X-ray optics for imaging

School on X-ray Imaging Techniques at the ESRF

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Acknowledgements for slides:
J. Susini, O. Hignette, U. Neuhaeusler, T. Weitkamp

Outline

Beam conditioning
- Monochromators

Wavefront sensors
- Diffractive gratings

High Resolution X-ray Imaging

- Setups: TXM - SXM - PXM
- X-ray lenses (diffractive - refractive - reflective) and examples

Beam Conditioning

Flat mirrors:

- Harmonic suppression
- Limited quality for imaging

Monochromators:

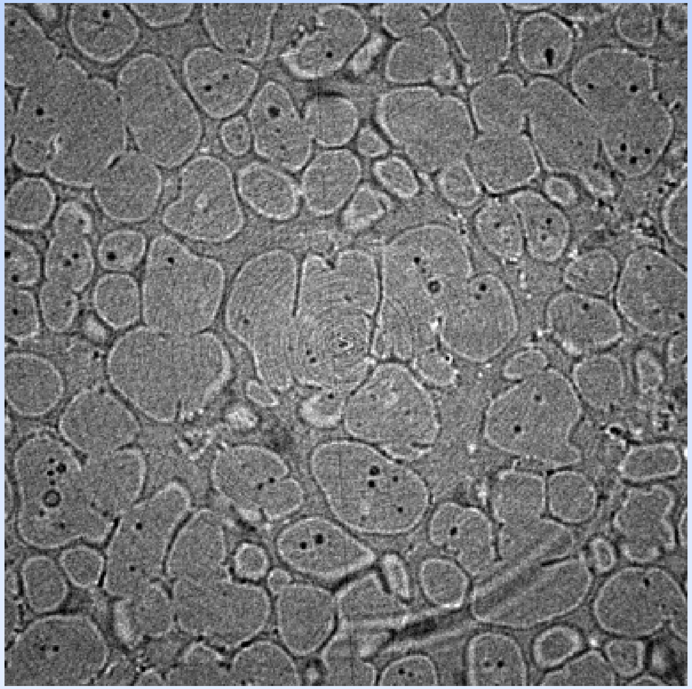
- Perfect crystals
  - nearly always Silicon
  - $\Delta E/E \approx 1.4\ 10^{-4}$
- Multilayer coated mirrors
  - high throughput monochromator
  - $\Delta E/E \approx 2\ 10^{-2}$
  - e.g. high spatial resolution imaging on ID19
- Filtered ‘white’ beam
  - $\Delta E/E \sim 1$
  - e.g. ultrafast tomography on ID15

Monochromator choice

Many X-ray imaging modes can accept a bandwidth larger than 10^{-4}

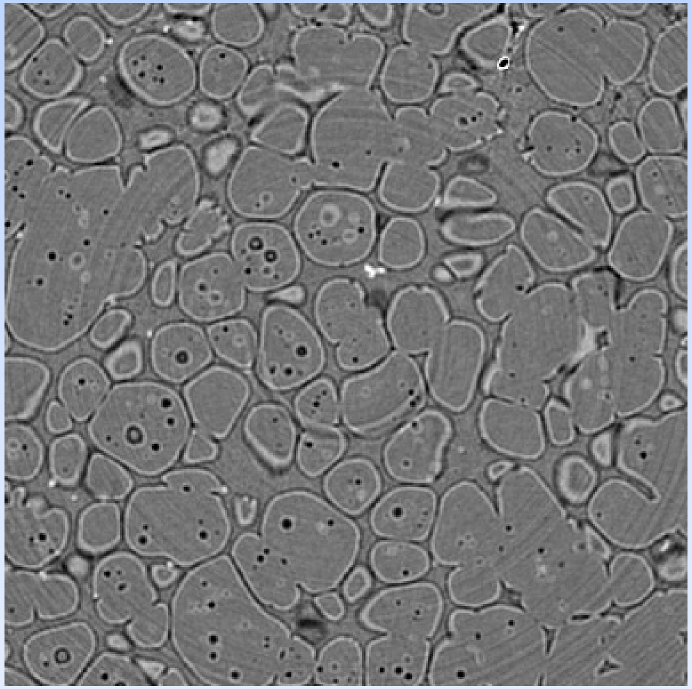
double Si crystal ($\Delta\lambda/\lambda \approx 1.4\ 10^{-4}$) or multilayer ($\Delta\lambda/\lambda \approx 2\ 10^{-2}$)

Double crystal monochromator



Scan-time ! 2 hours

Multilayer monochromator



Scan-time ! 9 minutes

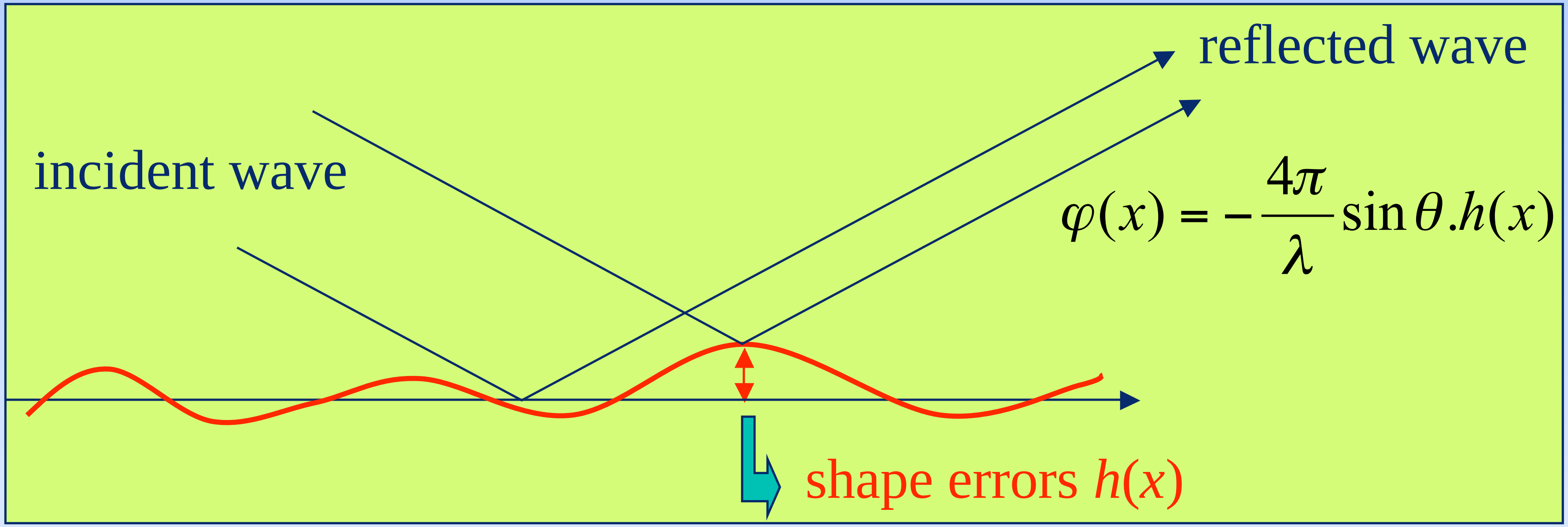
Sample: Al / Al-Si

Energy = 18 keV
D = 0.6 m
pixelsize = 2 µm

Gain > 100 , but …

Monochromator choice: multilayers

!! quality of the substrate !!



