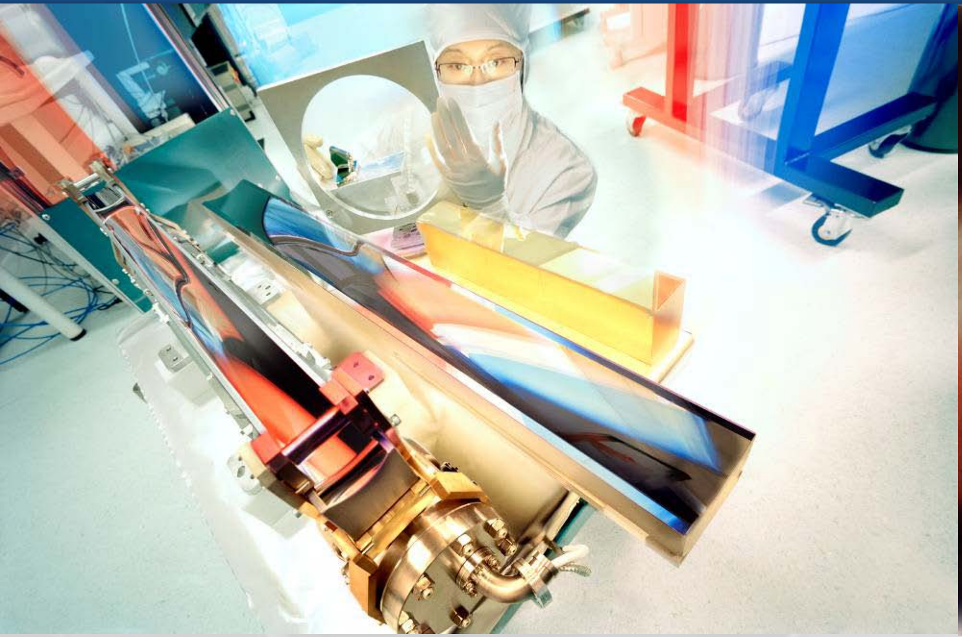
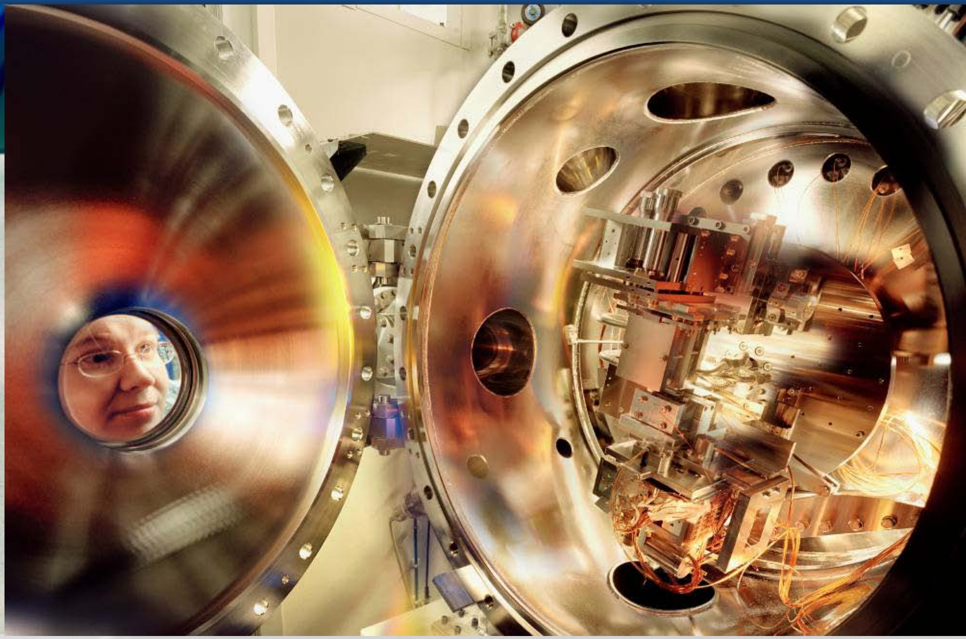




ESI2011: **2nd EIROforum School on Instrumentation**

X-ray Optics for Synchrotron Radiation Beamlines

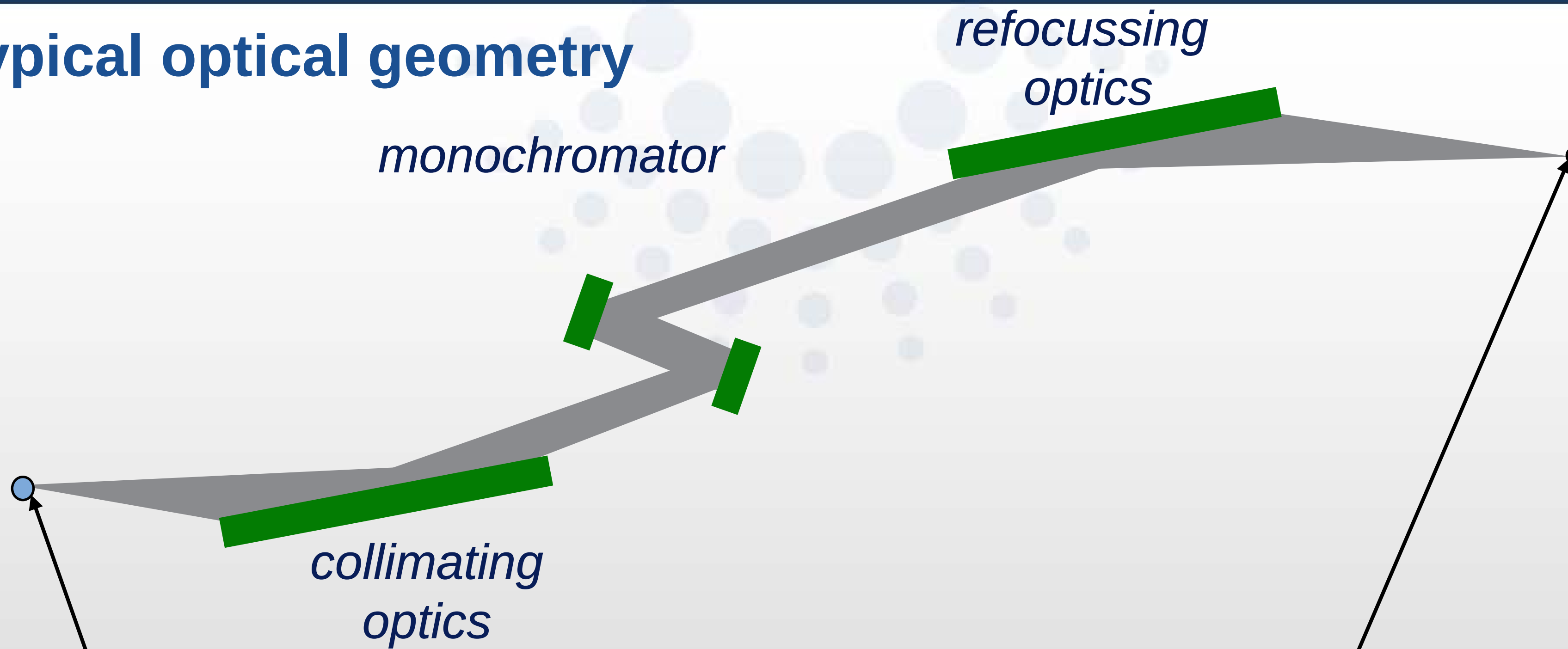
Ray BARRETT
X-ray Optics Group
European Synchrotron Radiation Facility
e-mail : barrett@esrf.fr

- ➢ **Introduction**
- ➢ General X-ray optics
- ➢ High heat-load optics
- ➢ X-ray micro-/nano-focusing
  - ➢ Reflectors
  - ➢ Zone plates
  - ➢ Refractive lenses
- ➢ Summary

Scope:

- •Non-exhaustive overview
- •Some general X-ray optics – foundation for X-ray focusing
- •Primarily ‘hard’ X-ray optics (i.e. photon energies >2keV)
- •Slight bias for ESRF applications
- •References for further reading

Typical optical geometry



Source
- spectrum (ΔE/E)
- emittance (size x divergence)
- degree of spatial coherence
- brilliance (ph/s/mm²/mrad²/0.1%bw)
- polarisation (linear, circular, elliptic)

OPTICS
?

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

Tasks:

- *transform beam to obtain the best match to experimental requirements*

- **Act upon:**
  - shape
  - wavelength/energy
  - divergence
  - polarisation

- slits, pinholes
- filters, windows
- mirrors
- beam splitters (crystals)
- monochromators (crystals, multilayers)
- phase plates (crystals, multilayers)
- lenses, zone plates
- combined elements (ML gratings, Bragg-Fresnel-lenses)

- Introduction
- **General X-ray optics**
- High heat-load optics
- X-ray micro-/nano-focusing
  - Reflectors
  - Zone plates
  - Refractive lenses
- Summary

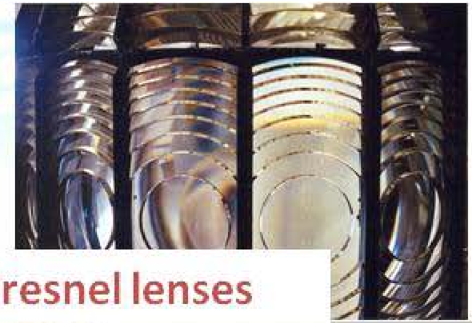
Anna Bertha Röntgen
Taken 1895

Wilhelm Conrad Röntgen
1845-1923

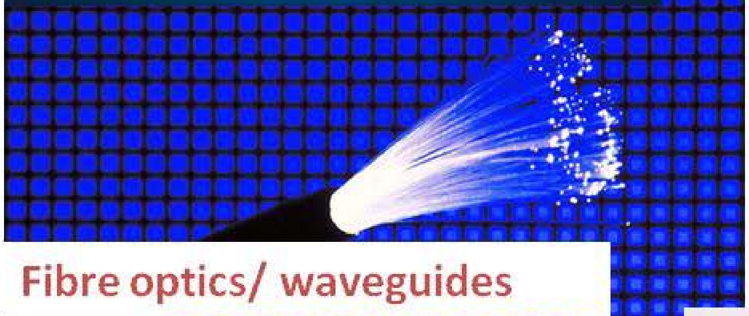
Visible Light Optics

Refractive lenses

Polarising Optics



Fresnel lenses



Diffractive optics

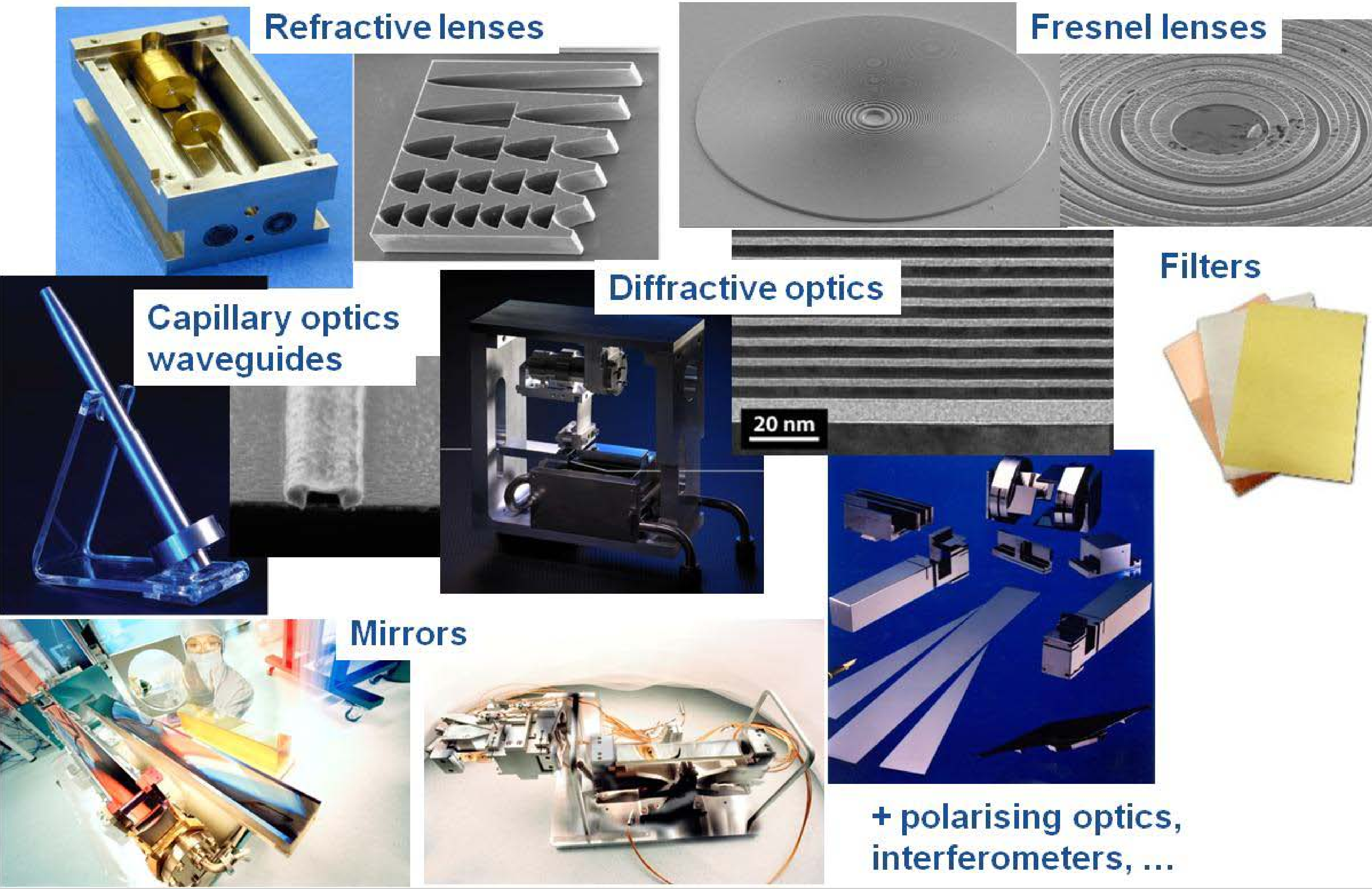
Fibre optics/ waveguides

Filters

Mirrors

+ interferometers, …

X-ray Optics


Refractive lenses
Fresnel lenses
Filters
Diffractive optics
20 nm
Capillary optics
waveguides
Mirrors
+ polarising optics,
interferometers, ...

X-ray optics: many approaches

"... The refractive index.... cannot be more than 1.05 at most....
....X-rays cannot be concentrated by lenses..."

