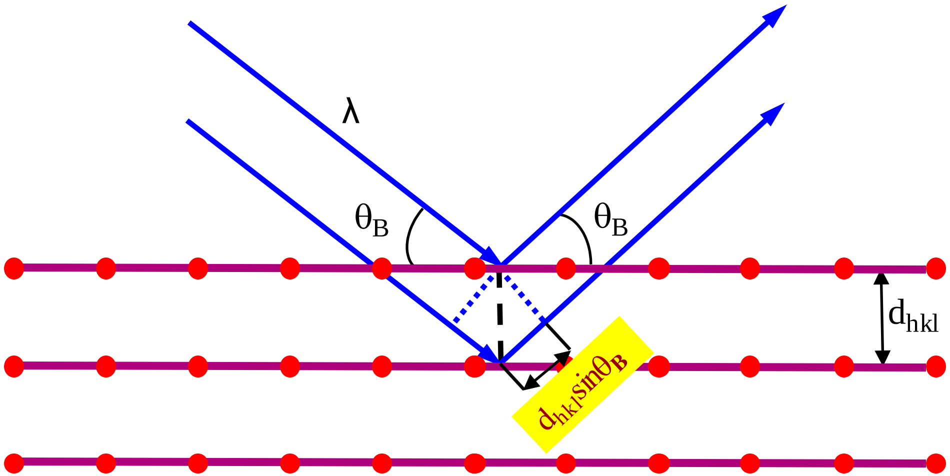


Fixed curvature KB mirror system with surface figure errors ~ 1nm
Focused beam size at 34 keV: 14 x 14 nm^2 with 6 10^{10} ph/s

X-RAY DIFFRACTION

X-ray diffraction results from elastic* scattering of X-rays from structures with long-range order. For X-ray optics we are generally concerned with highly perfect single crystals
cf neutron mosaic crystals

**elastic -> energy unchanged by scattering process*



Bragg equation: $2d_{hkl}\sin\theta_B = n\lambda$

- Incident X-rays are "reflected" at atomic planes in the crystal lattice
- Path difference of the rays: $2d_{hkl}\sin\theta_B$
- Constructive interference if the path difference amounts to λ (n λ)

h k l are usually used, (e.g. 1 1 1, 3 3 3, 4 4 4), these are not Miller indices, but Laue indices, or "general Miller indices".

CRYSTAL MONOCHROMATORS

A crystal monochromator slices out a narrow energy band from incident beam. Energy, E, determined by incidence angle, θ_B, of X-ray beam onto crystal planes according to Bragg equation:

$$E = \frac{hc}{\lambda} = \frac{hc}{2d_{hkl} \sin \theta_B}$$

c = light velocity
h = Plancks constant

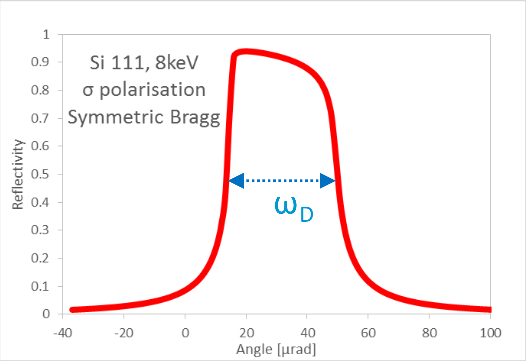
Energy width of beam depends upon type of crystal and reflecting planes used (described by angular Darwin width ω_D) & divergence of incident beam, ψ_0

$$\frac{\Delta E}{E} = \frac{\Delta\lambda}{\lambda} = \sqrt{\omega_D{}^2 + \psi_0{}^2}\, \cot\theta_B$$

If $\psi_0 = 0$, intrinsic (best) energy resolution

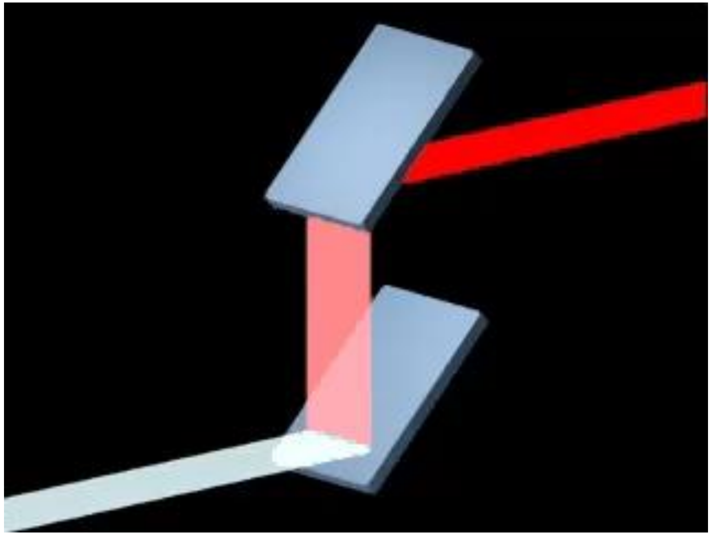
e.g. **Si 111 Bragg reflexion**,
d_{hkl}= 3.1355Å
ω_D = 34 µrad (@ 8keV):