incident wave

reflected wave

$$\varphi(x) = -\frac{4\pi}{\lambda}\sin\theta . h(x)$$

shape errors *h*(*x*)

$$\varphi(x) = -\frac{4\pi}{\lambda}\sin\theta . h(x)$$

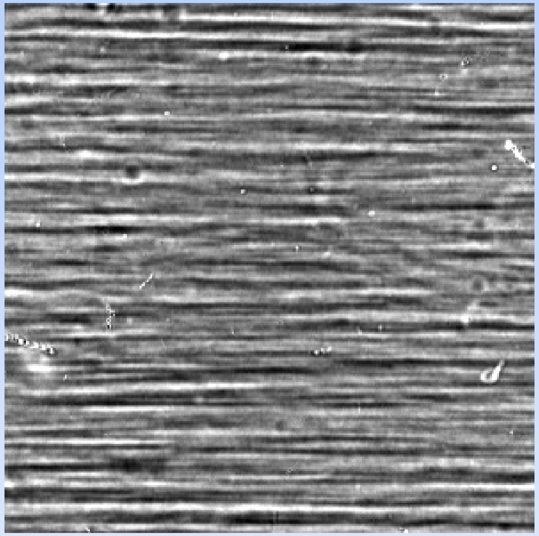
$$2d\sin\theta = \lambda$$

$$\varphi(x) = -2\pi\frac{h(x)}{d}$$

the shape errors should be much smaller than the layer period (0.01-10 mm^{-1})