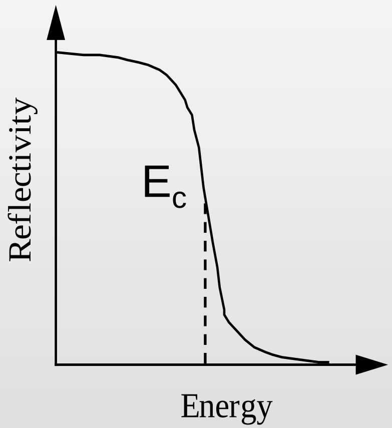
W.C. Röntgen
Über eine neue art von Strahlen.
Phys.-Med. Ges., Würzburg, **137**, p. 41, (1895)
*English translation in Nature **53**, p. 274*

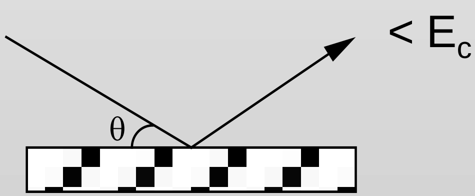
$n=1-\delta+i\beta$ with δ, $\beta <<< 1$

δ (phase-shift), β (absorption), materials (and energy) dependent optical constants

- Very weak refraction
- Quite high absorption

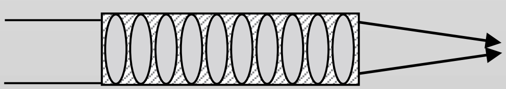
REFLECTION





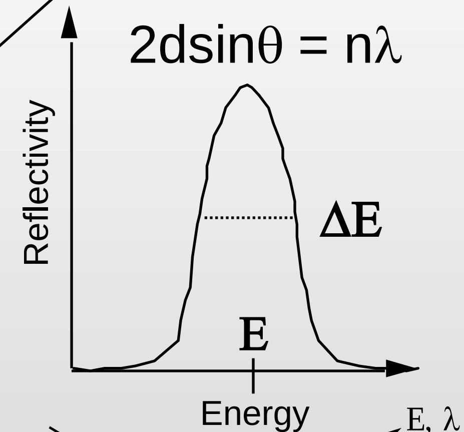
- X-ray mirrors
- Capillaries
- Waveguides

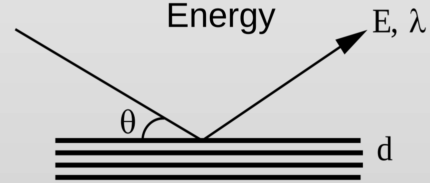
REFRACTION



- Refractive lenses

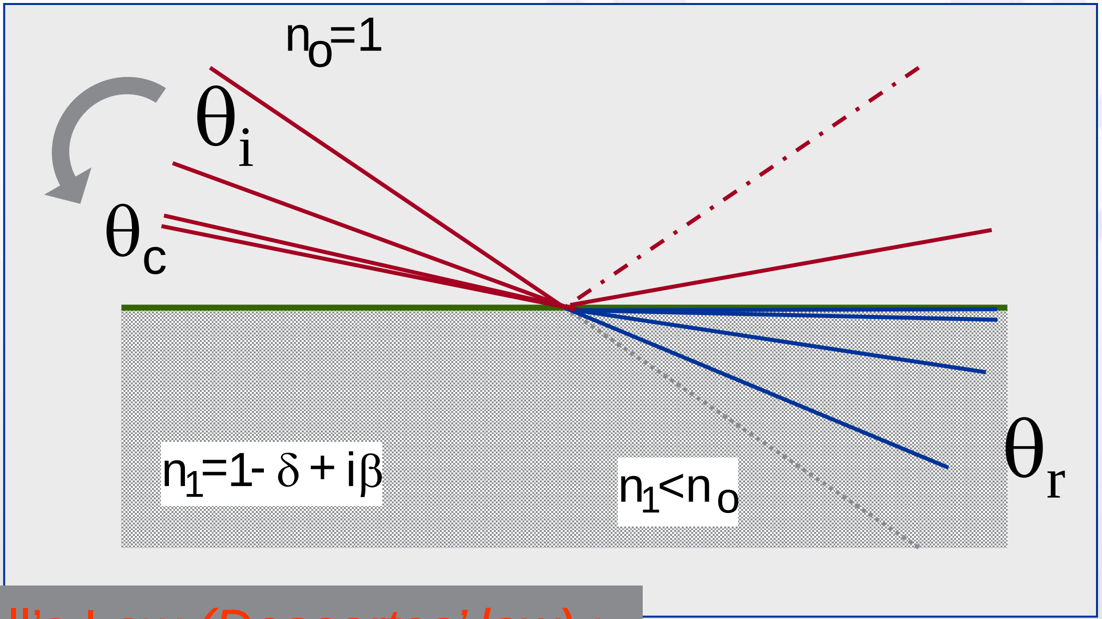
DIFFRACTION



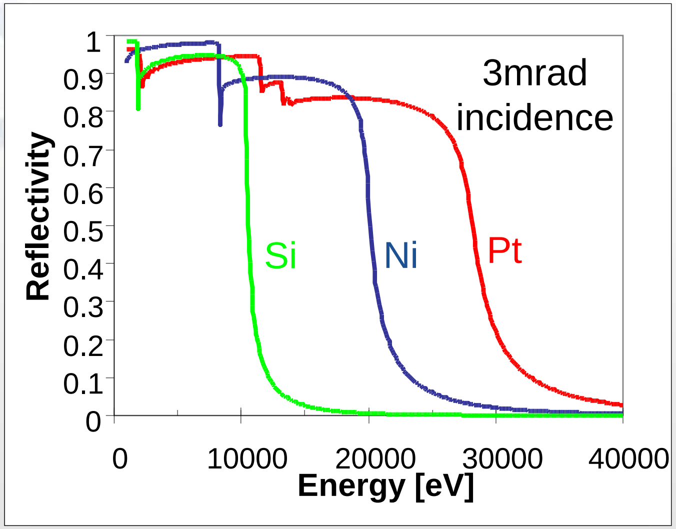


- Crystals & multilayers
- X-ray gratings
- Fresnel zone plates
- Bragg-Fresnel lens

Total external reflection



'real' materials



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

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

for $\delta << 1$ and $\beta << \delta$

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

The critical angle for total external reflection.

$$\boxed{\theta_{c_{[mrad]}} E_{c_{[keV]}} = 19.83\sqrt{\rho_{[g/cm^3]}}}$$

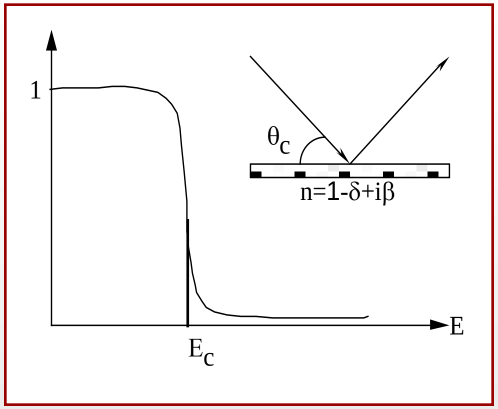
E=10keV

- Gold 9 mrad
- Nickel 6 mrad
- Silicon 3 mrad

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

X-ray mirrors : main functions

- ***Deflection***
  - beam steering (different experiments, Bremsstrahlung)



- ***Power filter***
  - lower incident power on sensitive optical components
- ***Spectral shaper***
  - energy low-pass filter (harmonic rejection)
  - mirror+filter = spectral window
- ***Focusing***
  - wiggler & bending magnet : spherical, cylindrical, and toroidal mirrors
  - microscopy & microprobe : source demagnification (ellipsoidal mirror, KB ……)
- ***Collimation***
  - parabolic mirror : matching the monochromator angular acceptance with the beam divergence

J. Susini, Optical Engineering, 34(2), (1995)

- Typical Requirements
  - *micro-roughness* < 3Å rms and *slope error* ~1 µrad rms for blur -10% source size
    - Ultra-precise shaping, figuring and super-polishing
    - Very accurate and stable mechanical mounting, bending mechanisms, UHV environment
    - Efficient cooling scheme

Technically limiting parameters

- gravity sag $\Delta_g \propto \frac{5g}{32} \frac{L^3}{t^2} \frac{\rho}{E}$
- vibration $f_o \propto \sqrt{\frac{E}{\rho}}$
- thermal deformation $\Delta_t \propto \frac{\alpha}{\kappa} P_s$
- bending $F_b \propto E \frac{\alpha}{\kappa} P_t\, w\, t^3$

$\frac{\rho}{E} \searrow \qquad E\frac{\alpha}{\kappa} \searrow \qquad \frac{\alpha}{\kappa} \searrow$

+

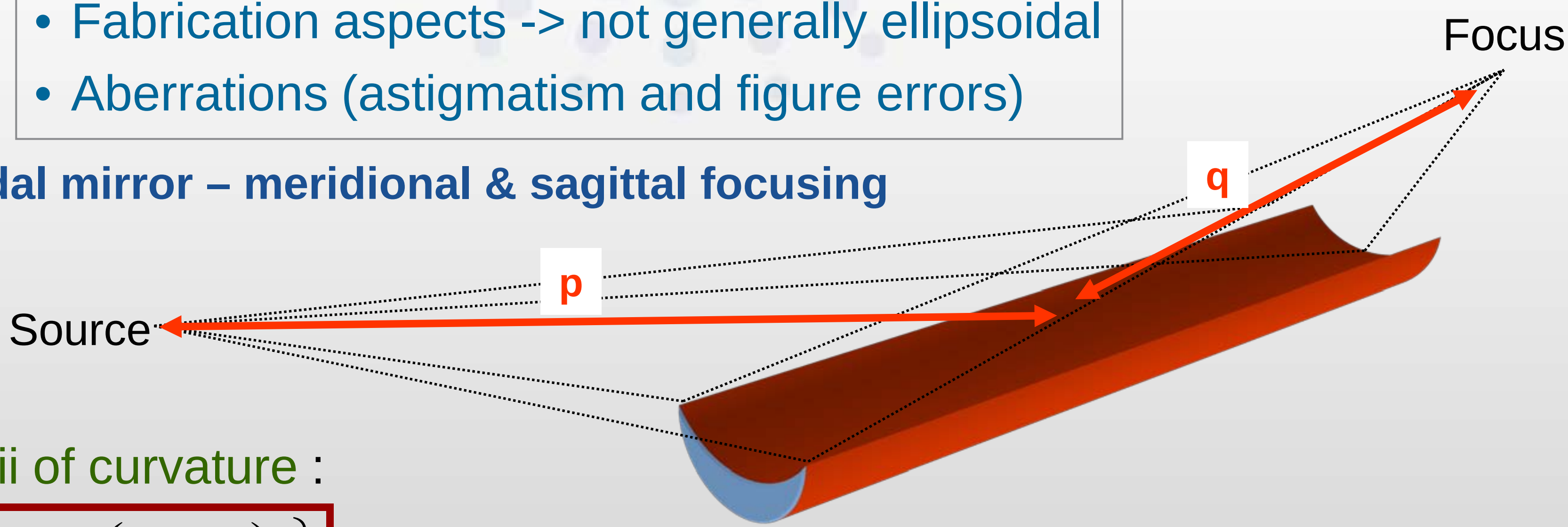
"Polishability"

Most SR Mirrors are manufactured from Si, SiC or Glidcop

Curved surfaces: Focusing

- Often long mirrors (> 1m)
- Off-axis geometry
- Point-to-point focusing => ellipsoidal figure
- Fabrication aspects -> not generally ellipsoidal
- Aberrations (astigmatism and figure errors)

e.g. toroidal mirror – meridional & sagittal focusing



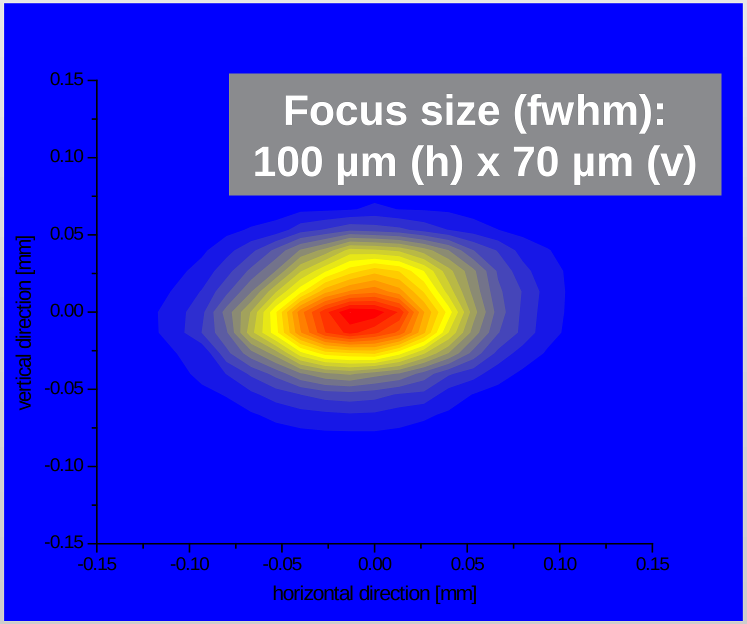
Radii of curvature :

$$\boxed{\begin{aligned} R_m &= \frac{2}{\sin\theta_i}\left(\frac{pq}{p+q}\right) \\ R_s &= 2\sin\theta_i\left(\frac{pq}{p+q}\right) \end{aligned}} \rightarrow \boxed{R_s \approx R_m \theta^2} \xrightarrow{\theta = 10\text{mrad}} \boxed{\begin{aligned} R_s &\sim mm \\ R_m &\sim km \end{aligned}}$$

Toroidal Mirror (ID09 ESRF)

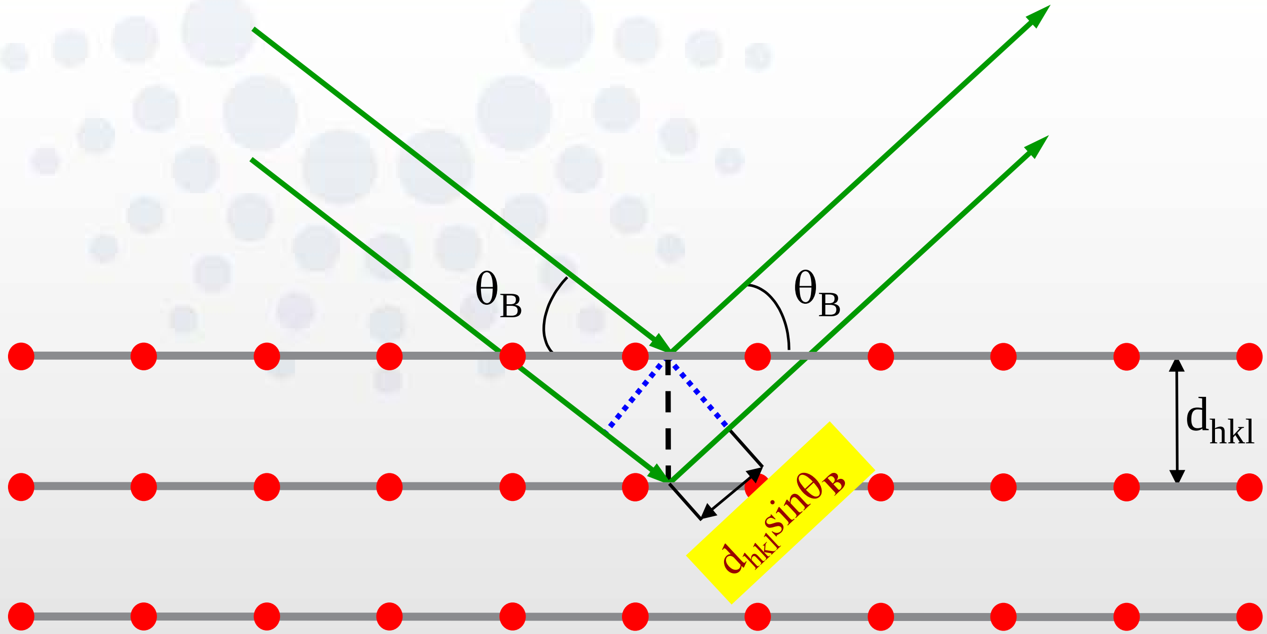
Material-coating: Silicon-Pt
Supplier: SESO (France)
Roughness ≤ 2Å rms
Radii of curvature:
- Sagittal: 71.60 mm
- Meridional: 25 km

Slope error (RMS)
- 0.7 µrad over 450 mm
- 1.0 µrad over 900 mm



X-ray Diffraction

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



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 ($n\lambda$?)

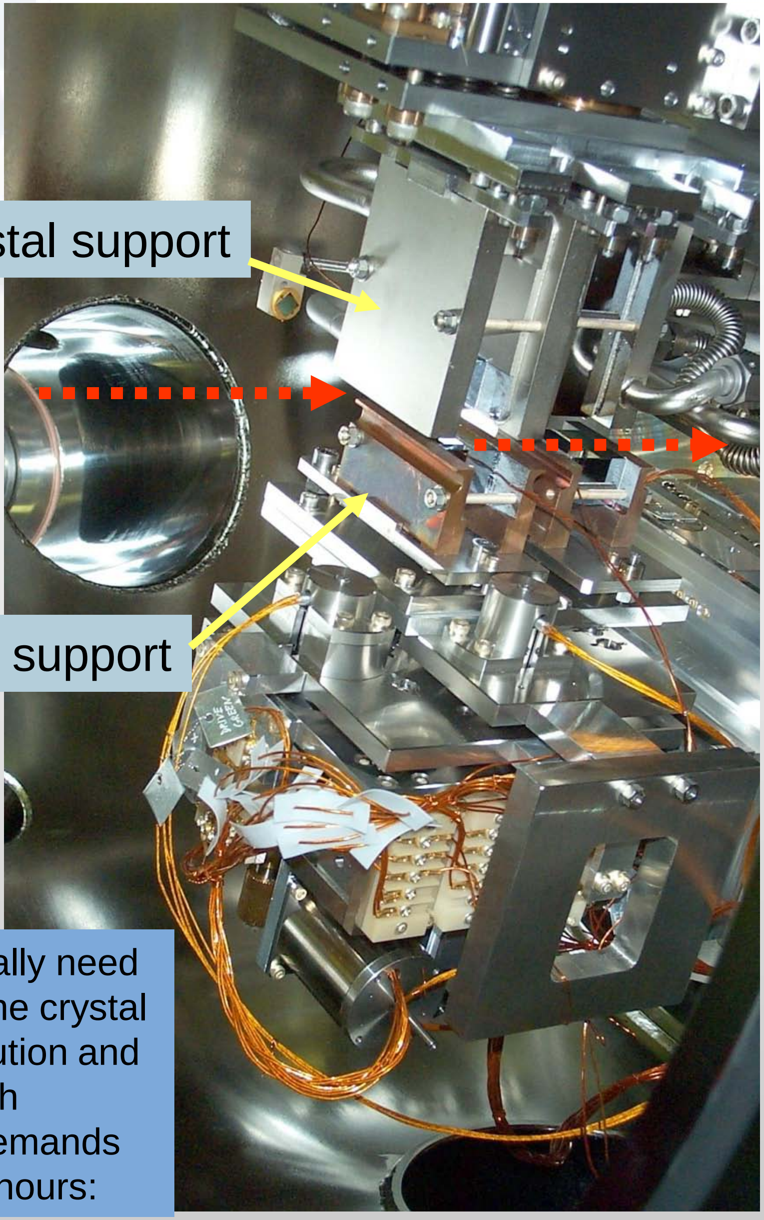
h k l are usually used, (e.g. 1 1 1, 3 3 3, 4 4 4), these are not Miller indices, but Laue indices, or “general Miller indices”.