θ_B = 14°
with a parallel incident beam:
$\Delta E/E = 1.4.10^{-4}$, ΔE=1.1eV

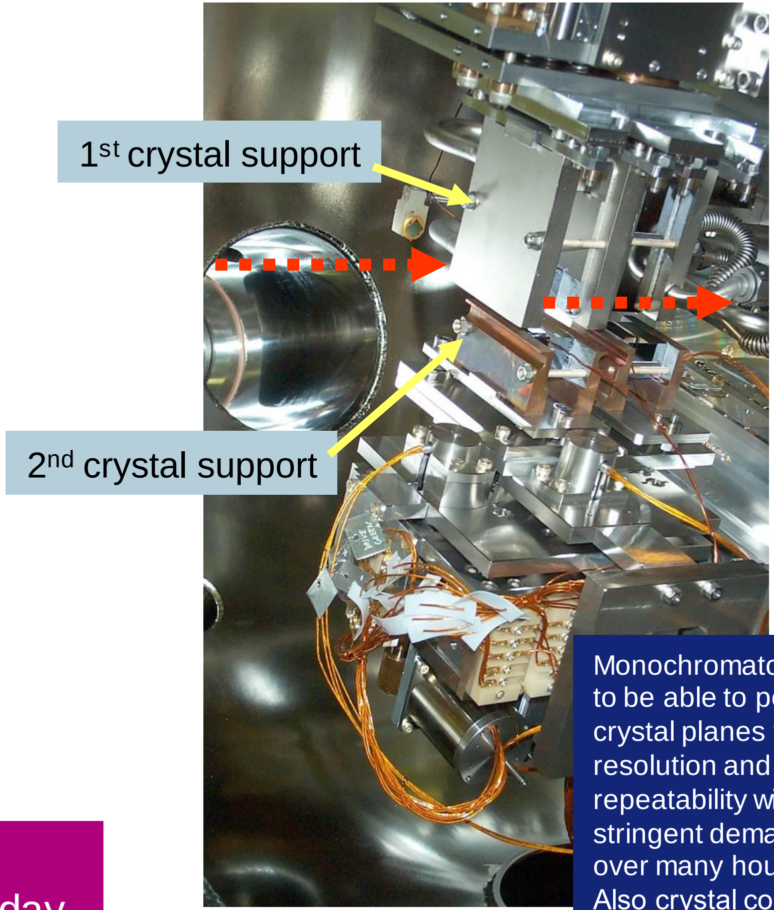


Refl.	θ_B (12keV) [°]	ω_D [µrad]	ΔE [eV]
111	9.48	22.4	1.6
220	15.61	15.9	0.7
311	18.39	8.9	0.3
333	29.62	4.8	0.1

DOUBLE CRYSTAL MONOCHROMATORS



First crystal monochromatizes beam – the second ensures incident and exit beams have the same direction



Monochromators typically need to be able to position the crystal planes with µrad resolution and similar repeatability with particularly stringent demands on stability over many hours.
Also crystal cooling required…

Increasingly mechatronics used for monochromators – L. Ducotté's talk Tuesday

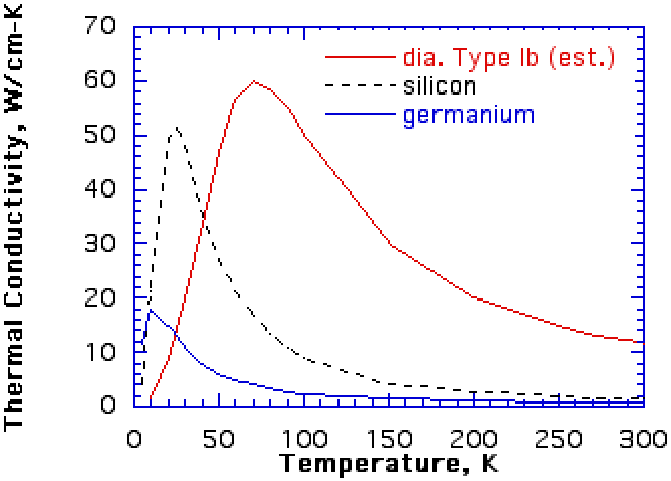
MONOCHROMATOR COOLING

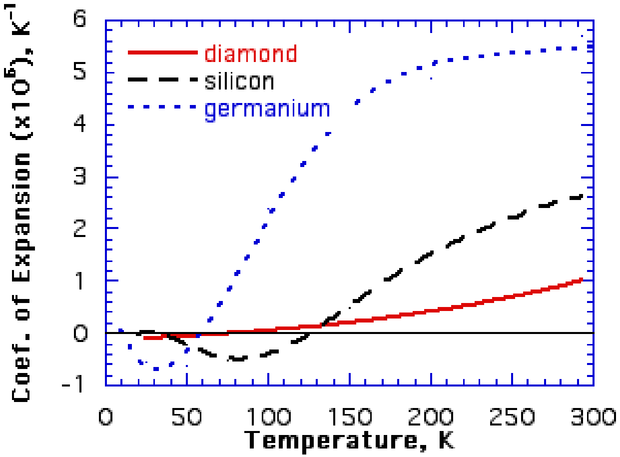
The first monochromator crystal absorbs considerable power

Thermal gradients ΔT, and coefficient of thermal expansion, α, contribute to crystal distortions:

$$\alpha\Delta T = \Delta d/d = \cot(\theta)\,\Delta\theta < \cot(\theta)\,\omega_D$$

We therefore need to look for materials that have a very low coefficient of thermal expansion, α, and/or have a very high thermal conductivity, k, so that the material cannot develop large ΔT's.