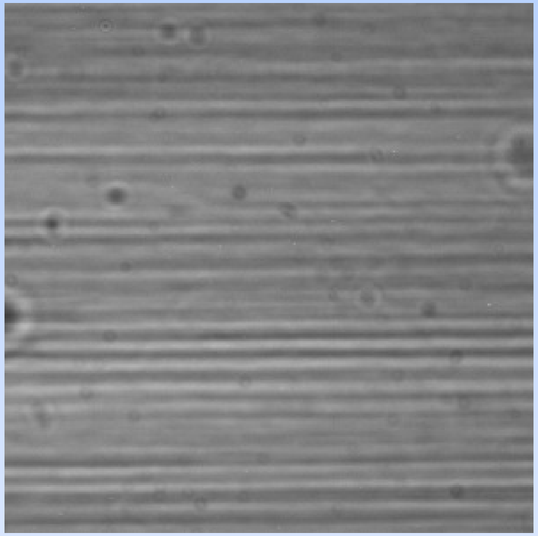
Influence of multilayer period

d = 2.6 nm



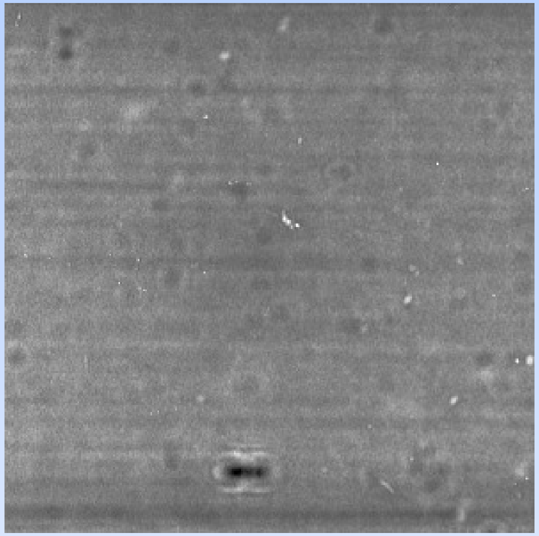
W/B_4C
100 periods

d = 4 nm



Ru/B_4C
65 periods

d = 6 nm



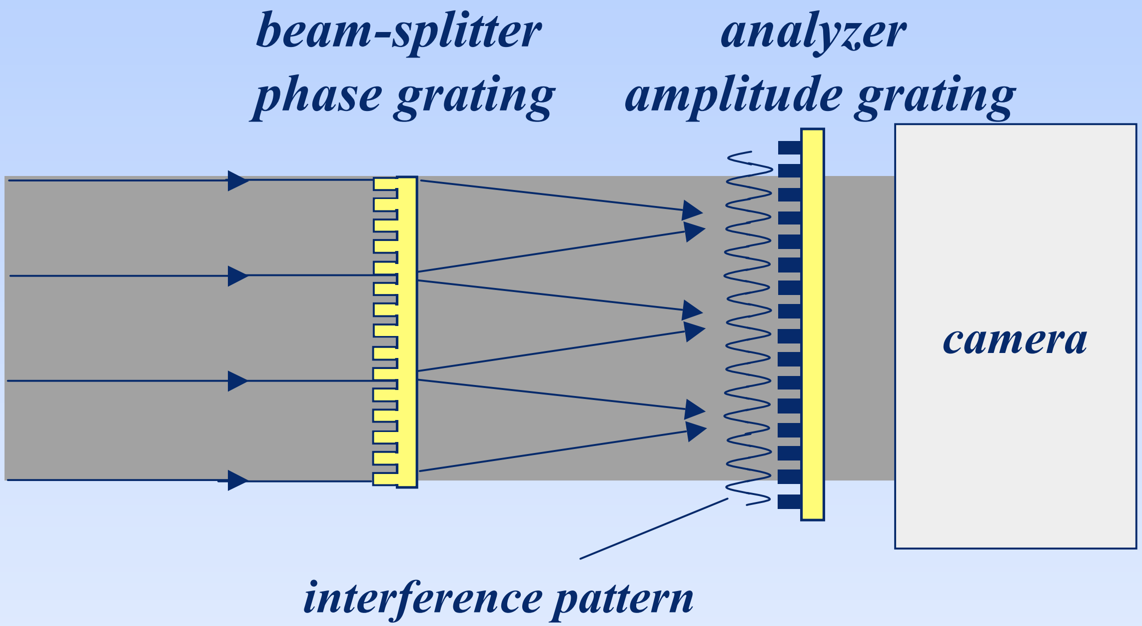
W/B_4C
20 periods

GO substrates
Distance *D* = 1.8 m
X-ray Energy ! 20 keV

100 µm

Wavefront Sensor

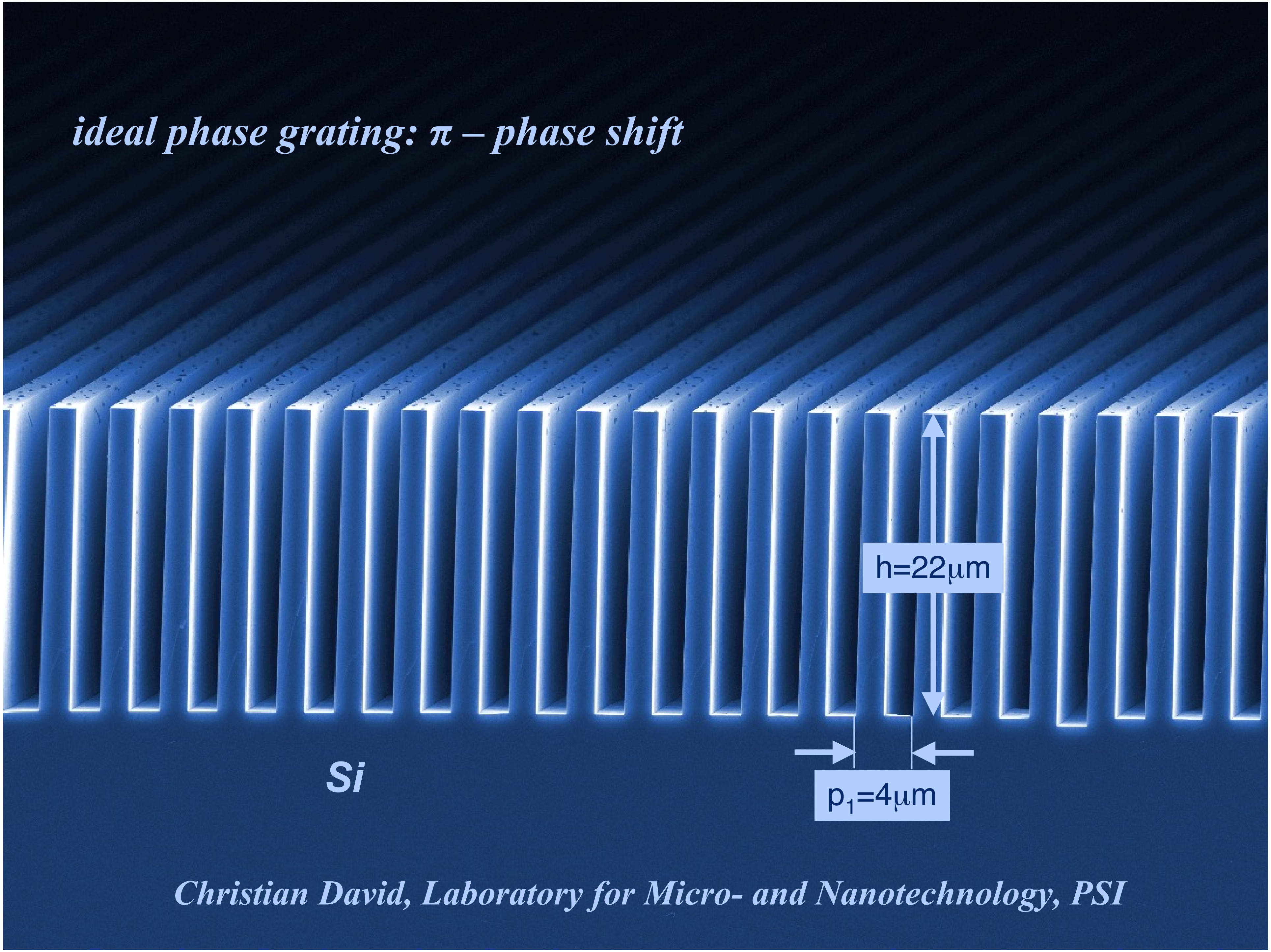
cf. Grating Based Phase Contrast Imaging



- ***phase grating as beam splitter***
- ***absorption grating as transmission mask***
- ***x-ray wavelength λ ~ 0.1 nm, grating periods ~ 2-4 μm***