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_s) & divergence of incident beam, ψ_0

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

e.g. **Si 111 reflexion**,
d_{hkl}= 3.1355Å
ω_s = 10.7 µrad (@ 8keV):

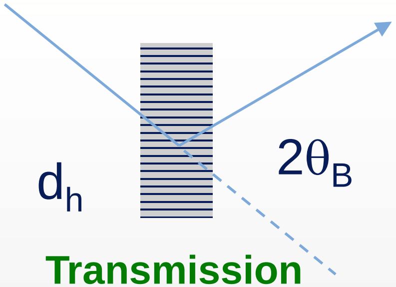
θ_B = 14°
with a parallel incident beam:
$\Delta E/E = 1.4.10^{-4}$, ΔE=1.1eV

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:

Single crystals : main applications

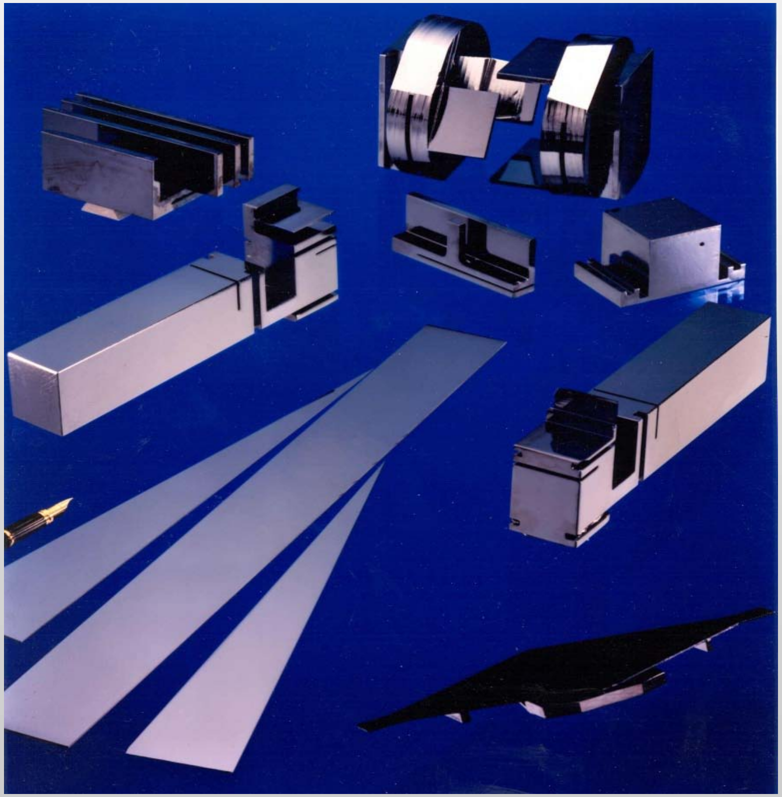
2 principal geometries:

Reflection
(Bragg case)
Most common

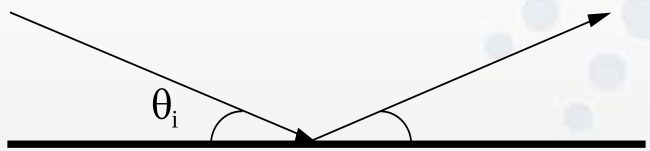


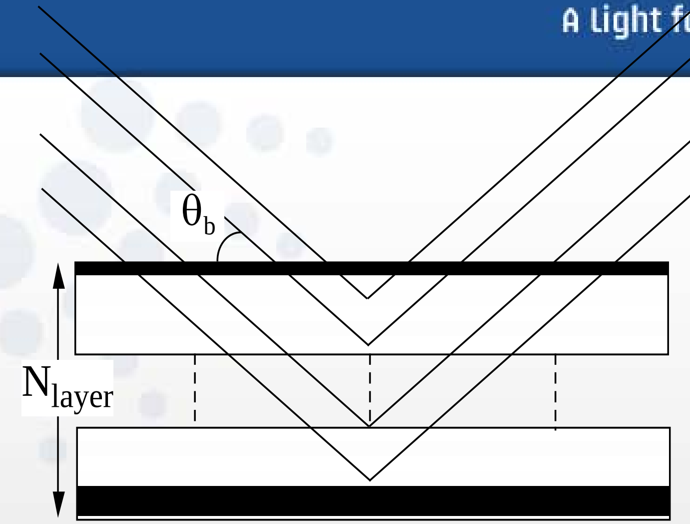
Transmission
(Laue case)
High energy, beam splitters

- **Applications**
  - High resolution monochromator / analyser
    - $10^{-8} < \Delta E/E < 10^{-3}$
  - Focussing monochromators
    - bent crystals
  - Collimation / beam expander
    - asymmetrical cut
  - Phase retarder (polariser)
- **Technical requirements**
  - Perfect crystals exist (Si, Ge, Diamond?...) but they must be tailored into monochromators
    - orientation, cutting, etching and polishing
    - strain free crystal preparation, accurate and stable mounting
  - Appropriate cooling scheme



high reflectivity x-ray mirrors… or synthetic crystals





single boundary

For $\theta_i > \theta_c$

$E_r = rE_0$ where E_r, E_0 are reflected and incident wave amplitudes, r is the amplitude reflectivity

$r < 10^{-2}$ and $R = |r|^2 < 10^{-4}$

$$R \propto \frac{1}{\sin^4 \theta_i}$$

multiple boundaries

ideally $|r| \times N_{layer}$ □R → 1

$n_l = 1 - \delta_1 + i\beta_1$ and $n_2 = 1 - \delta_2 + i\beta_2$

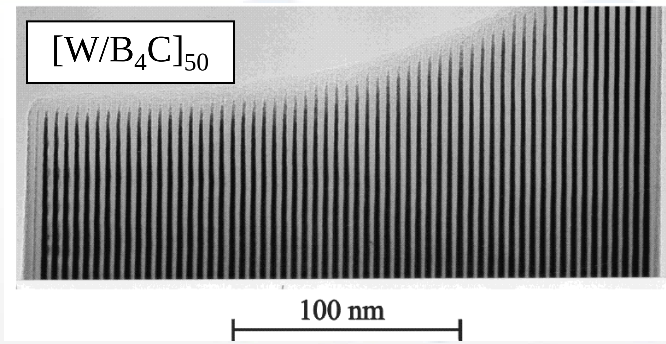
$$R \propto \frac{\Delta\delta^2 + \Delta\beta^2}{4} \frac{N_{layer}^2}{\sin^4 \theta_b}$$

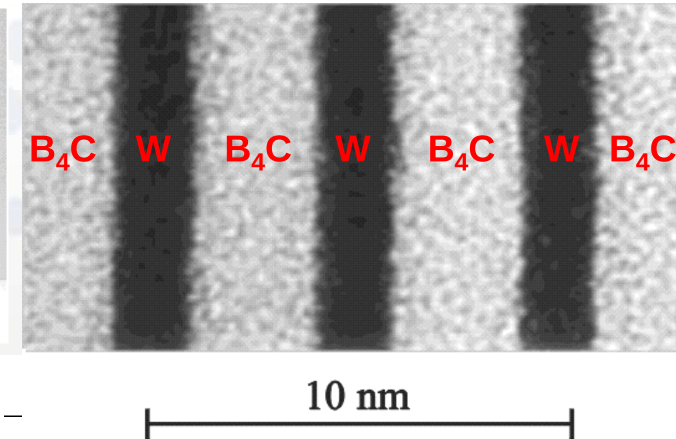
- **Wide energy band-pass monochromators, some soft X-ray monochromators**
- **Focusing : θ_b multilayer >> θ_c mirror → multilayer length << mirror length**

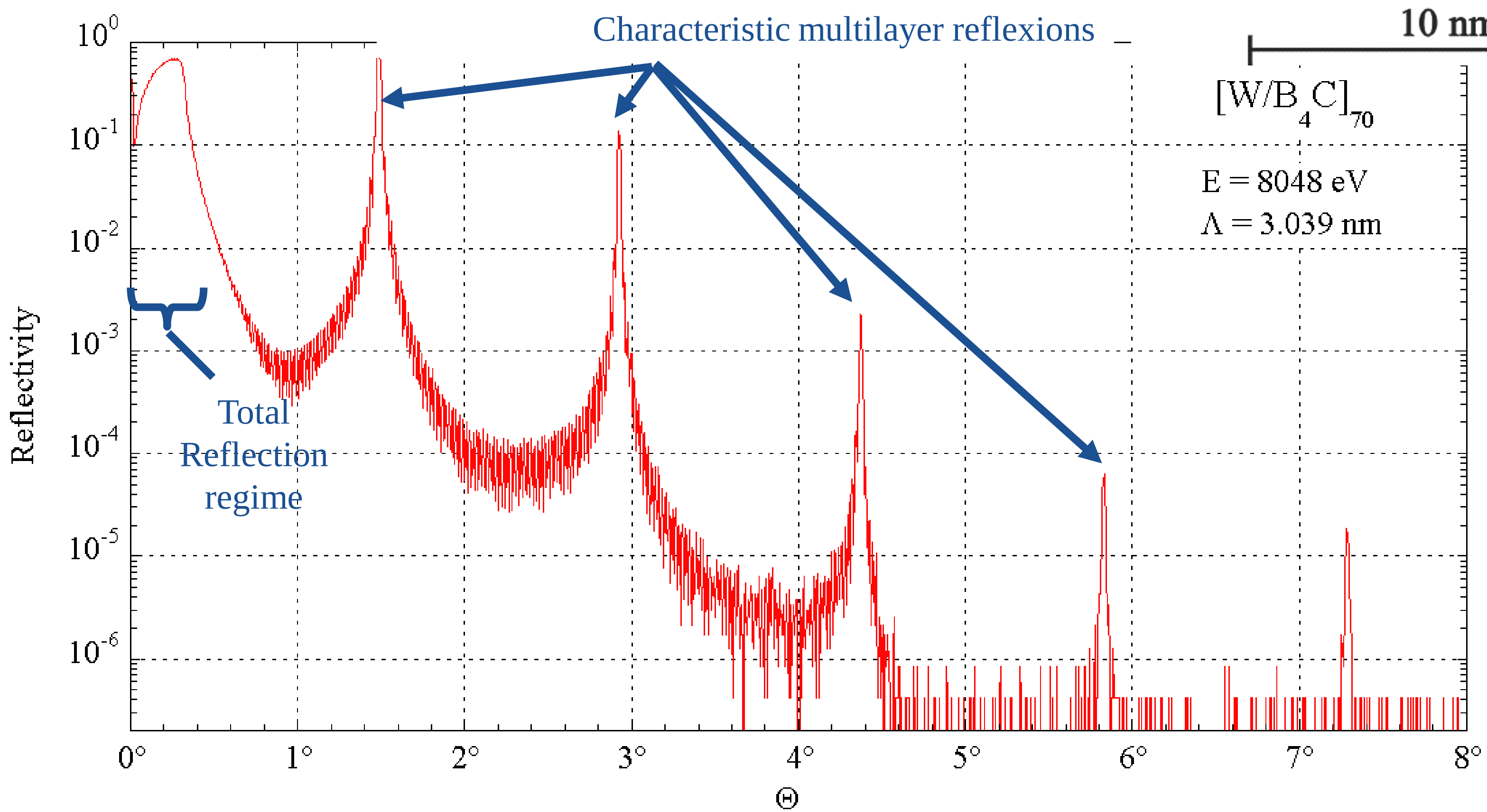
lower spherical aberrations (~L^2), increased numerical aperture

X-ray Multilayer Characterization

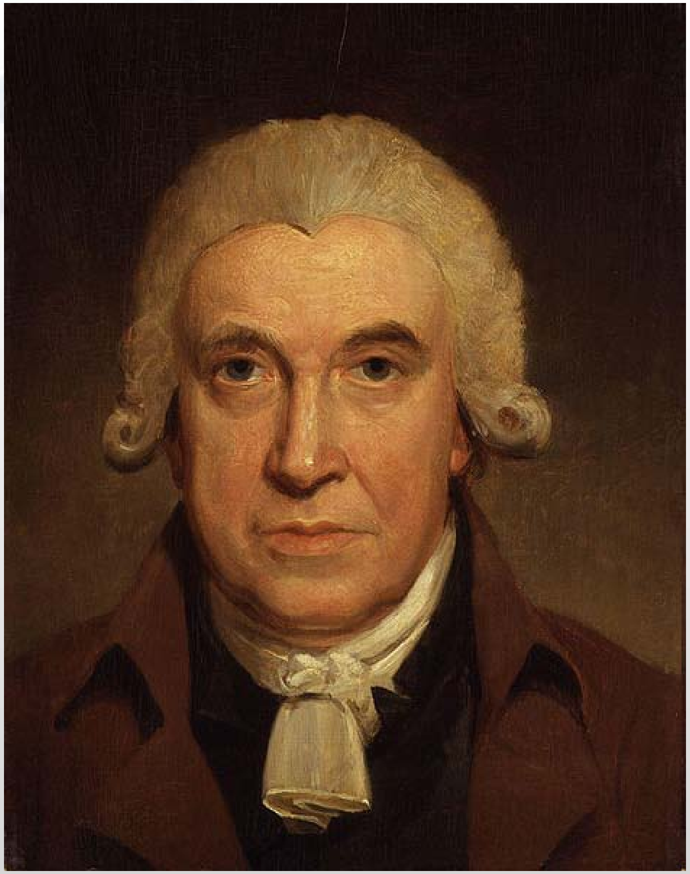
Typical X-ray reflectivity scan of a multilayer







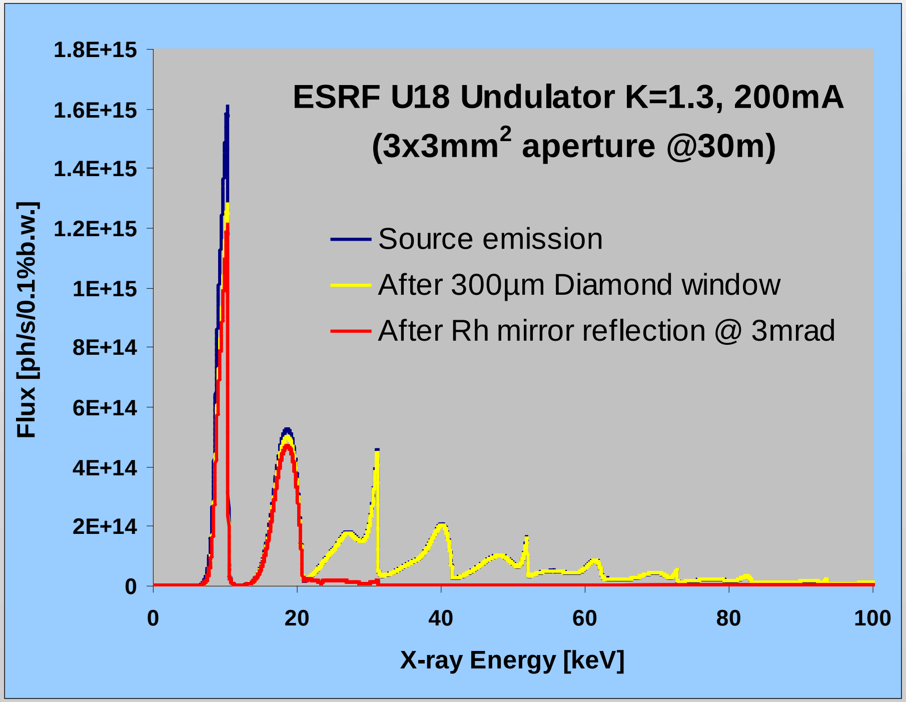
- Introduction
- General X-ray optics
- **High heat-load optics**
- X-ray micro-/nano-focusing
    - Reflectors
    - Zone plates
    - Refractive lenses
- Summary



James Watt
1736-1819

High Heat Load Optics

- ESRF U18 Undulator L=1.65m K=1.3, 90 periods, 200mA Storage Ring Current
- Total Emitted Power = 4.5kW
- Through 3x3mm aperture @ 30m = 1.4kW
- On-axis Power density 210W/mm²