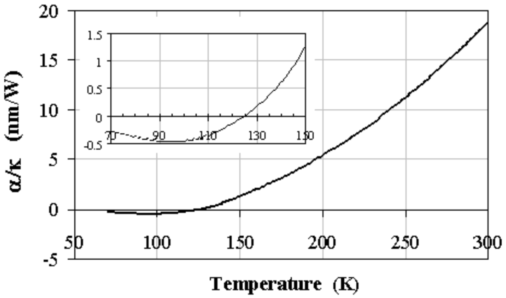
courtesy D. Mills, APS

FIGURE OF MERIT (FOM) FOR CHOICE OF CRYSTAL FOR HIGH HEAT LOADS

These properties incite us to use cryogenically cooled silicon or room temperature diamond as high heat load monochromators

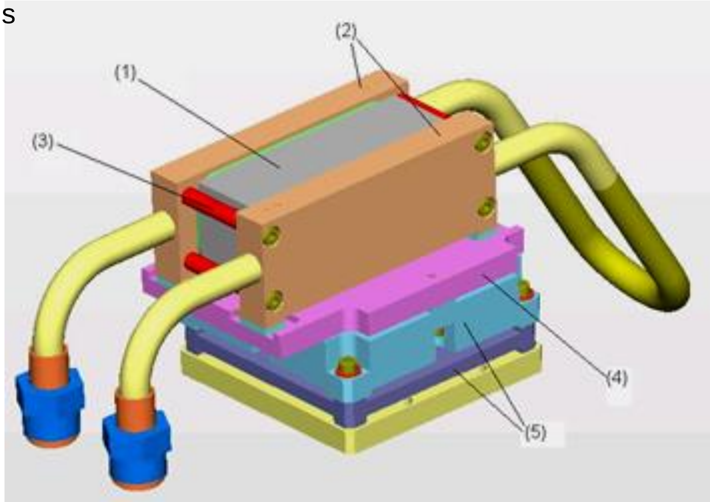
FOM for various crystals –higher is better

Material	k –thermal conductivity [W/cm.K]	α –coeff thermal expansion $[K^{-1}]$	k/α FOM
Si (300K)	1.2	2.3×10^{-6}	0.5
Si (78 K)	14	-0.5×10^{-6}	28
Diamond (300K)	20	0.8×10^{-6}	25



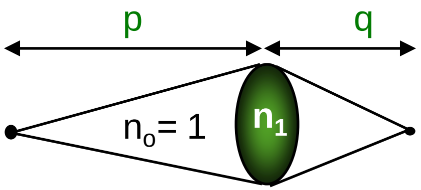
Ratio of thermal expansion and thermal conductivity of Si α/κ vs temperature

Example of ESRF first crystal assembly:
(1) silicon crystal; (2) copper cooling blocks with internal fins; (3) invar clamping rods; (4) invar base plate; (5) ceramic insulating plates



- Crystal is side cooled with cooling blocks clamped with pressures between 5-10bar
- Deformation of crystal planes due to clamping <1μrad
- Most ESRF beamlines using LN2 cooled monochromators

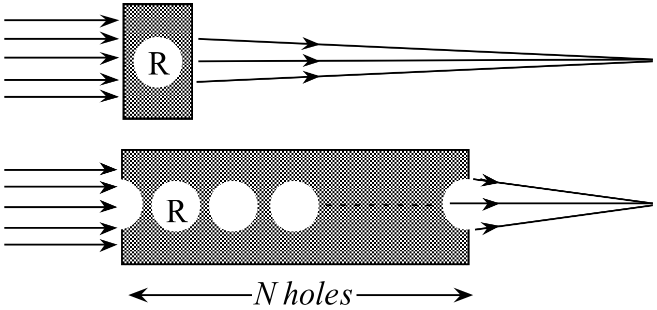
COMPOUND REFRACTIVE LENS



Gaussian lens equation: $\frac{1}{f} = \frac{2(n_1 - 1)}{R}$

Thin lens equation : $\frac{1}{f} = \frac{1}{p} + \frac{1}{q}$

R = radius of curvature of lens apex



$$\frac{1}{f} = \frac{2\delta}{R}$$

$$\boxed{\frac{1}{f} = N\frac{2\delta}{R}}$$

X-rays : $n = 1 - \delta - i\beta$

$n_1 < 1$: concave lens

Typically Be or Al lenses – e.g.

Aluminium @ 10keV $\delta = 5.5\ 10^{-6}$

1 hole 100 µm radius : f = 9 m

15 holes 100 µm radius: f = 60 cm

A. Snigirev et al. Nature, 384 (1996)

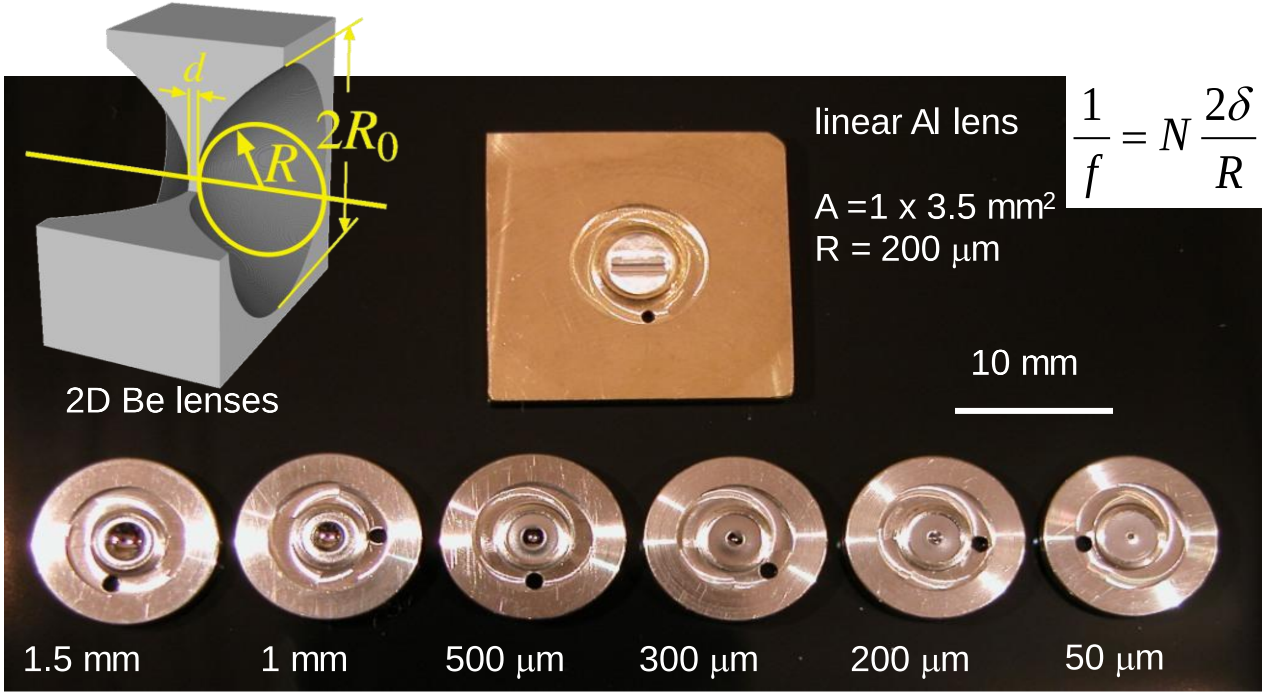
Advantages
- simplicity and relatively low cost
- low sensitivity to heat load
- on-axis focusing