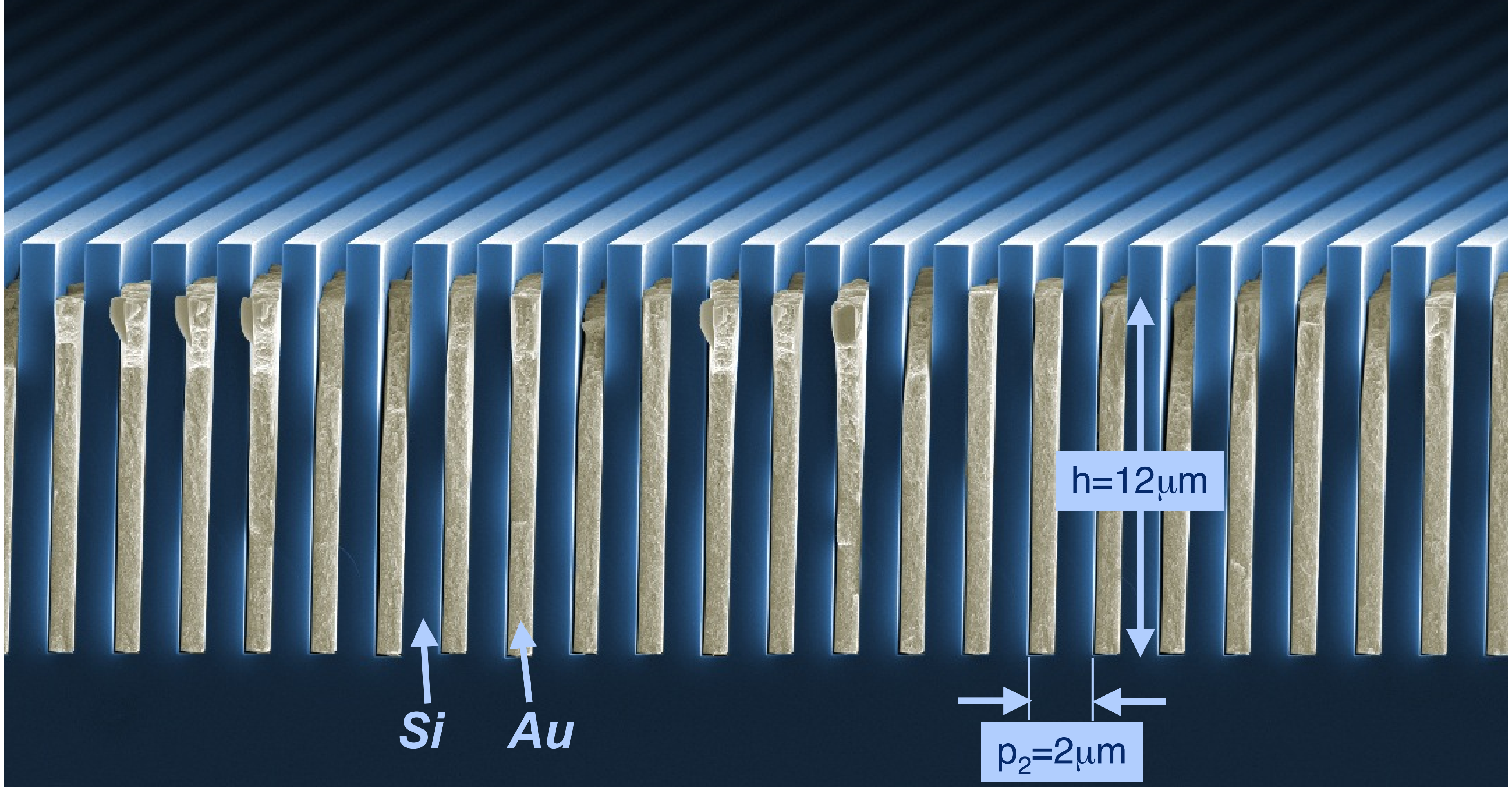
T. Weitkamp, F. Pfeiffer, Ch. David et al

ideal phase grating: π – phase shift



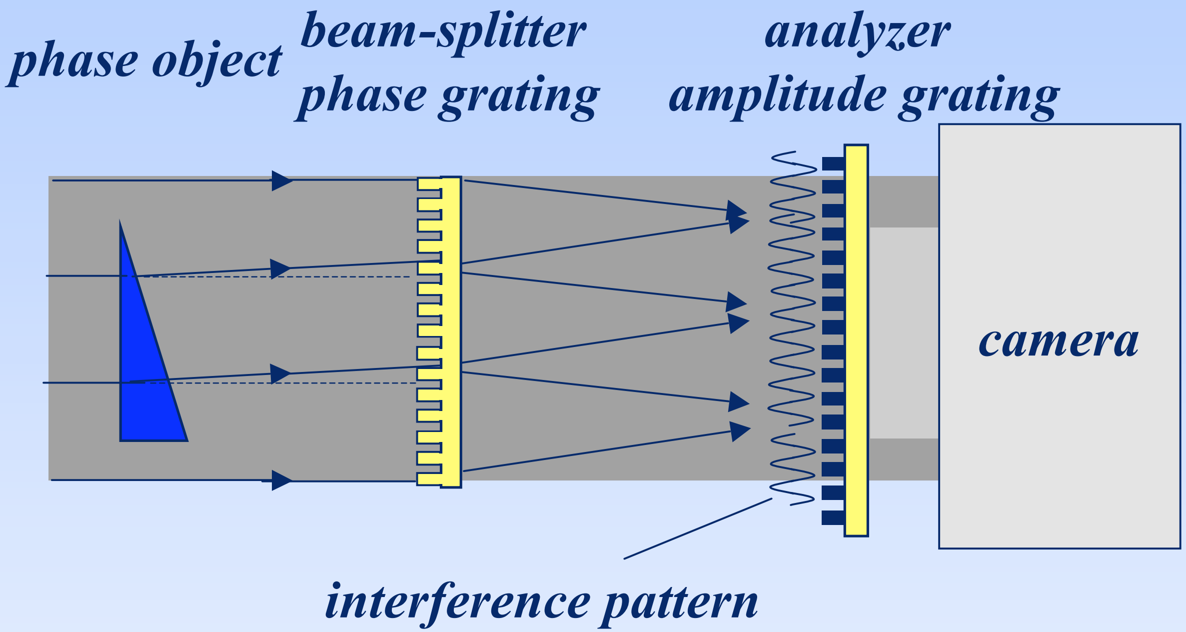
Christian David, Laboratory for Micro- and Nanotechnology, PSI