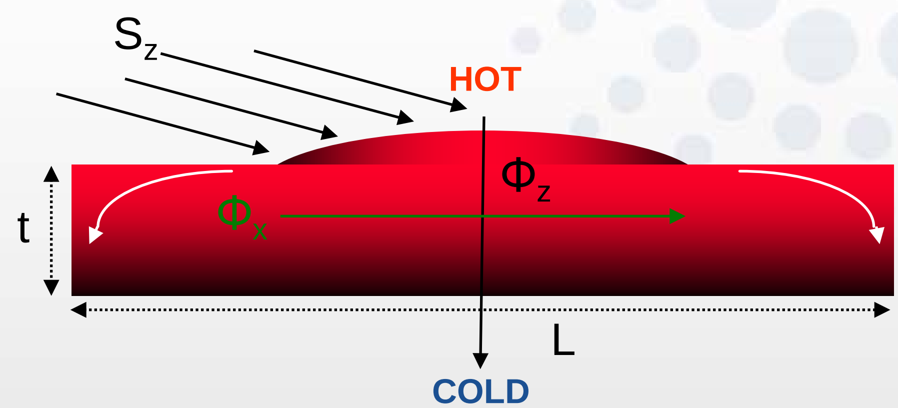


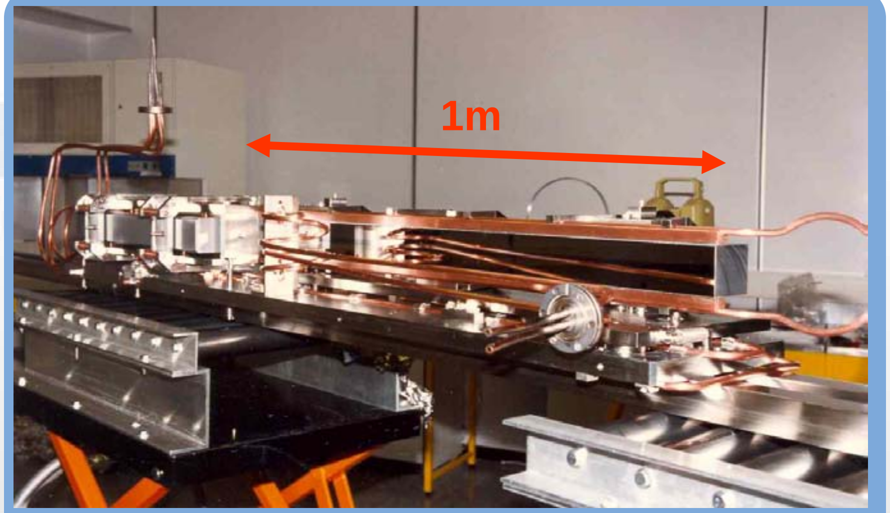
- 300µm polished diamond absorber (high pass Energy filter) absorbs 135W
- Rh-coated mirror reflecting at 3mrad (low-pass energy filter) absorbs 700W
- Around 550W incident on monochromator crystal – essentially all absorbed

- efficient cooling to prevent from melting
- minimization of induced thermal deformation
- materials resistant to intense X-ray beams

Thermal deformation of Mirrors

Gaussian intensity profile





Double Si mirrors (mounted face-to-face) with side-cooling geometry (water)

- Φ_x : local variation of thickness (thermal "bump")

$$\Delta_{bump} = \frac{\delta z}{\delta x} \propto \frac{\alpha}{\kappa} G P_s$$

- Φ_z : differential expansion hot-cold sides (thermal "bending")

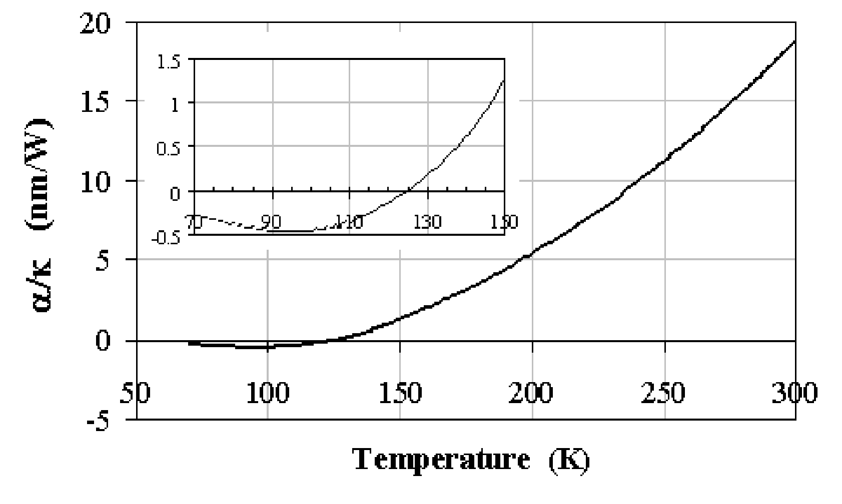
$$\Delta_{bending} = \frac{\delta x}{\delta z} \propto \frac{\alpha}{\kappa} G P_t$$

$$\begin{cases} \alpha : \text{thermal expansion} \\ \kappa : \text{thermal conductivity} \\ \text{G} : \text{cooling geometry factor } (\sim 0.01\text{-}1) \\ \text{P}_\text{s} : \text{Power density} \\ \text{P}_\text{t} : \text{Total power} \end{cases}$$

Generally mirror is over-illuminated to minimise Φ_x

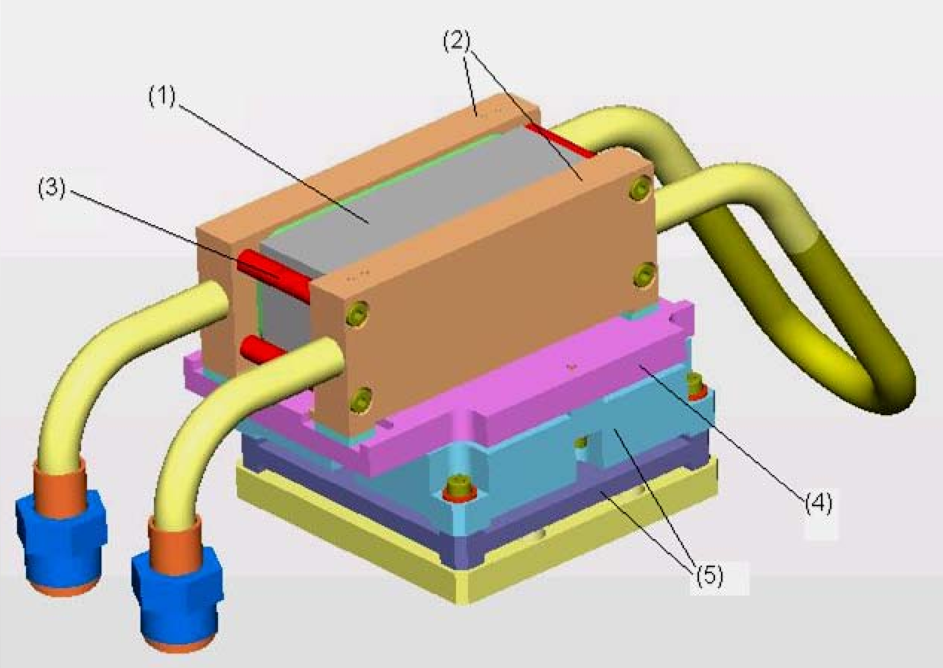
Monochromator Cooling

- Darwin widths of typical crystal reflexions are in the µrad range and below:
  - Monochromator performance is particularly sensitive to thermal deformations of diffracting crystals
  - By cooling Si to cryogenic temperatures (LN2 sufficient) – thermal deformations due to beam absorption can be minimised.



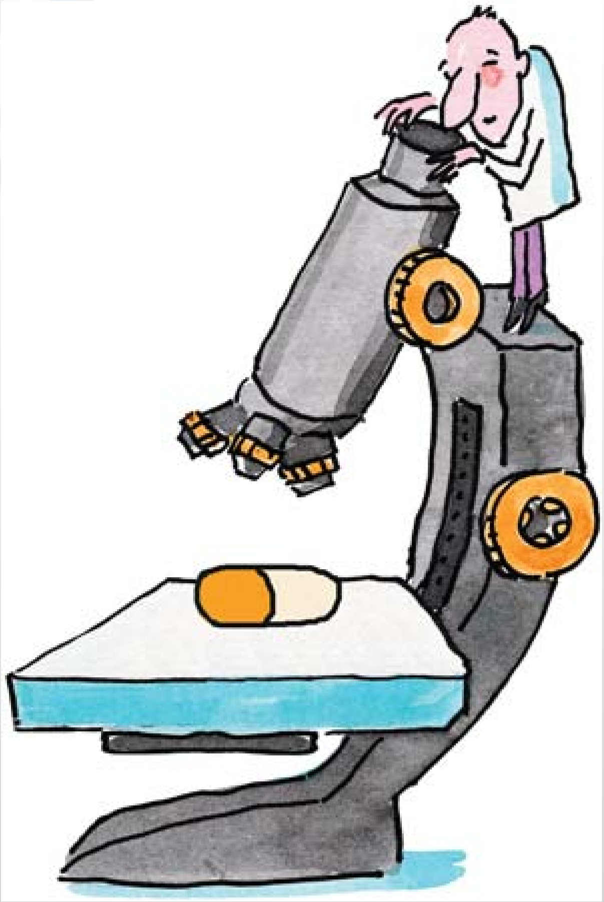
Ratio of thermal expansion and thermal conductivity of Si α/κ vs temperature

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



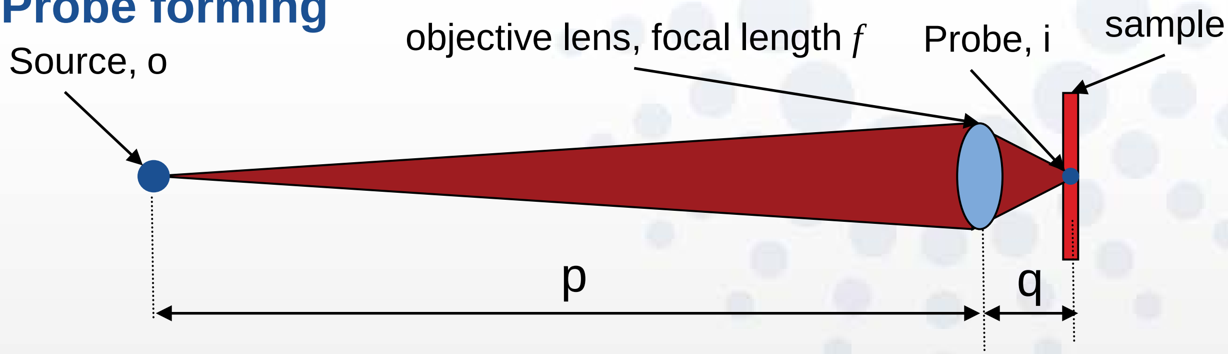
- **Crystal is side cooled with cooling blocks clamped with pressures between 5-10bar**
- **Deformation of crystal planes due to clamping <1µrad**
- **Over 17 ESRF beamlines using LN2 cooled monochromators**

- Introduction
- General X-ray optics
- High heat-load optics
- **X-ray micro-/nano-focusing**
  - Reflectors
  - Zone plates
  - Refractive lenses
- Summary



Typical X-ray focusing schemes

Probe forming



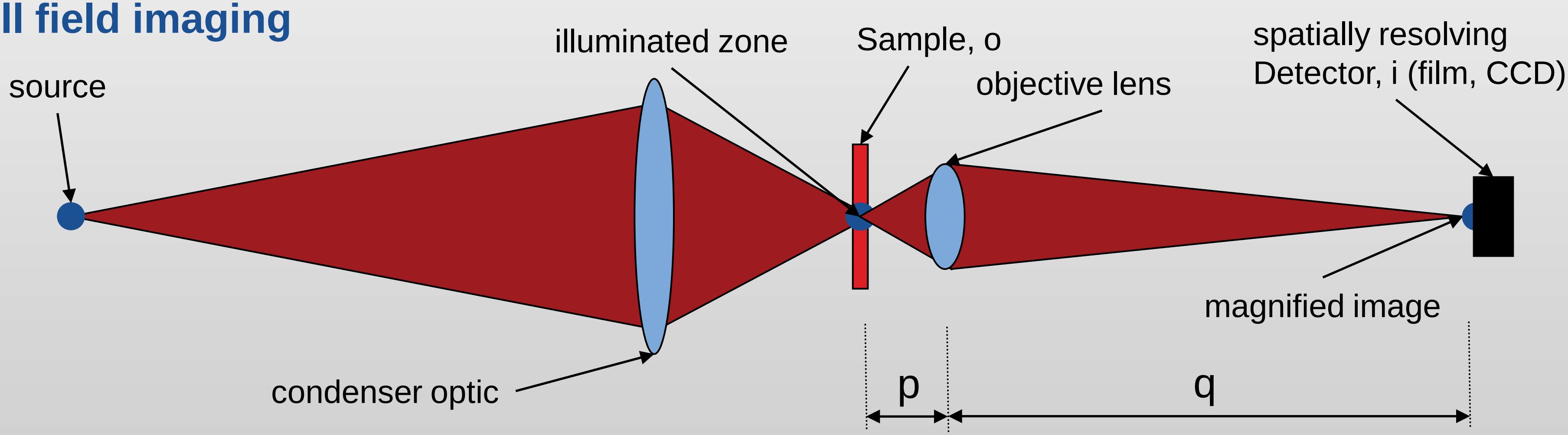
Thin lens $\frac{1}{p}+\frac{1}{q}=\frac{1}{f}$

Magnification, $M=\frac{i}{o}=\frac{q}{p}$

For fixed p, $M\uparrow \Rightarrow f\downarrow$

- Detection of signals arising from interaction of small probe with sample
- Sample scanned through beam to build ‘image’ pixel by pixel

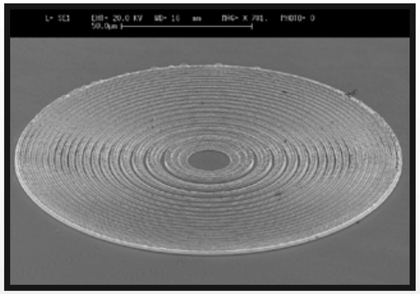
Full field imaging



- Condenser optic → illuminated zone on sample
- Objective lens → magnified image of sample on spatially resolving detector

X-ray focusing optics

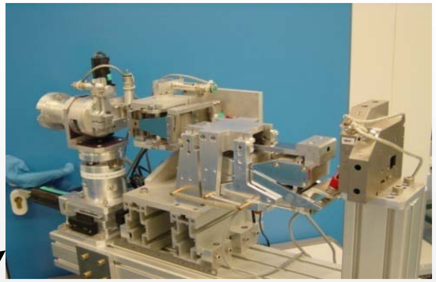
Diffractive optics

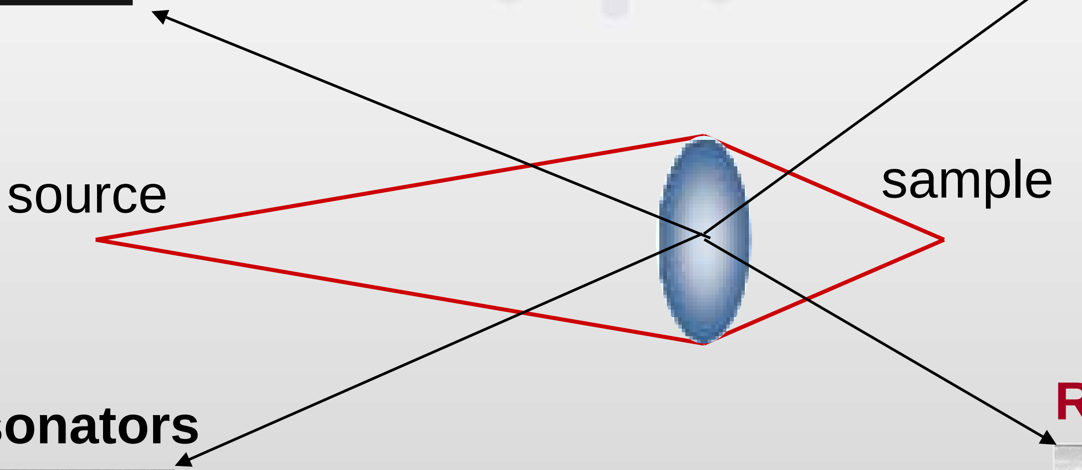


- *Fresnel zone-plate (FZP)*
- *Bragg-Fresnel*
- *Crystals*
- *Multilayers*

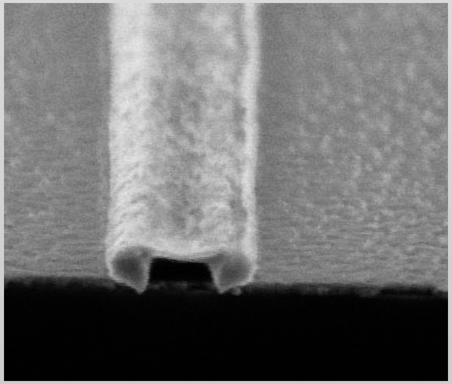
X-ray reflectors

- *Kirkpatrick-Baez*
- *Wolter mirror*
- *Ellipsoidal mirror*
- *Micro-channel*
- *(Poly)capillaries*