Disadvantages
- efficiency limited by absorption
- small aperture (limited resolution)
- strong chromatic aberrations $f \propto E^2$

PARABOLOIDAL & PARABOLIC CYLINDER X-RAY LENSES

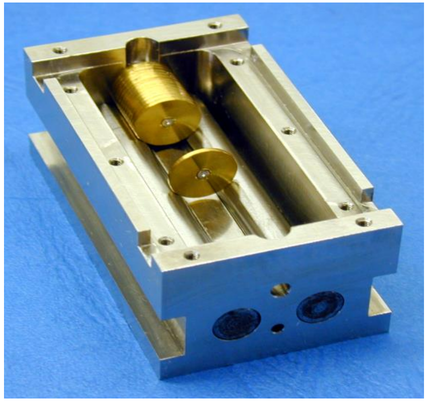
- Parabolic/oidal profile ⇒ no spherical aberration
- Produced by embossing
- Be ~2-40 keV ⇒ absorption↓
- Al ~40-80 keV
- Ni ~80-150 keV

Typical parameters :
R = 50 to 1500 µm
$2R_0$ = 0.45 to 2.5 mm
d < 30µm



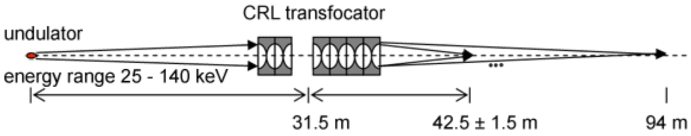
$$\frac{1}{f} = N\frac{2\delta}{R}$$

Stack lenses according to required focal length

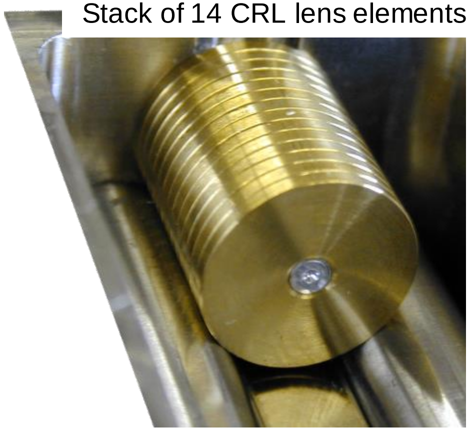


REFRACTIVE LENS ‘TRANSFOCATORS’

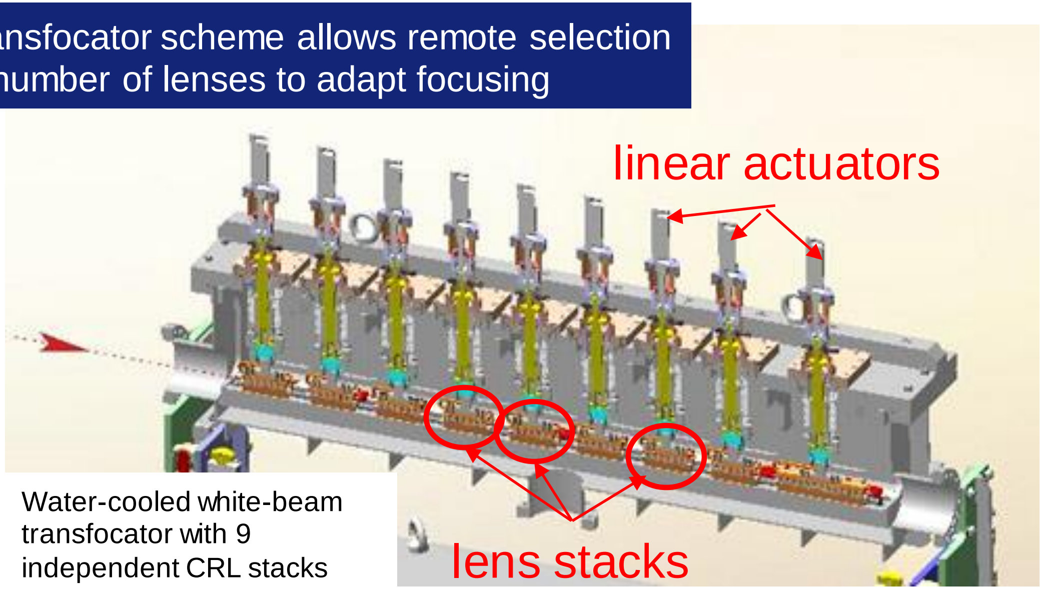
Need to adapt number of lenses according to required focusing energy and focal length