ideal analyzer grating: strongly absorbing lines



Christian David, Laboratory for Micro- and Nanotechnology, PSI

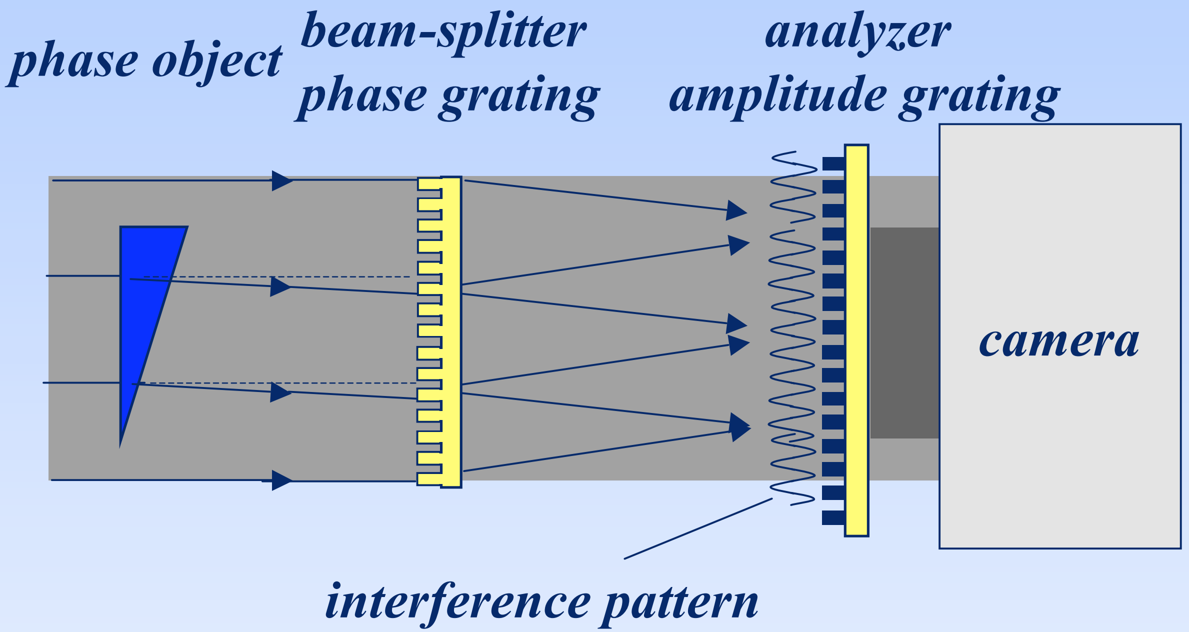
Wavefront Sensor



- *phase grating as beam splitter*
- *absorption grating as transmission mask*
- *x-ray wavelength $\lambda \sim 0.1$ nm, grating periods ~ 2-4 µm*
- *phase gradient*

T. Weitkamp, F. Pfeiffer, Ch. David et al

Wavefront Sensor



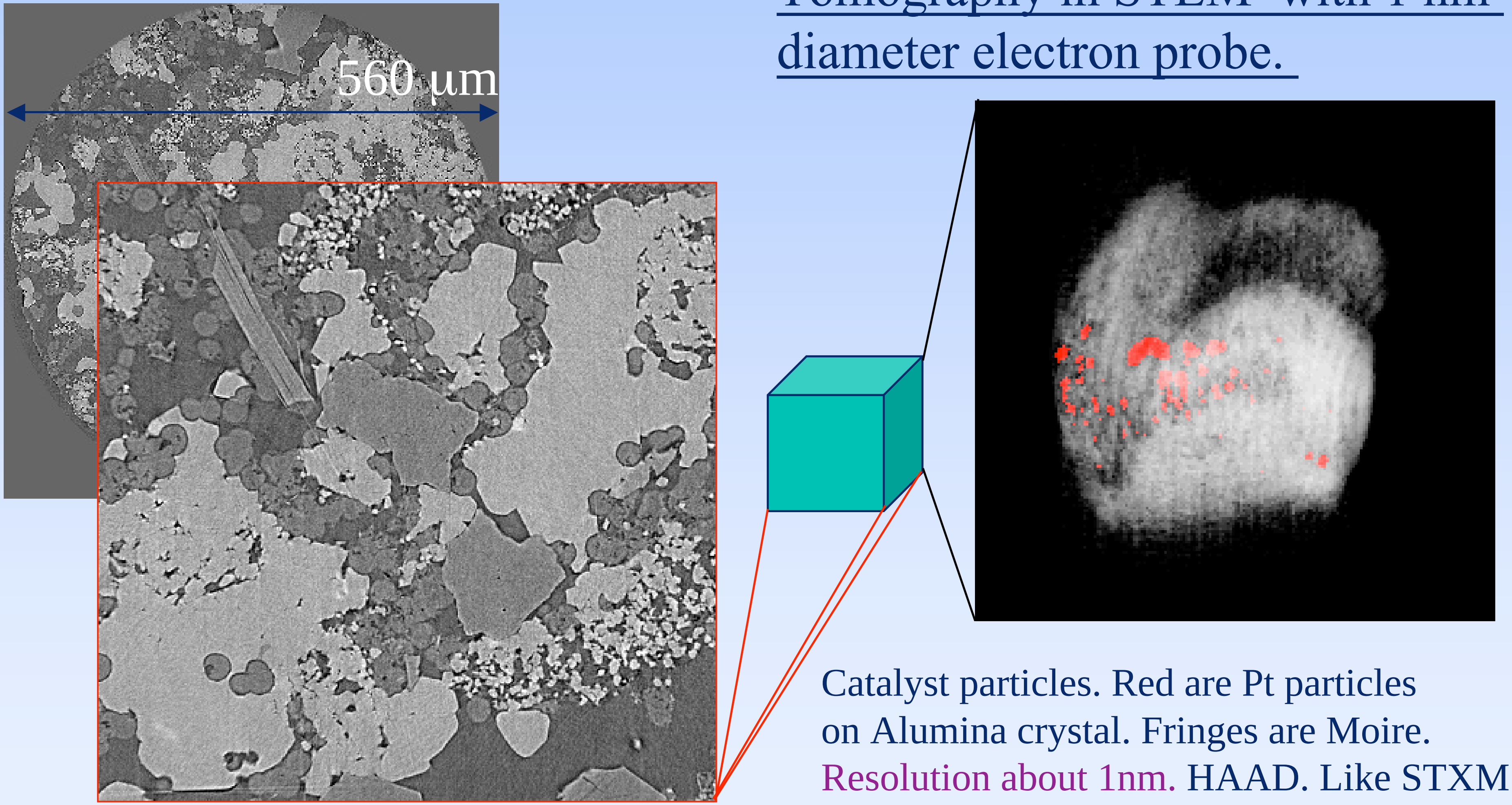
- *phase grating as beam splitter*
- *absorption grating as transmission mask*
- *x-ray wavelength $\lambda \sim 0.1$ nm, grating periods ~ 2-4 µm*
- *phase gradient*

T. Weitkamp, F. Pfeiffer, Ch. David et al

The resolution gap

X-ray micro-tomography

Tomography in STEM with 1 nm diameter electron probe.



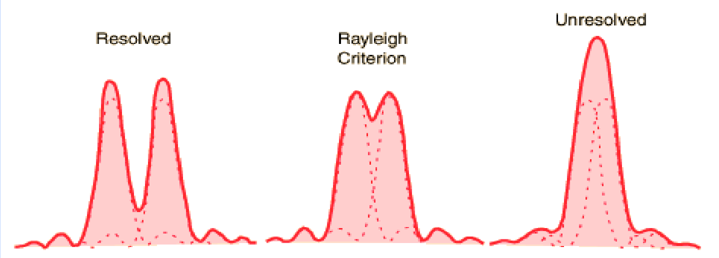
1 voxel = 280 nm

O Rozenbaum Univ. d'Orléans/ISTO, P. Cloetens

Catalyst particles. Red are Pt particles on Alumina crystal. Fringes are Moire. Resolution about 1nm. HAAD. Like STXM. 100 keV. P.Midgely et al, 2002

Spatial Resolution (1)