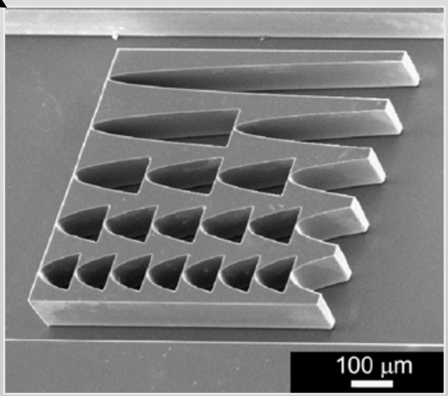
X-ray resonators



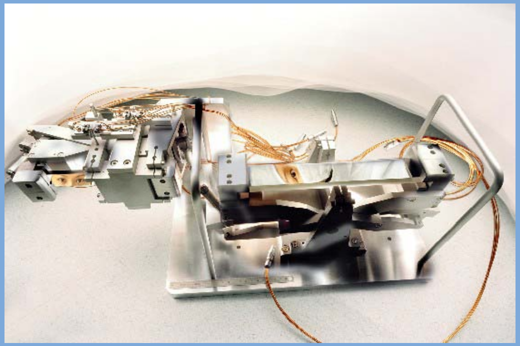
- ***Waveguides***

Refractive lenses

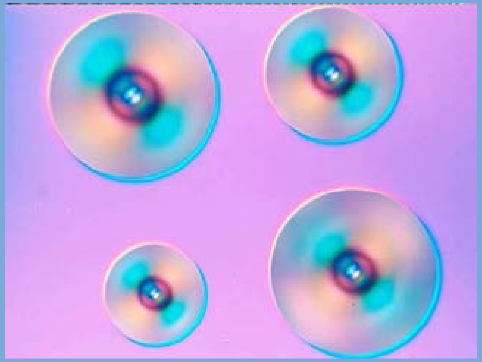
- *Compound refractive lenses (CRL)*



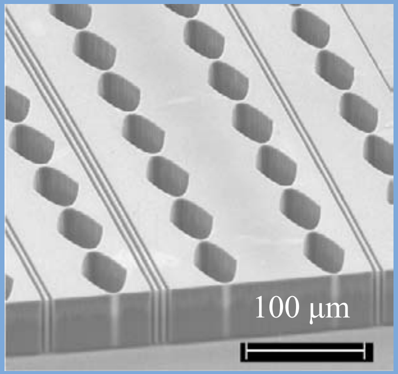
- There are no insurmountable theoretical barriers to achieving sub-10nm resolution X-ray focusing
- BUT the technological challenges are considerable



Reflective Optics:
Figure Error
Thermal, mechanical stability,
Graded multilayers



Diffractive Optics:
Feature size/placement
Structure heights (efficiency)

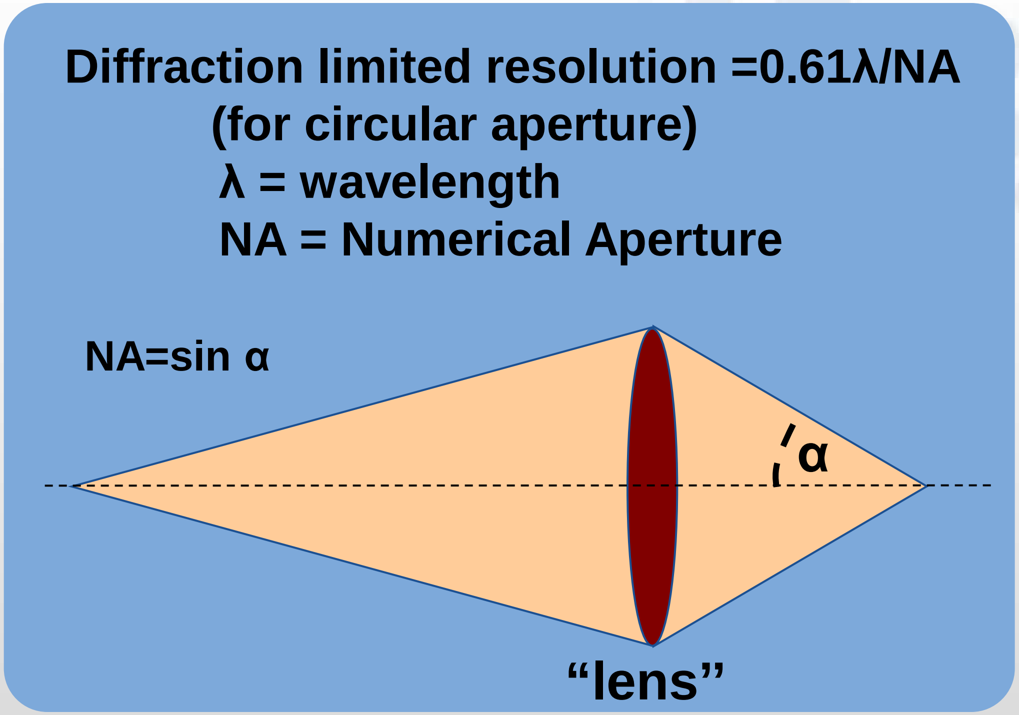


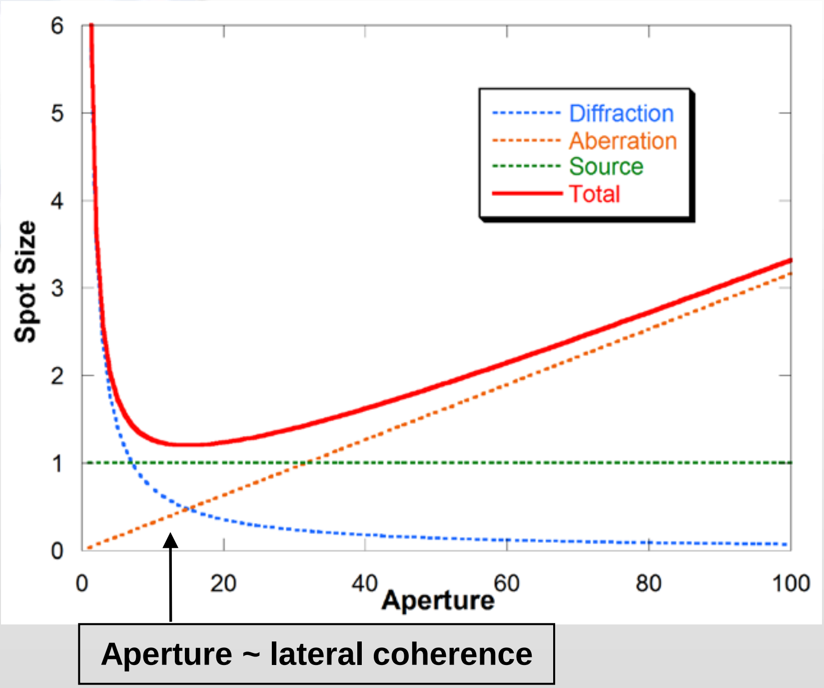
Refractive Optics:
Absorption limited numerical aperture
Side-wall roughness

Resolution is not the only parameter. Often photon flux, focus stability is at least as important. Large acceptance, high efficiencies…

How to reach high resolutions?

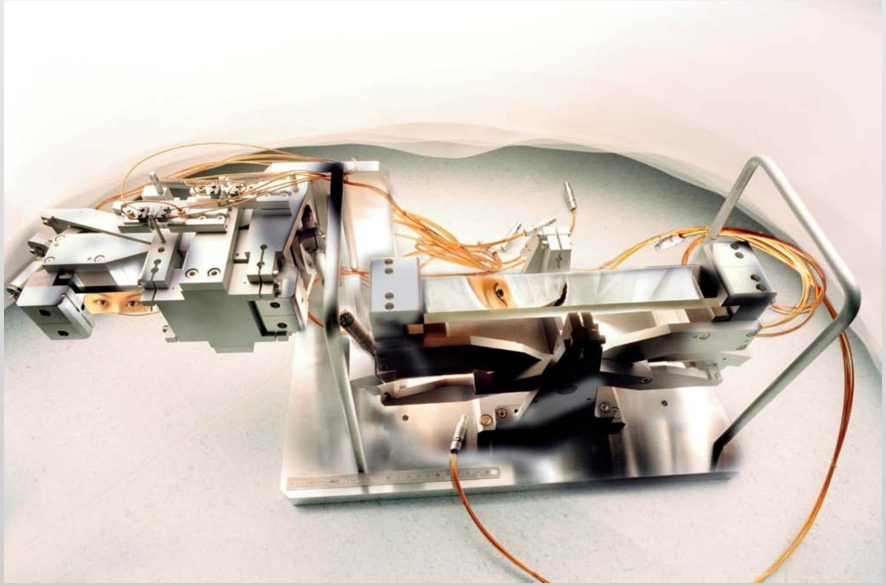
- High demagnifications **but** fundamental limit: Rayleigh criterion:





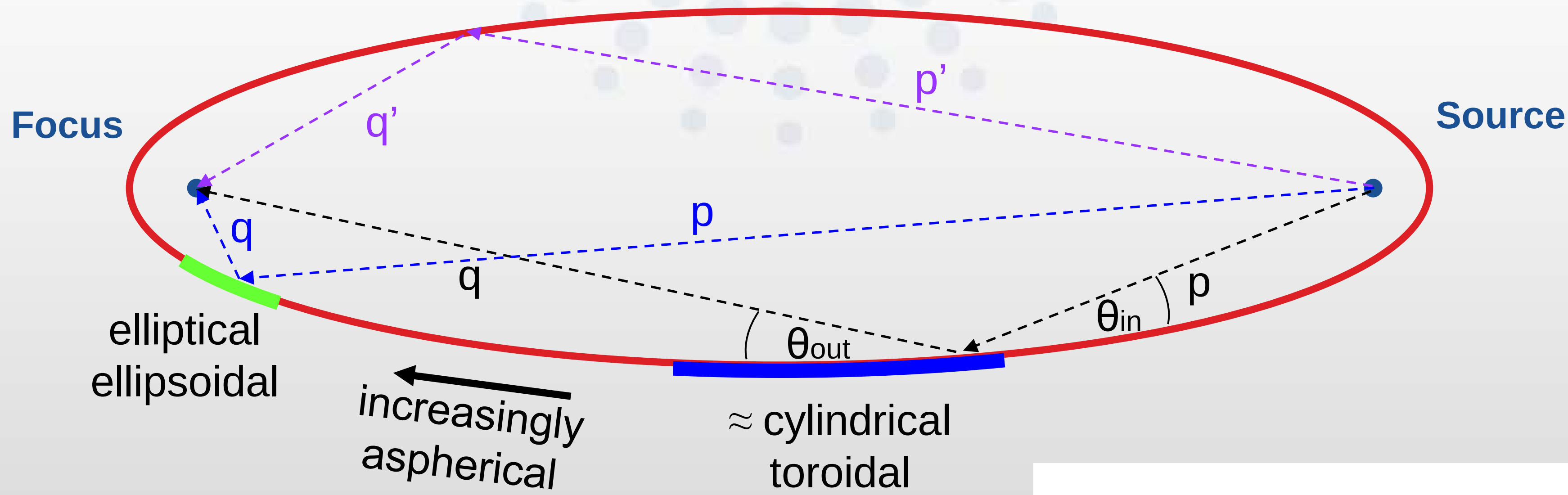
Increase NA – longer mirrors & higher grazing angles (MLs), larger diameter CRL, ZPs
Generally aberrations or flux loss will impose resolution limits
Best current technologies for hard X-rays NA ~ 0.003

- Introduction
- General X-ray optics
- High heat-load optics
- X-ray micro-/nano-focusing
  - **Reflectors**
  - Zone plates
  - Refractive lenses
- Summary



Ideal mirror focusing of a point source

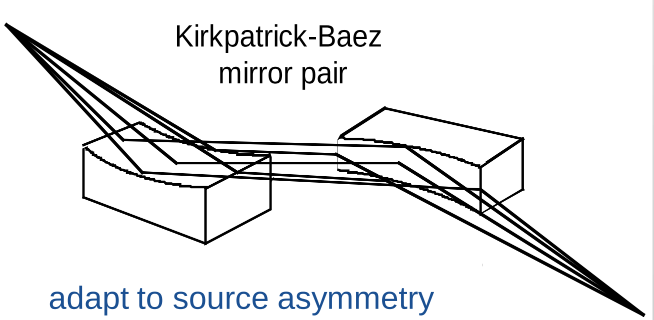
Ellipse: p + q = constant, $\theta_{in} = \theta_{out}$
Focusing: equivalent optical path lengths from source-to-focus
'Ideal' mirror would have an elliptical (2D) or ellipsoidal (3D) surface figure



$\boxed{\text{focus size} \approx \text{source size x q/p}}$

Micro/nanofocusing applications:

- good ellipsoidal mirrors not readily manufacturable
- perpendicular elliptical mirrors
  - Kirkpatrick-Baez (KB) configuration

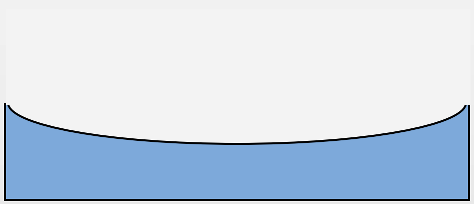


adapt to source asymmetry
- apertures
- demagnification

Two basic approaches:

Static Figuring

- Elliptical figure polished into mirror substrate



- Relatively simple mechanics
- OK for very short radius ellipses
- Lengthy/expensive fabrication
- Only optimised for one set of operating conditions (incidence angle, focusing distance)

Dynamic Figuring

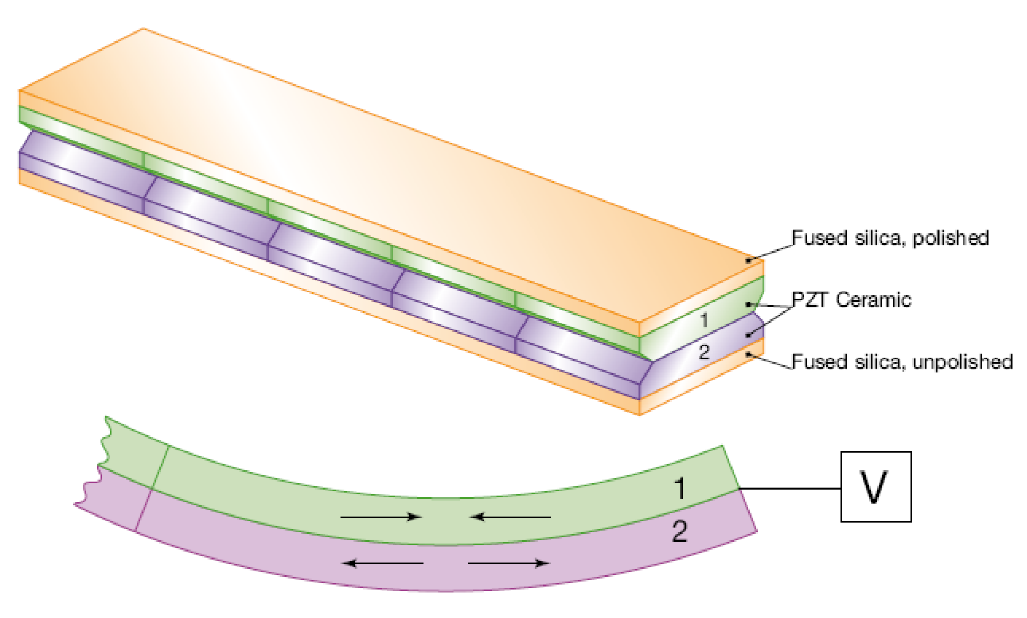
- Elliptical figure by mechanical bending of a (usually flat) mirror substrate

- Simple substrate polishing
- Relatively cheap systems
- Active systems allowing modification of focusing parameters (permits use at variable energy with Multilayer coatings)
- Not well adapted for very short radius ellipse (mirror will break!)