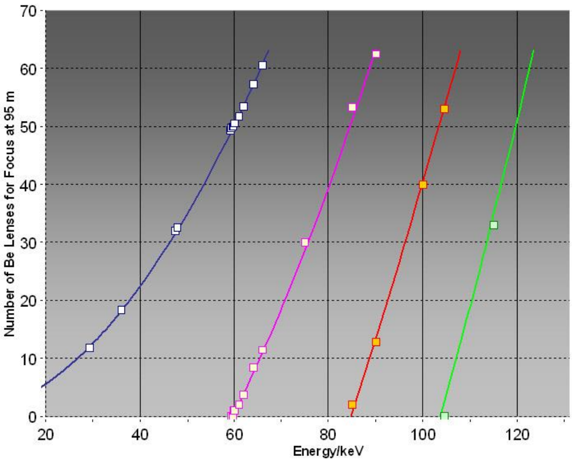
Stack of 14 CRL lens elements



Transfocator scheme allows remote selection of number of lenses to adapt focusing



Water-cooled white-beam transfocator with 9 independent CRL stacks



Vaughan et al. J.Synch. Rad. **18** 125-133 (2011)

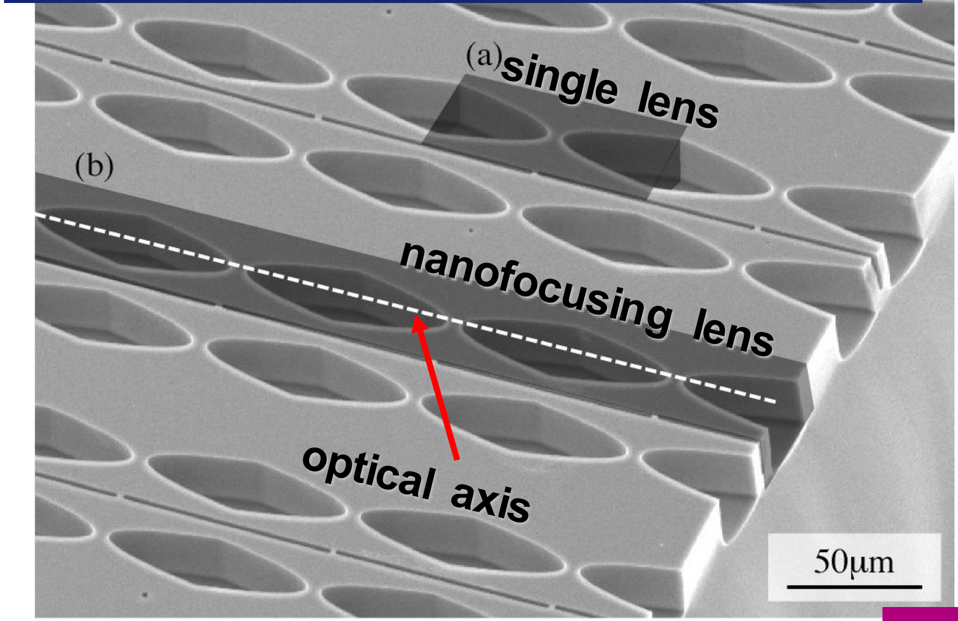
NANOFOCUSING REFRACTIVE LENSES

$$\frac{1}{f} = N\frac{2\delta}{R}$$

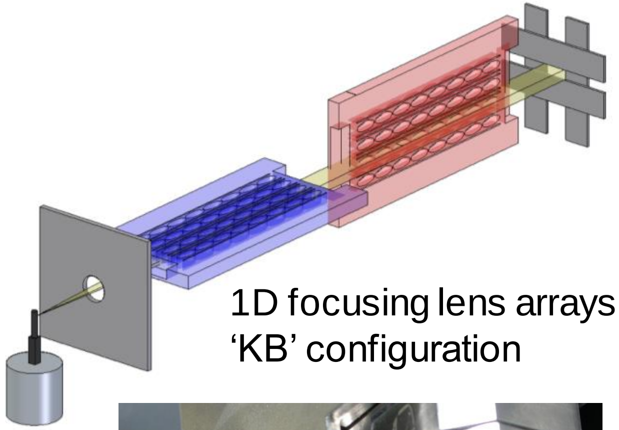
Very short focal lengths require large N or small R

extreme curvature: *R* = 1µm - 3µm *N* = 50 - 100

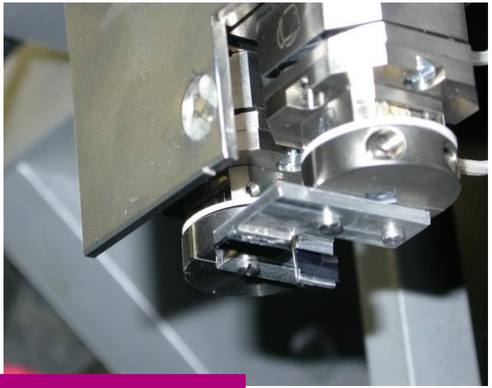
Si lens arrays made by MEMS technologies