Rayleigh Criterion for resolving two adjacent objects



Point spread function - Transfer function

$R_{\text{Rayleigh}} = c\,\lambda/NA$

Noise-less world

Rose Criterion: influence of noise on spatial resolution

Photon statistics and/or dose limit the obtainable resolution

SNR > 5 for detection

e.g. tomography (Flannery 87)

$$N_{\text{Phot}}^{\text{tot}} \propto \left(\frac{D}{R}\right)^4 \frac{\exp(\mu D)}{[\mu D(\sigma/\mu)]^2}$$

With sample diameter D constant: if R ↓ then $N_{\mathbf{Phot}}$ ↑ as $(1/R)^4$

Pixel / Voxel size

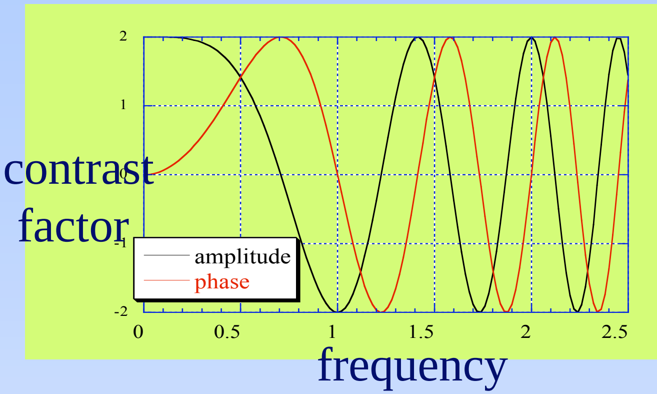
Often stated as ‘resolution’ in tomography!

Correct sampling requires: $q_{\max} < q_{Nyquist} = \dfrac{1}{2\,pixelsize}$

Spatial Resolution (2)

Information Limit:

Highest frequency containing (scrambled) phase information



Detection Limit:

smallest object that can be detected

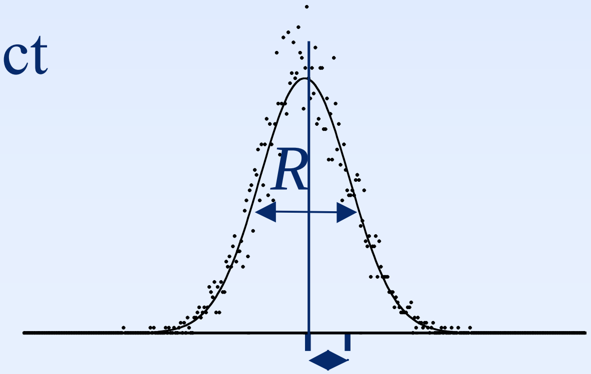
will depend on contrast and noise

can be << R_{Rayleigh} especially using phase contrast

Precision:

e.g. on the position of an object

$$\text{Precision} \propto \frac{R}{\sqrt{N_{\text{phot}}}}$$



High Resolution X-ray Imaging

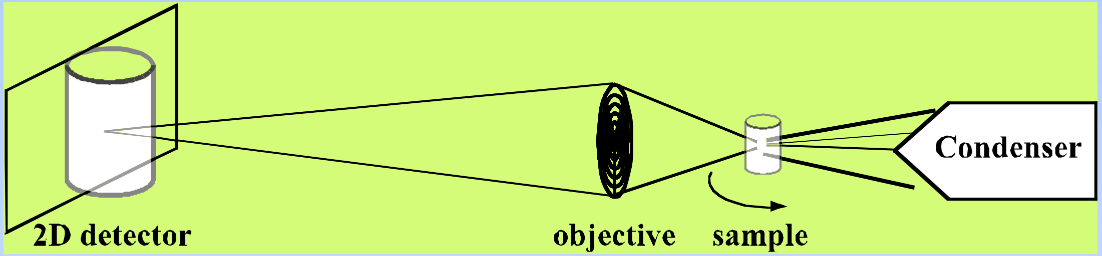
Full-field microscope:

Structure

Dose inefficient, fast

Absorption + phase

TXM



Scanning microscope:

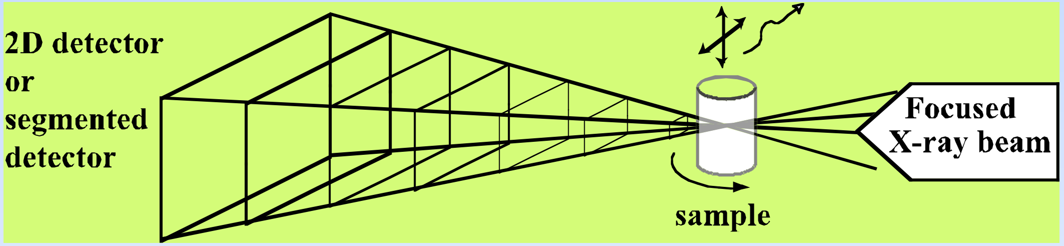
Nano-analysis

Slow

Rich, trace elements

Phase contrast

SXM



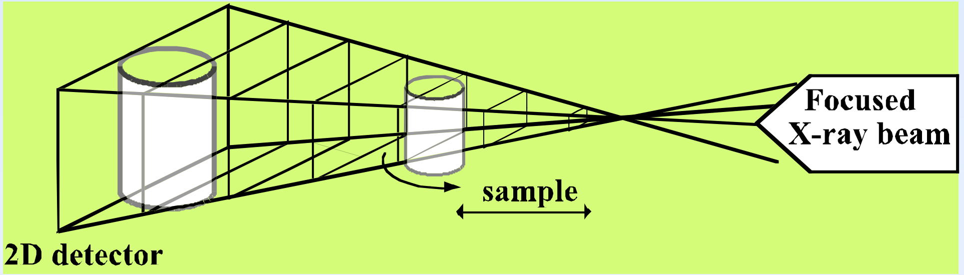
Projection Microscopy:

Structure

Dose efficient, fast

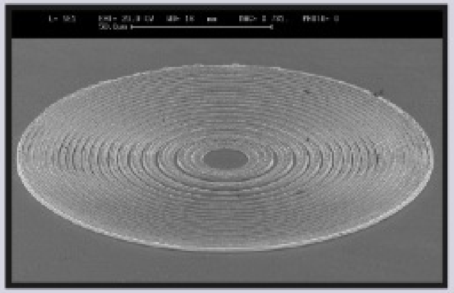
Phase contrast

PXM



X-ray ‘lenses’