Dynamically bent mirrors

- 2 major classes:
- Piezoelectric bimorph systems (ESRF, SESO, FMB-Oxford, ACCEL)
- Mechanically actuated systems (ESRF, ALS, APS, SOLEIL, Irelec, X-Radia)
- Extension of these technologies – increase number of actuators to correct local figure errors – active optics (several projects)

2 independent bending moments: Elliptical Figure



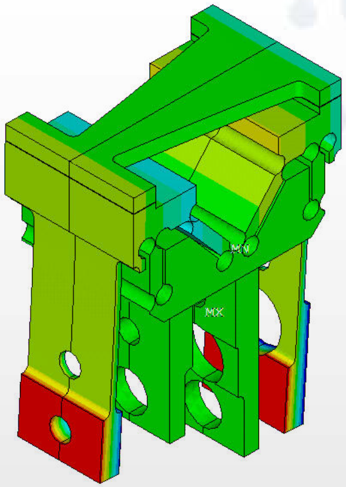
Principle of Bimorph Mirror (from FMB-Oxford)

2 independent actuators

ESRF mirror bender based on monolithic flexure hinge technology

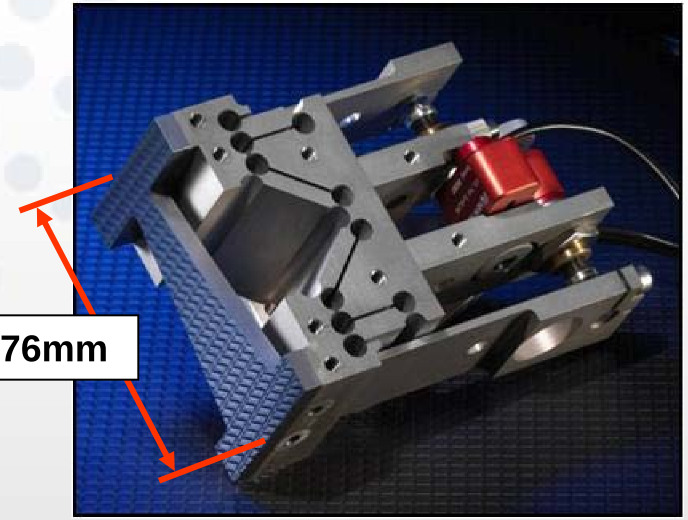
ESRF dynamic focusing KB system

Full FEA modelling for shape optimisation

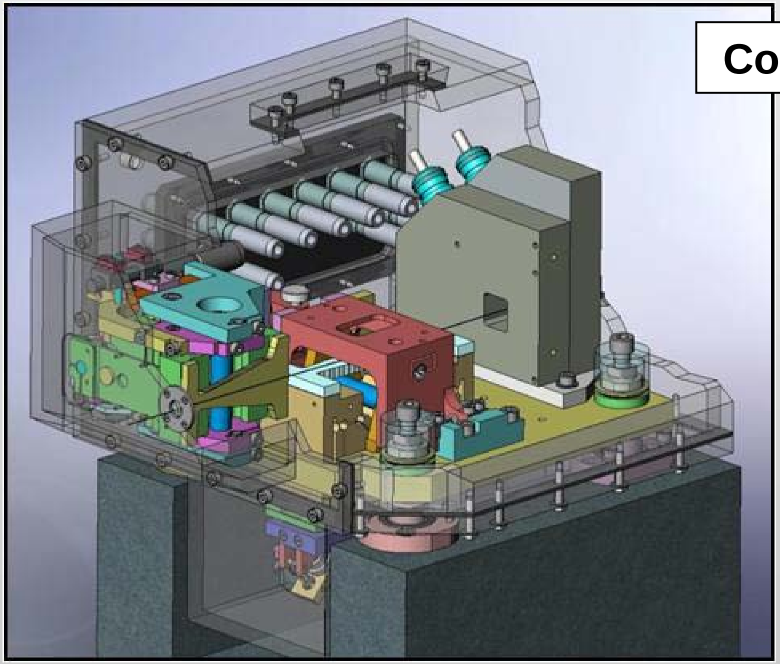


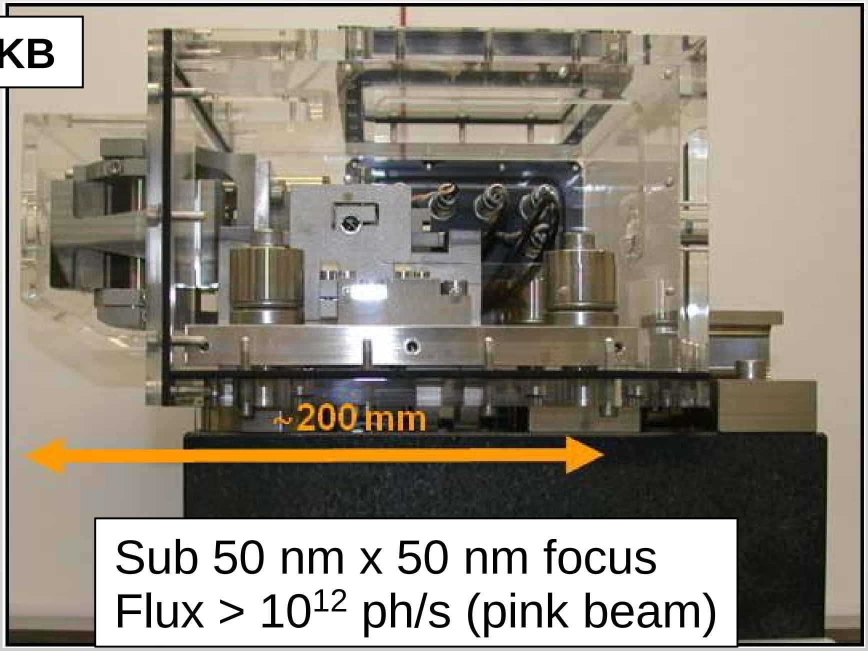
ID22-NI horizontally focusing bender

- Bonded optics
- Mirror width profile machined to +/-3µm
- Close to fracture stress limit
- Optimized Si orientation



Complete KB



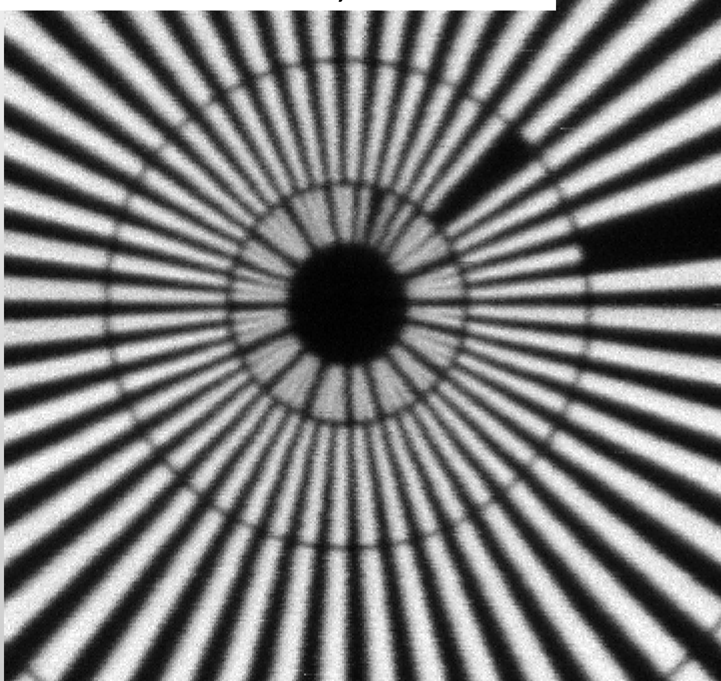


Sub 50 nm x 50 nm focus
Flux > 10^{12} ph/s (pink beam)

Projection Microscopy using KB optics

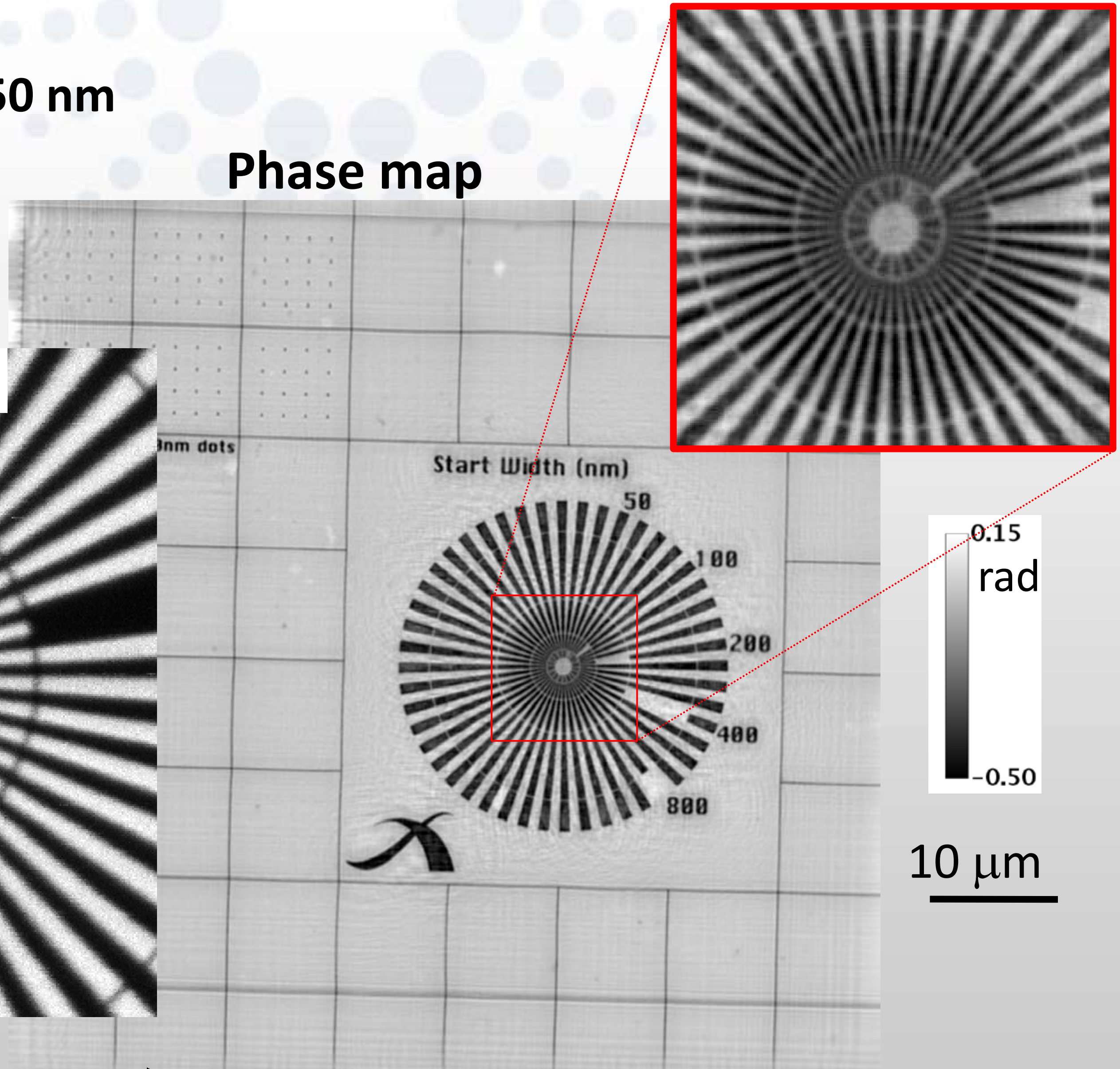
Thin gold test pattern
Innermost line width: 50 nm
Energy = 17.3 keV
Field of view: 80 ✪m
Pixel size: 53 nm

Au Fluorescence; 25 nm



9 ✪m

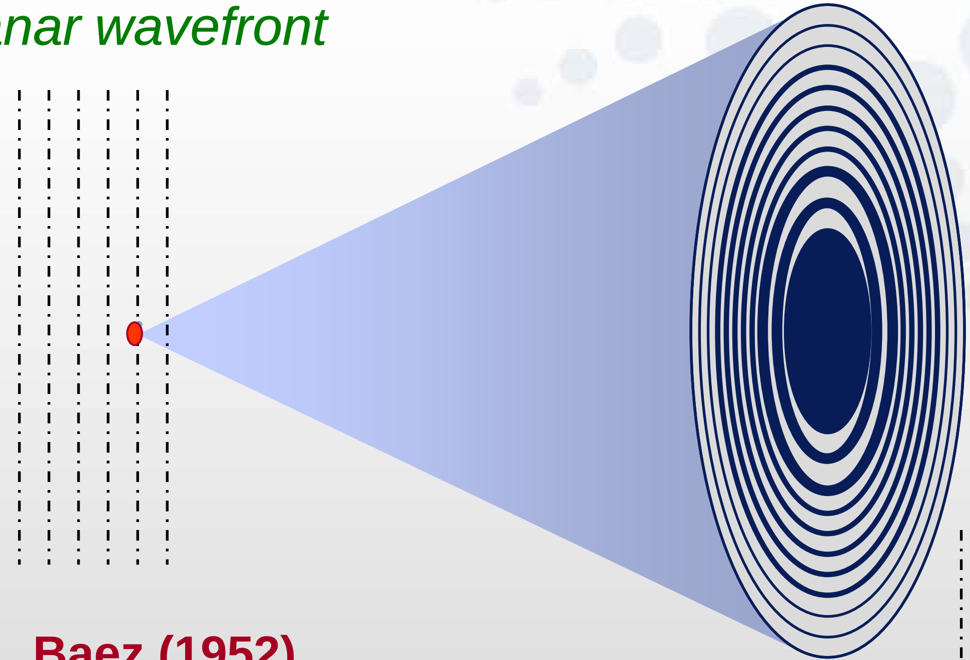
Phase map

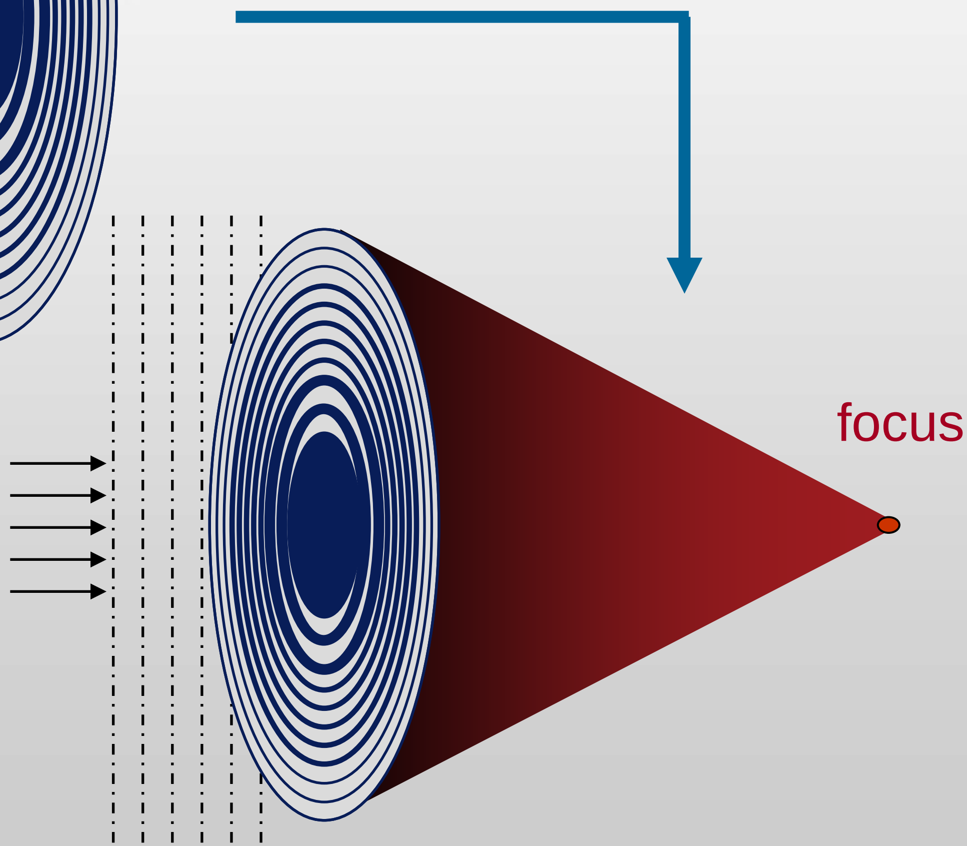


- Introduction
- General X-ray optics
- High heat-load optics
- X-ray micro-/nano-focusing
  - Reflectors
  - Zone plates
  - Refractive lenses
- Summary