C. Schroer *et al*, *Applied Physics Letters*, **82**(9), 2003

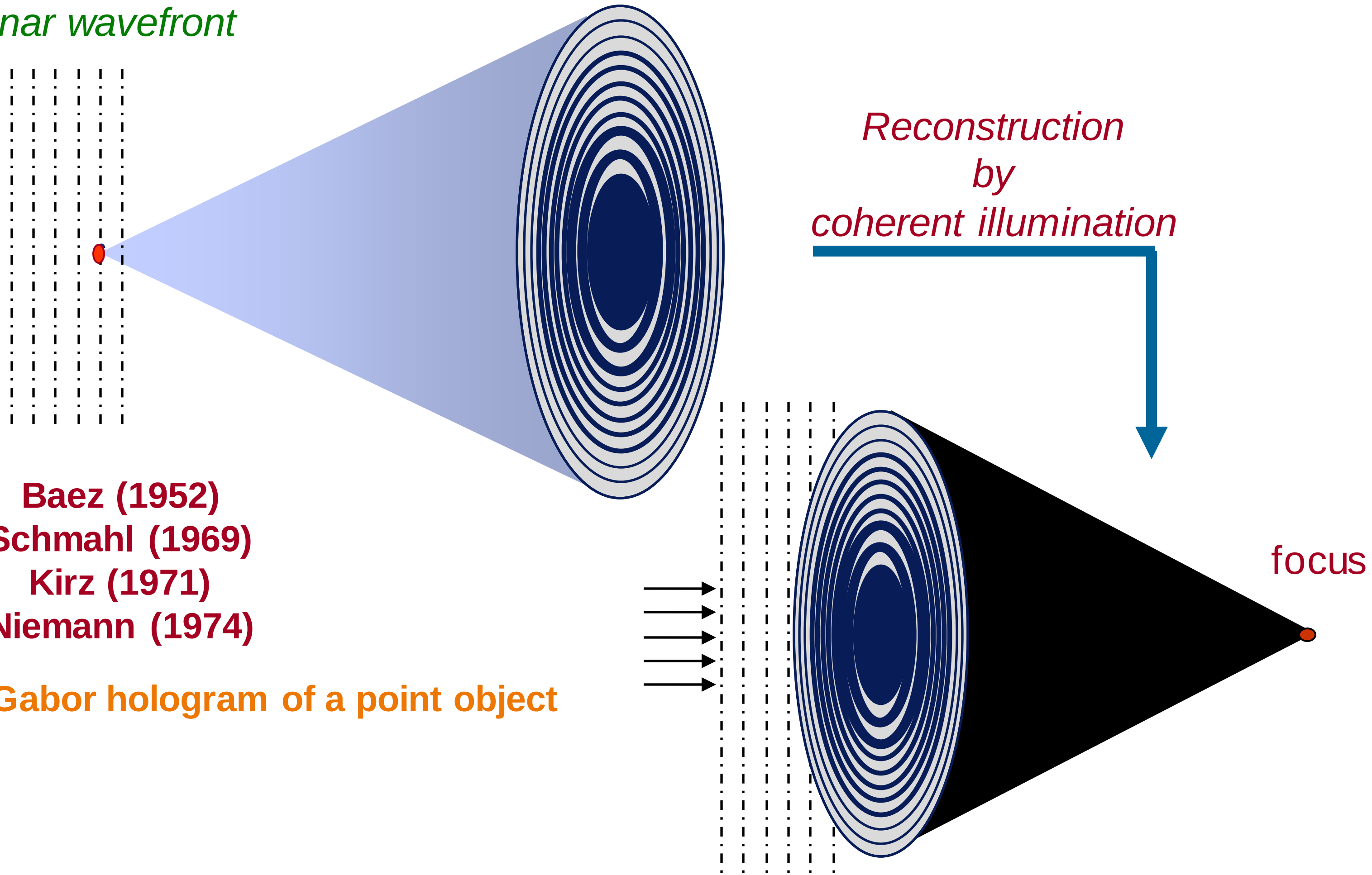


1D focusing lens arrays:
'KB' configuration



Minimum focus size reported with refractive lenses 47nm

DIFFRACTIVE FOCUSING OPTICS: FRESNEL ZONE PLATES



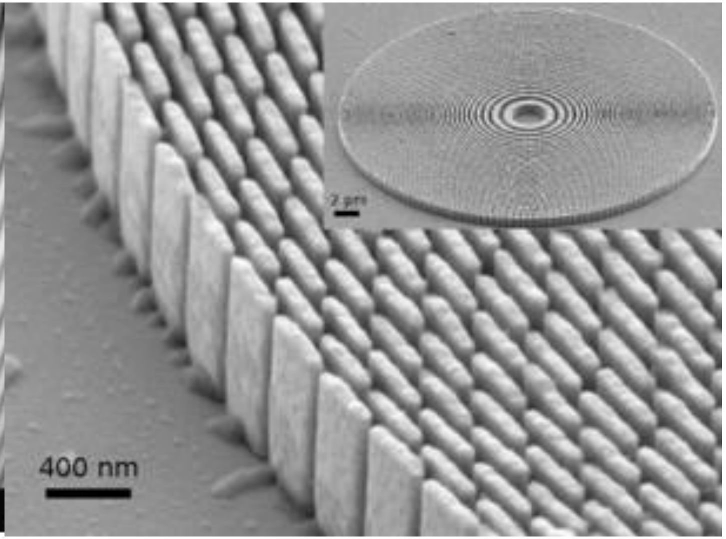
Baez (1952)
Schmahl (1969)
Kirz (1971)
Niemann (1974)

Gabor hologram of a point object

ZONE PLATES

Zone plates are circular diffraction gratings composed of alternating concentric zones

- grating line spacing decreases with the zone radius
- outermost zone has a line width Δr_N (which has to be precisely placed)
- usually manufactured (on thin transmissive membranes) using e-beam lithography
- diffracts/focuses into many orders, m – usually only the first order is used
- filtering the other orders and undiffracted light complicates the implementation



Resolution: $$\delta_m = 1.22 \frac{\Delta r_N}{m}$$

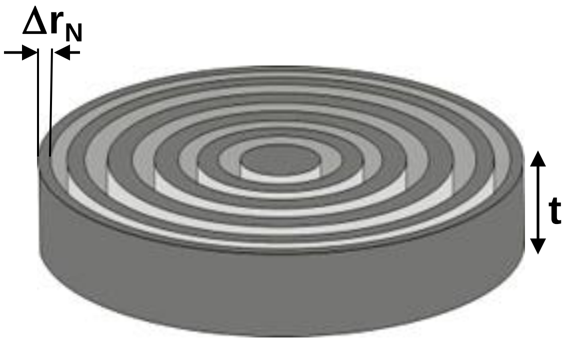
(smallest Δr_N ~ 10 nm)

Focal length: $$f_m = \frac{D\,\Delta r_N}{m\,\lambda}$$

Consequently zone plates are chromatic with $f \propto E$

DIFFICULT TO FABRICATE EFFICIENT ZONE PLATES!

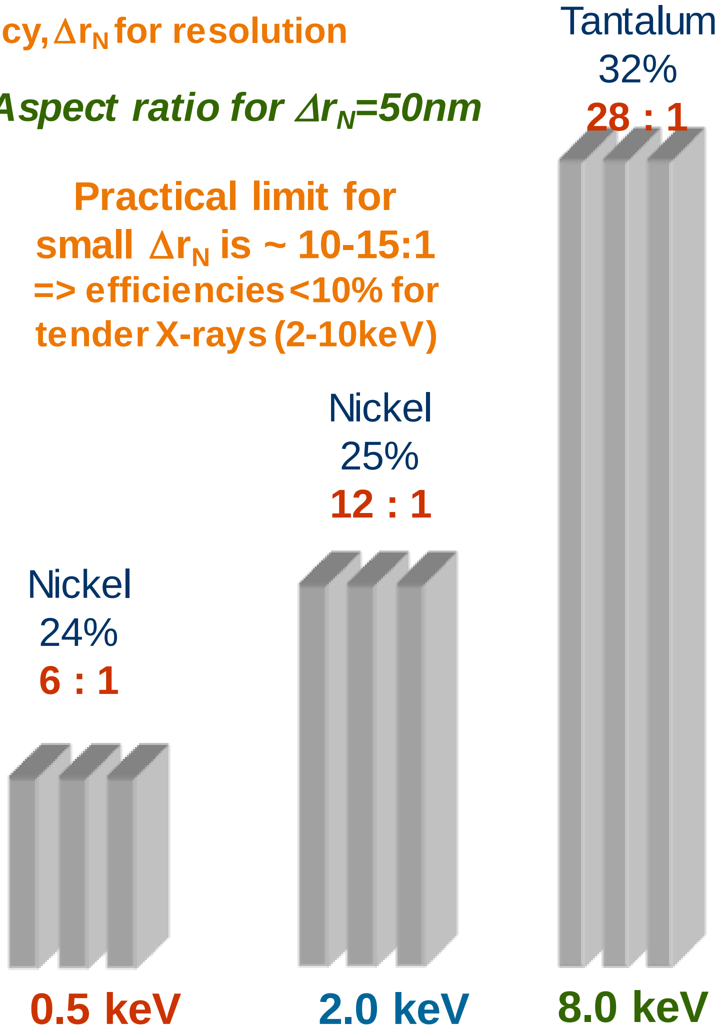
structure height, t, critical for efficiency, Δr_N for resolution



Aspect ratio for Δr_N=50nm

Practical limit for small Δr_N is ~ 10-15:1 => efficiencies <10% for tender X-rays (2-10keV)

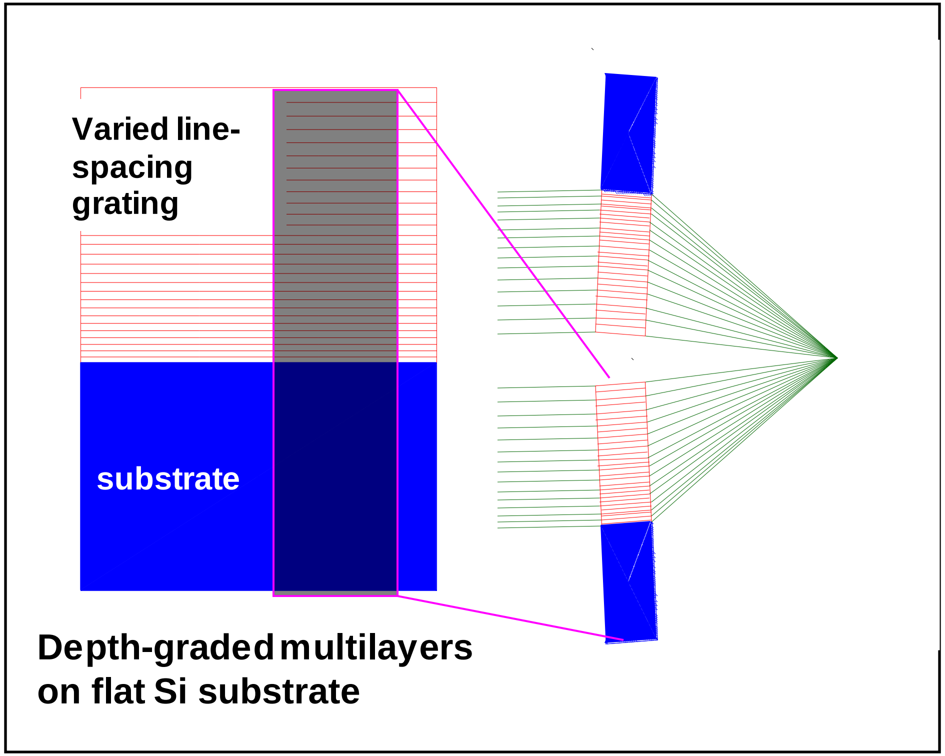
Material	t (μm)	ε (%)
E=0.5keV		
Ge	0.28	16
Ni	0.25	24
E=2.0keV		
Ni	0.60	25
Au	0.45	24
E=8.0keV		
Ta	1.70	32
W	1.50	33