Augustin-Jean Fresnel
1788-1827

Fresnel zone plates



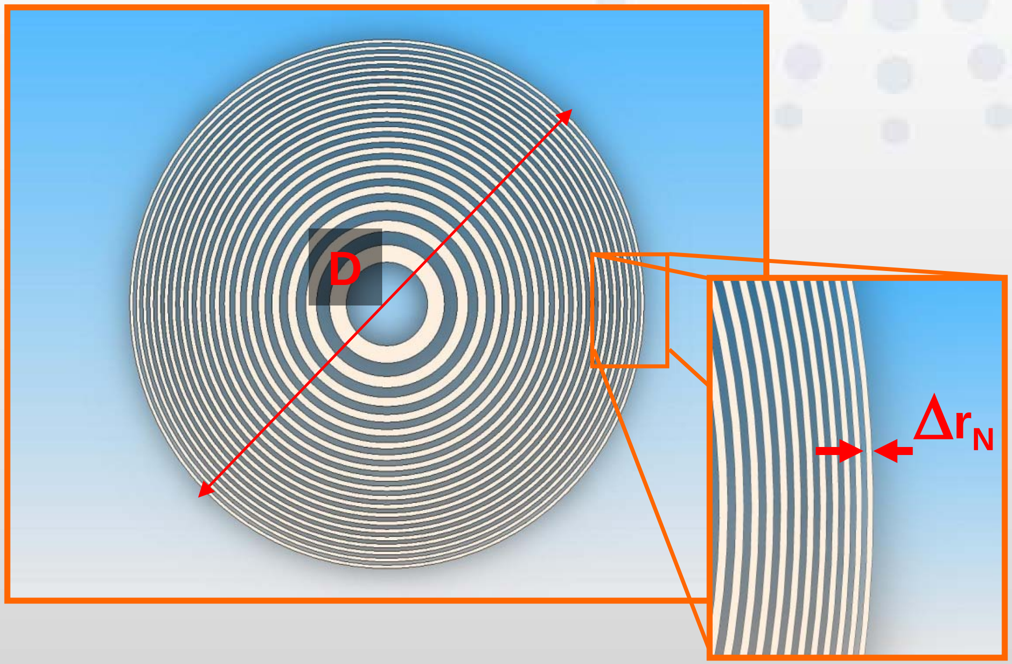


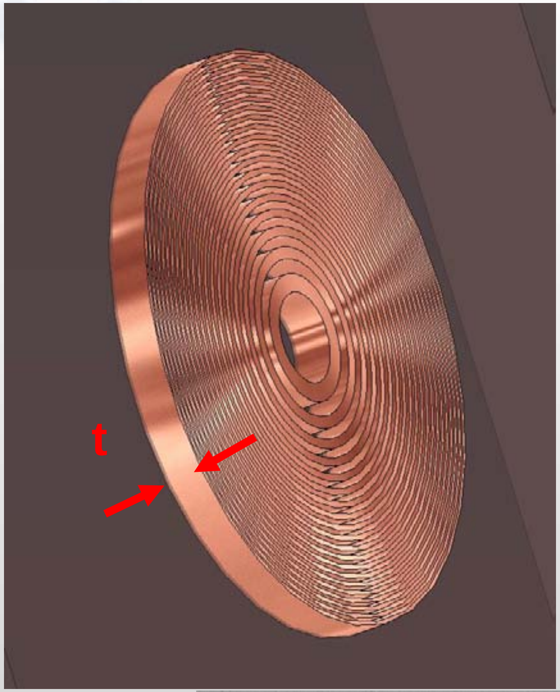
Baez (1952)
Schmahl (1969)
Kirz (1971)
Niemann (1974)

Gabor hologram of a point object

Fresnel Zone Plates

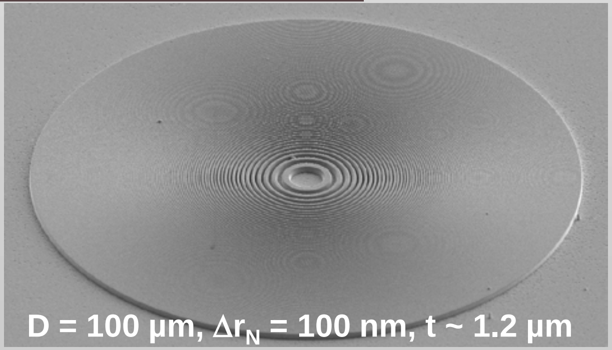
- **Diffractive X-ray Lenses: Circular transmissive diffraction gratings with radially decreasing line width giving focusing effect**





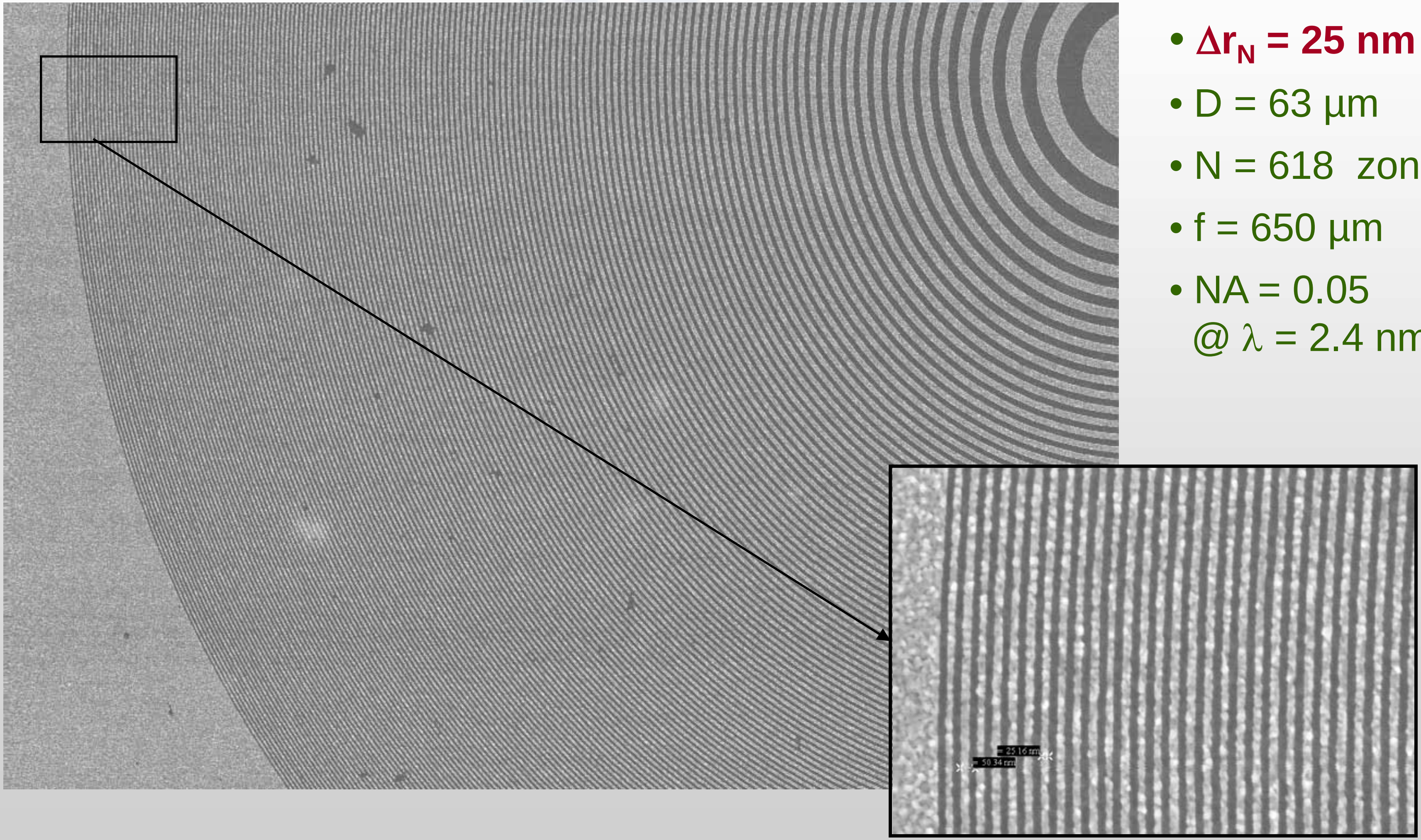
Alternate ‘zones’ modify phase/amplitude of incident wavefront: for material of thickness, t, wavelength, λ, refractive index $1-\delta-i\beta$, phase shift, $\Delta\phi$, is:

$$\Delta\phi = \frac{2\pi\delta t}{\lambda}$$



D = 100 µm, Δr_N = 100 nm, t ~ 1.2 µm

Soft X-ray zone-plates

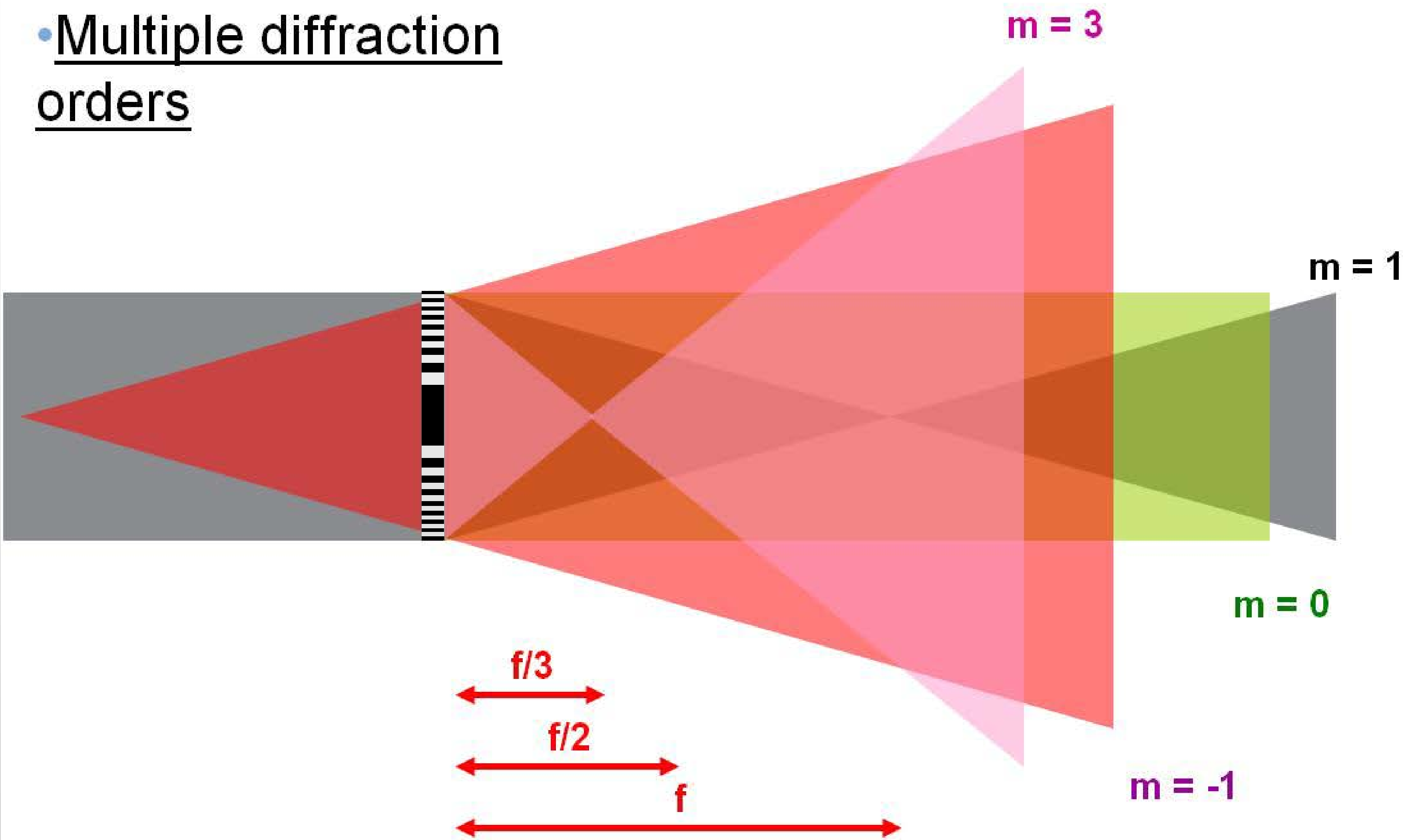


- **Δr_N = 25 nm**
- D = 63 µm
- N = 618 zones
- f = 650 µm
- NA = 0.05
  @ λ = 2.4 nm

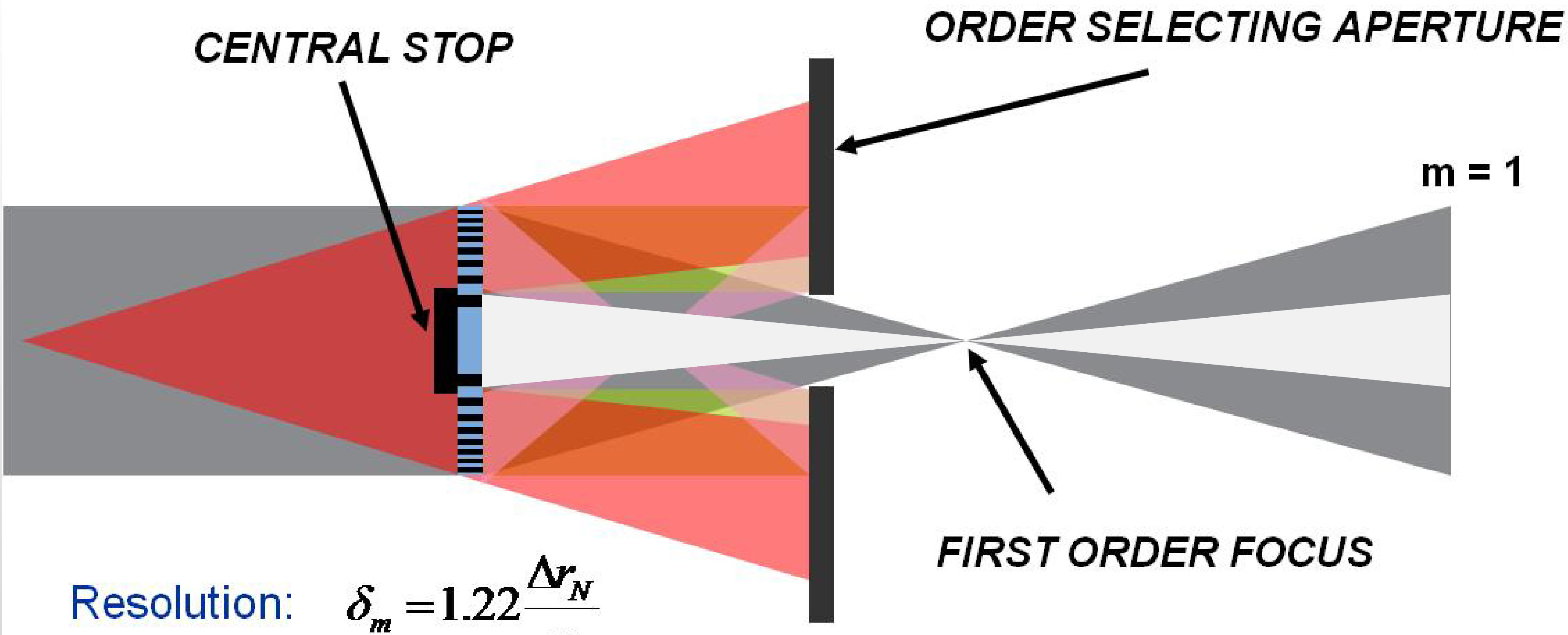
E. Anderson, **C**enter for **X**-**R**ay **O**ptics, LBNL, USA

Focusing behaviour of Fresnel Lenses

- Multiple diffraction orders



Rejection of the unwanted diffraction orders



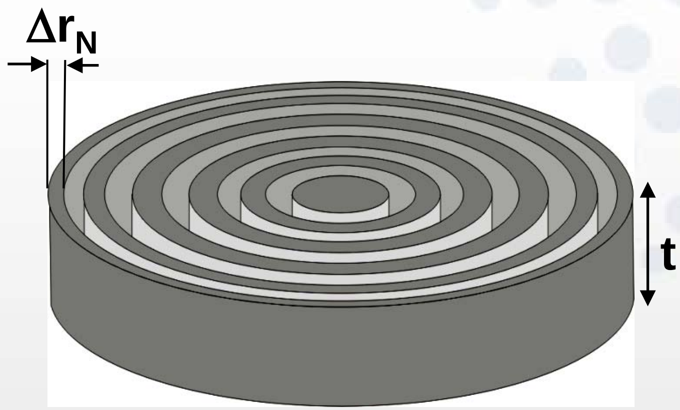
Resolution: $\delta_m = 1.22\dfrac{\Delta r_N}{m}$

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

Depth of focus: $DOF = \pm\dfrac{2\Delta r_N^{\,2}}{m\,\lambda}$

Zone plate aspect ratio

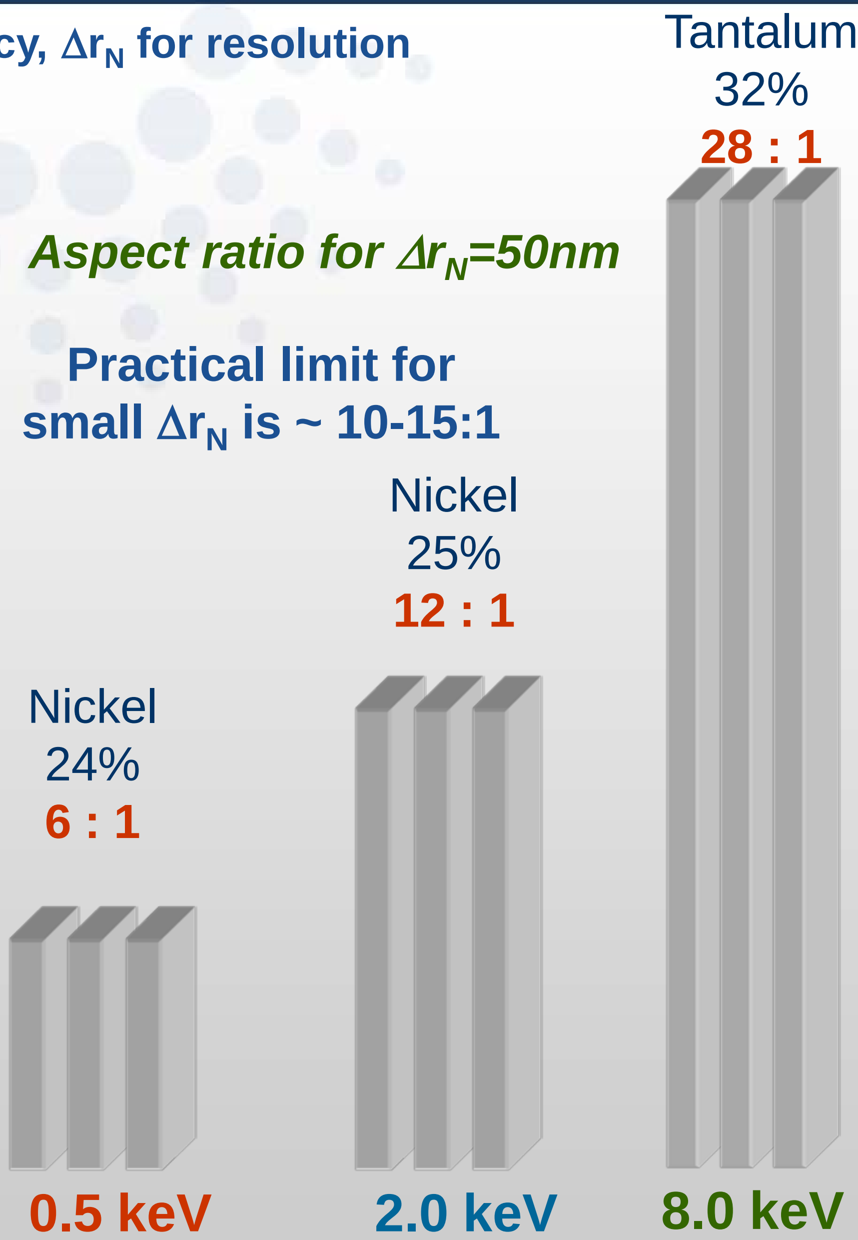
structure height, t, critical for efficiency, Δr_N for resolution



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

Aspect ratio for Δr_N=50nm

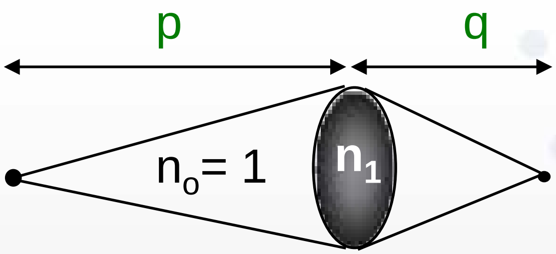
Practical limit for small Δr_N is ~ 10-15:1



- Introduction
- General X-ray optics
- High heat-load optics
- X-ray micro-/nano-focusing
  - Reflectors
  - Zone plates
  - **Refractive lenses**
- Summary

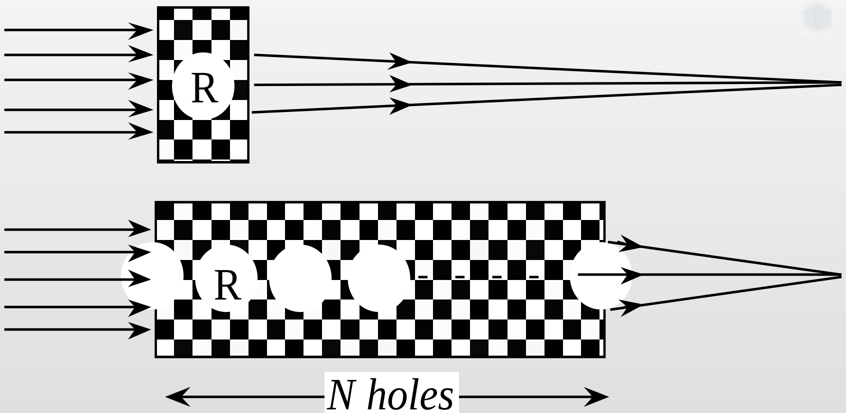
Willebrord Snellius 1580-1626

Compound refractive lens



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

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



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

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

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

$n_1 < 1$: concave lens

Example :

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

1 □hole of 100 µm radius : f = 9 m

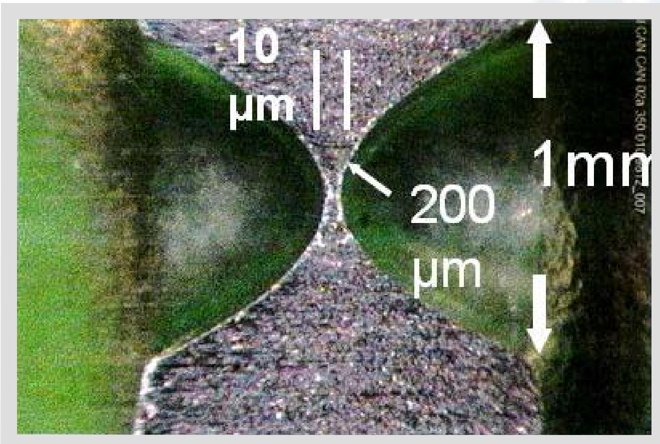
15 holes of 100 µm radius: f = 60 cm

Advantages
- simplicity and low cost
- low sensitivity to heat load

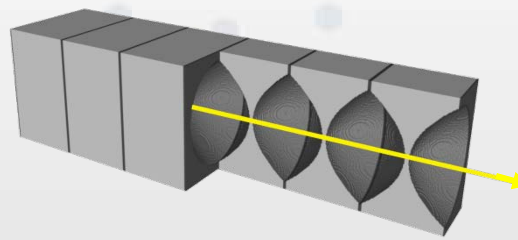
Disadvantages
- efficiency limited by absorption
- small aperture (limited resolution)
- strong chromatic aberrations

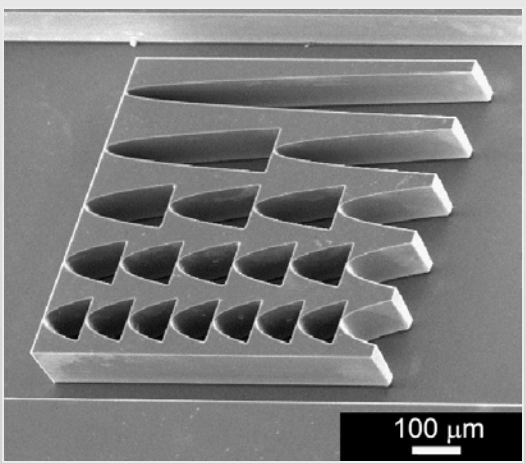
A. Snigirev et al. Nature, 384 (1996)

Parabolic Refractive lenses

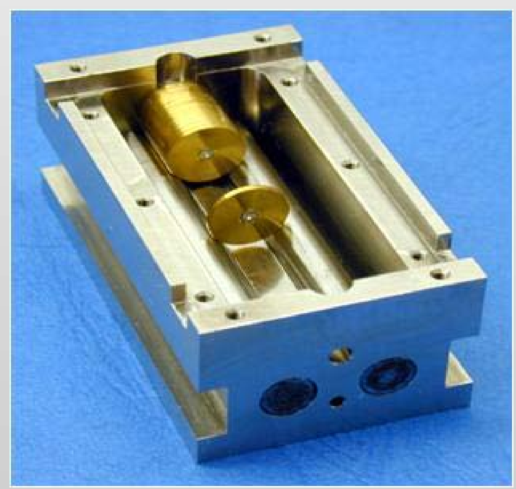


Materials:
low Z, high density
Al, Be, B, Si, …



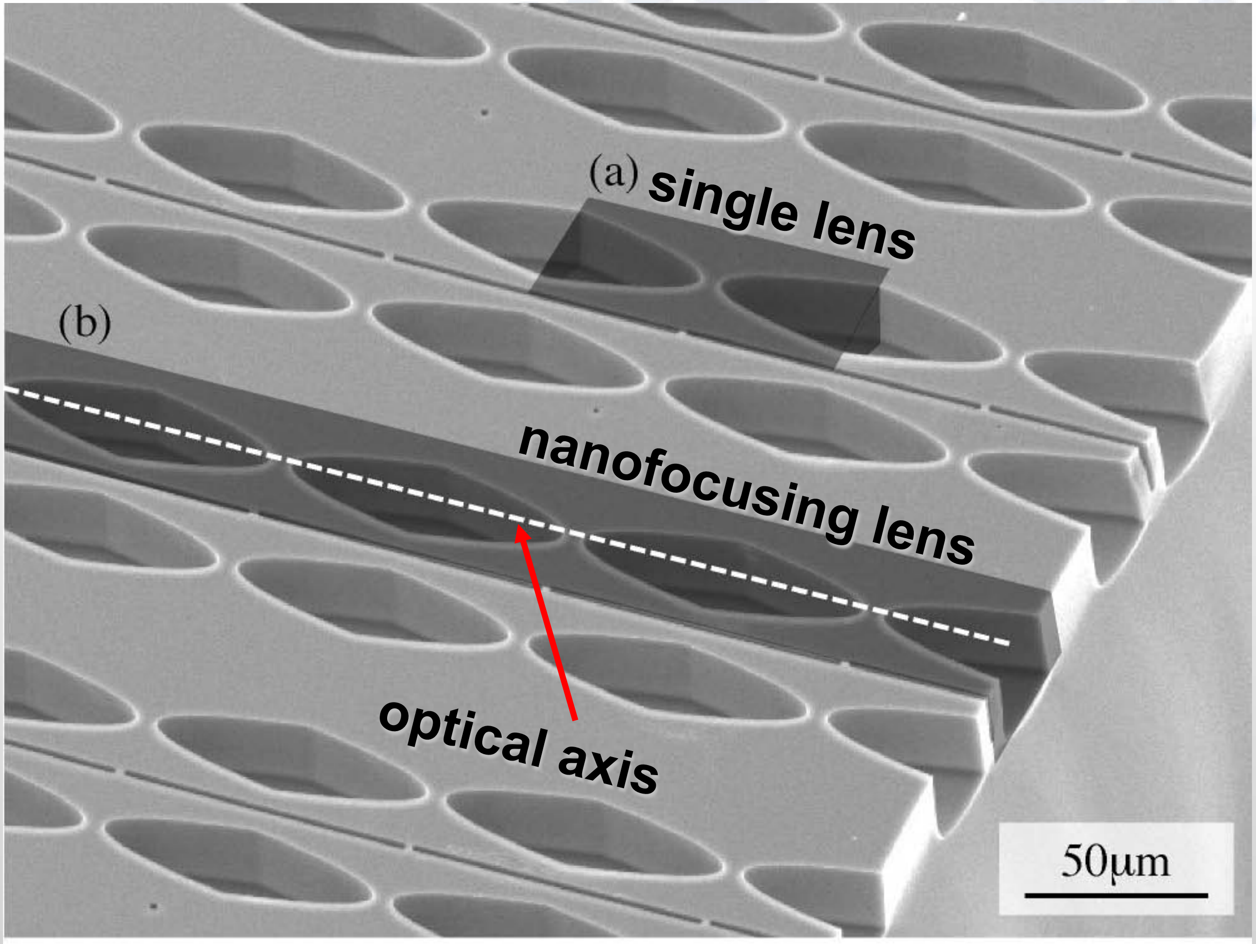


C. David et al.
PSI, Villigen, Switzerland



B. Lengeler, C. Schroer, M. Richwin,
RWTH, Aachen, Germany

Planar Compound Refractive Lenses



lens made of Si by e-beam lithography and deep trench reactive ion etching

extreme curvature:

R = 1µm - 3µm

N = 50 - 100

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

Progress in Hard X-ray Focusing

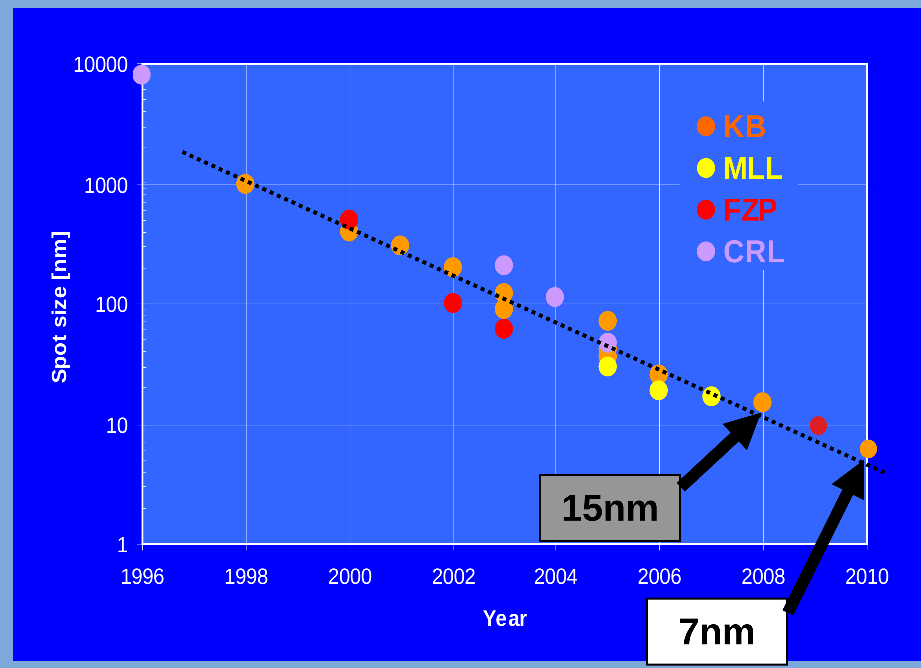
Moore's law adapted to the X-ray world:

ESRF Red Book (1987): very few beamline projects aiming even for 10 micron sized beams

Now optics ~~realistically aiming~~ for 10nm beams

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)*

- H. Mimura *et al. Nature Physics*, **6**, 122-125 (2010).
- J. Vila-Comamala et al., Ultramicroscopy,**109**,1360–1364 (2009)
- 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

Summary & conclusion

The advent of 3rd generation synchrotron X-ray sources has encouraged the development of new X-ray optics

- dramatic improvement in manufacturing and preparation techniques
  - low roughness, high-accuracy figuring, perfect crystals (Ge, Si), diamond, ...
- improved power management strategies
- focusing optics (spot size ~ 50- 0.01µm)
  - zone-plate and refractive lenses, elliptically figured mirrors (Bragg-Fresnel lenses, capillaries, wave guides)
- wide range of experimental requirements – no one ideal optic

- ***R&D programs continuously in progress**: current hot-topics*
  - preservation of the wave-front quality – especially important for anticipated use of fully coherent XFEL sources
  - sub-10nm focusing

Going further : useful reading

General books and reviews

- **X-ray science and technology** edited by A.G. Michette and C.J. Buckley (King's College London), Institute of Physics Publishing, (1993)
- **Soft X-ray Optics** by E. Spiller SPIE Engineering Press, ISBN 0-8194-1655-X, (1994)
- **Gratings, Mirrors and Slits *(Beamline design for Soft X-ray Synchrotron Radiation Sources)*** by W. B. Peatman, Gordon and Breach Science Publishers, ISBN 90-5699-028-4, (1997)
- **Soft X-rays and extreme ultraviolet radiation; Principles and Applications** by D. Attwood, Cambridge University Press, ISBN 0-521-65214-6, (1999)
- **Third Generation Hard X-ray Synchrotron Radiation Sources, *"Source properties, Optics and Experimental techniques",*** edited by D. Mills, Wiley & Sons, ISBN 0-471-31433-1, (2002)
- **Modern Developments in X-ray and Neutron Optics** edited by A. Erko, M. Idir, T. Krist, A.G. Michette, Springer, ISBN 978-3-540-74560-0

- **Selected Publications**

- X-ray Interactions with Matter
- J. Kirz et al. Quarterly Reviews of Biophysics, **28**:33130 (1995)
- Best focus experiments
- H. Mimura *et al. Applied Physics Letters*, **90**:051903 (2007)
- W. Chao *et al., Nature*, **435**:1210 (2005)
- Ultimate Resolution Theory
- H. Kang *et al., Physical Review Letters*, **96**:127401 (2006)
- C. Schroer *et al., Physical Review Letters*, **94**:054802 (2005)