MULTILAYER LAUE LENSES

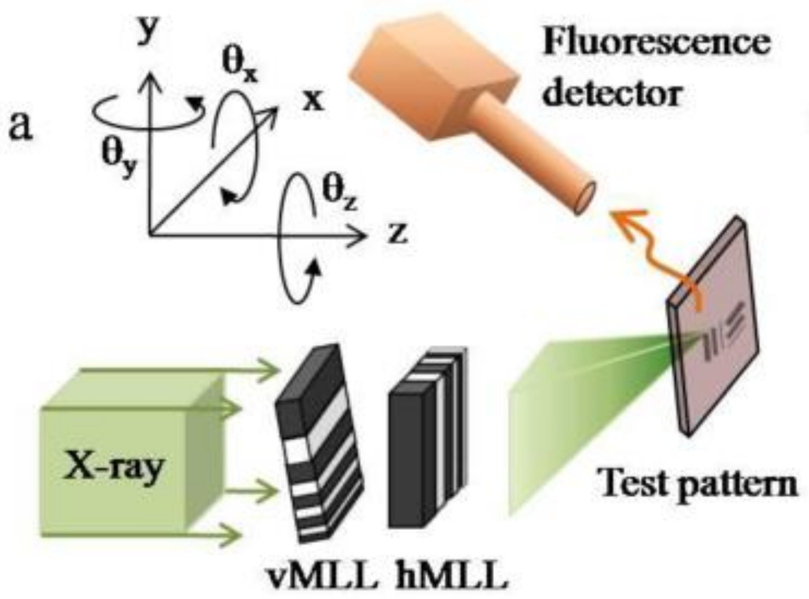
Initial development at CNM, APS group, Argonne Nat. Lab. USA,
H. C. Kang, J. Maser, G. B. Stephenson, C. Liu, R. Conley, A. T. Macrander, and S. Vogt, Phys. Rev. Lett. **96**, (2006).

- Deposit varied line-spacing grating on flat substrate (thinnest structures first)
- Section to 5-20 μm thickness (high aspect ratio structure) but small aperture (~ 40 μm)
- One dimensionally focusing volume zone plate well adapted to higher photon energies
- Assemble two (four) in orthogonal orientations for 2D focusing (MLL)



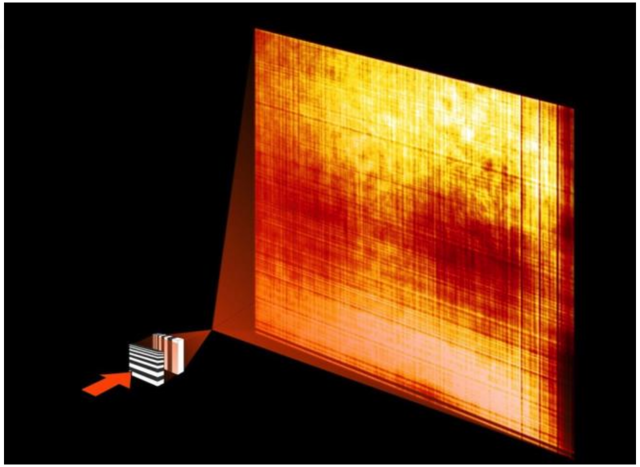
< 10nm focusing @ 22keV

MULTILAYER LAUE LENSES



H. Yan et al., Opt. Express
19, 15069-15076 (2011)

sub-3 nm 2D focusing at 17.5 keV
Dresselhaus et al. *Opt. Express* 32, (2024): 16004–15



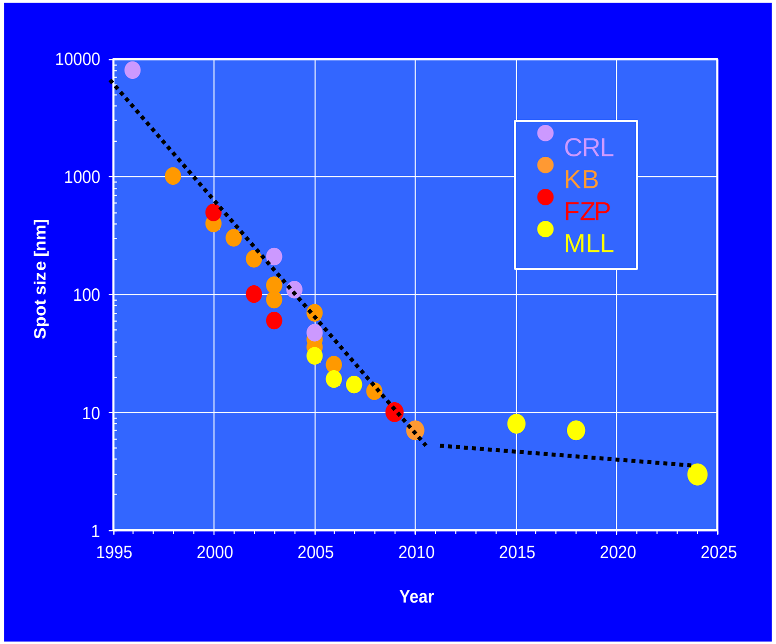
S. Bajt et al., Light Science & Applications (2017); doi: 10.138/lsa2017.162

PROGRESS IN HARD X-RAY FOCUSING

Optics exist for focusing hard X-rays to sub-10nm dimensions

Routine application of sub-micron beams still complicated

Also many engineering issues in implementing stable, reliable X-ray nanofocusing systems



Historical evolution of the measured spot size for different hard x-ray focusing elements *(courtesy C. Morawe)*

- A. Morgan et al. Scientific Reports, 5, 9892 (2015)
- H. Mimura *et al. Nature Physics*, **6**, 122-125 (2010).
- J. Vila-Comamala et al., Ultramicroscopy,**109**,1360–1364 (2009)
- J. Dresselhaus et al. *Opt. Express* 32, (2024): 16004–15
- H. Kang *et al., Physical Review Letters*, **96**:127401 (2006)
- C. Schroer *et al., Physical Review Letters*, **94**:054802 (2005)

Best focus Experiments

Ultimate resolution Theory