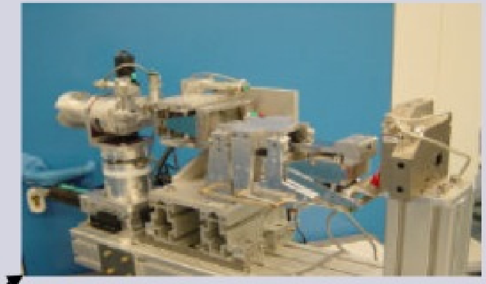
Diffractive lenses

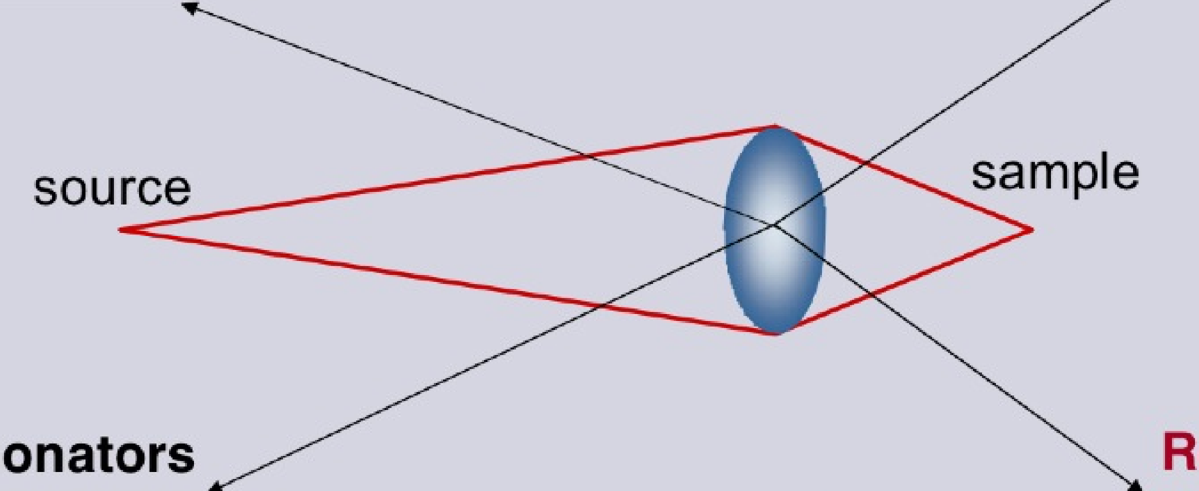


- *Fresnel zone-plate*
- *Bragg-Fresnel*
- *Crystals*
- *Multilayers*

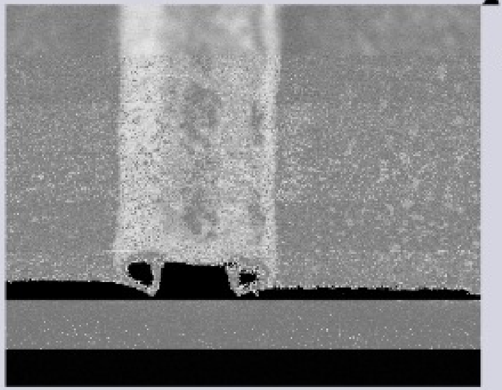
X-ray Reflectors

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





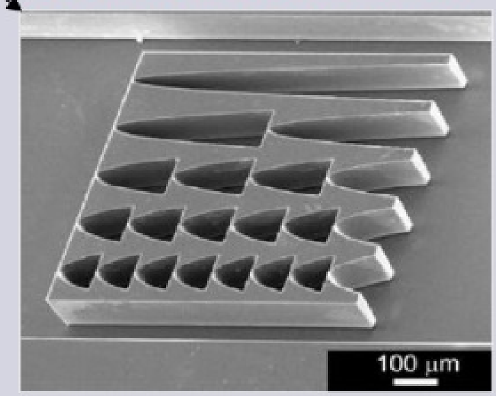
X-ray resonators



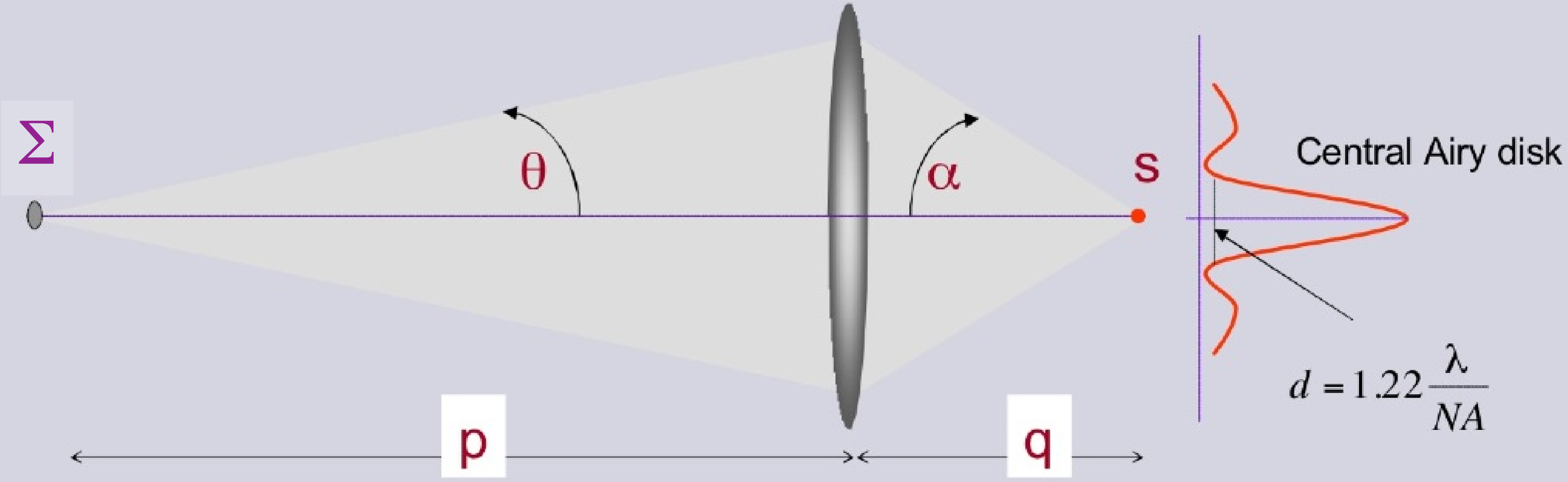
- *Waveguides*

Refractive lenses

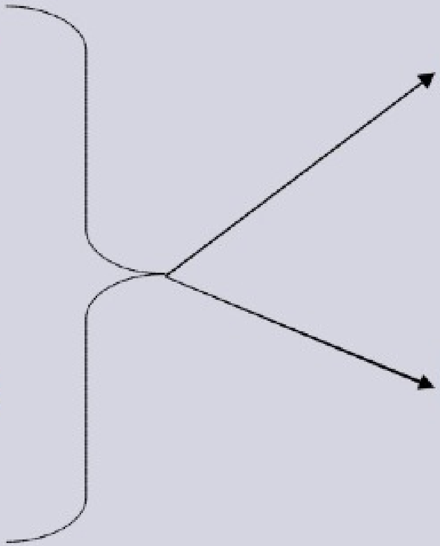
- *Compound refractive lenses*



Limits in X-ray focusing



$$d = 1.22\frac{\lambda}{NA}$$



Thin lens equation

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

Numerical aperture

$$NA = \sin\alpha$$

Geometrical demagnification:

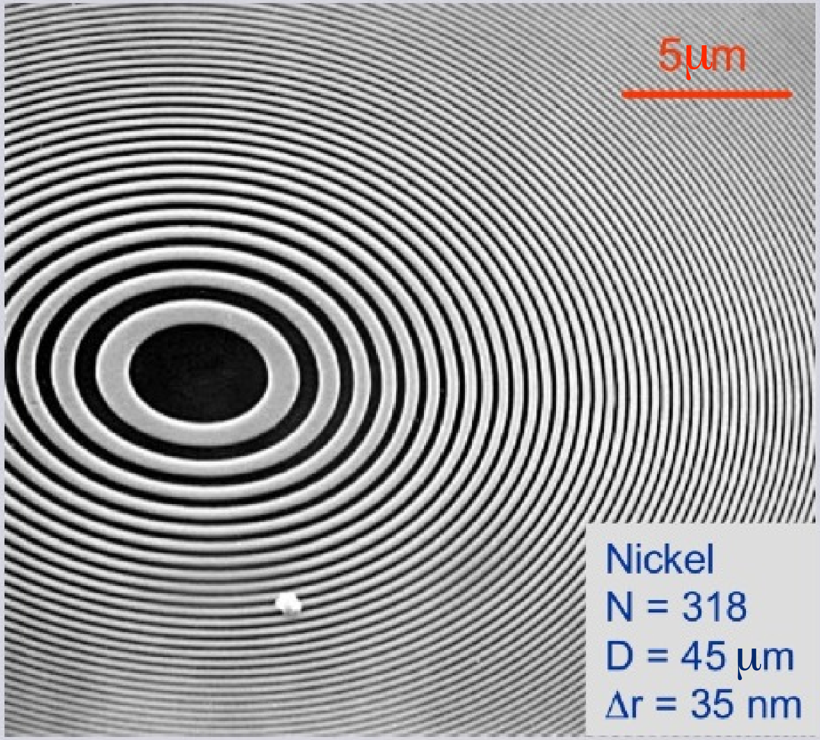
$$s_G = \Sigma \times \frac{q}{p}$$

Diffraction limited focusing if $\Sigma \times \theta = \frac{\lambda}{4\pi}$

$$s_{DL} = c\,\frac{\lambda}{NA}$$

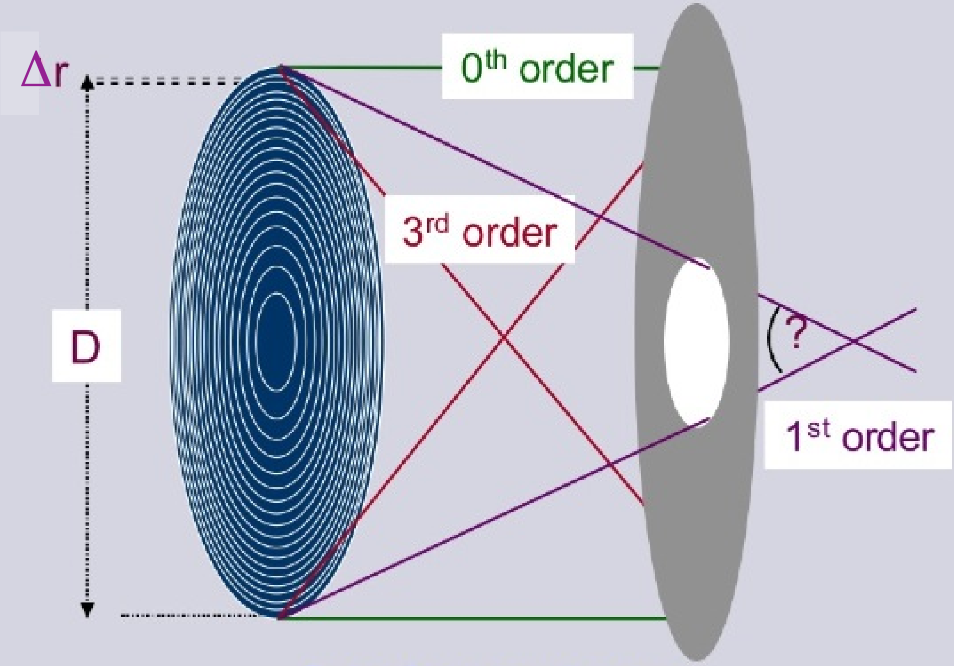
+ Optics imperfections

Fresnel zone plates



Nickel
N = 318
D = 45 µm
Δr = 35 nm

Diffractive focusing elements made up of concentric circular zones:



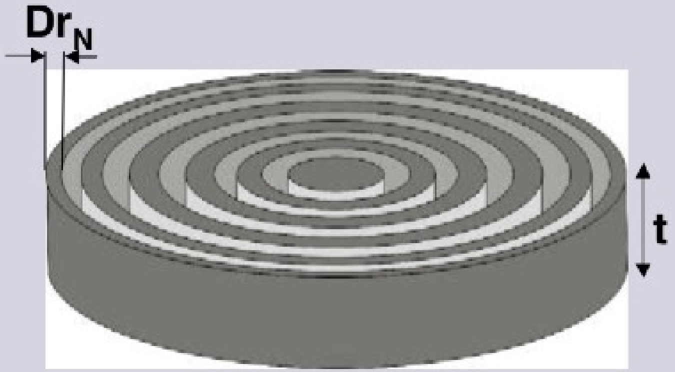
Order Selecting Aperture

- Resolution: $\delta_m = 1.22\,\frac{\Delta r}{m}$

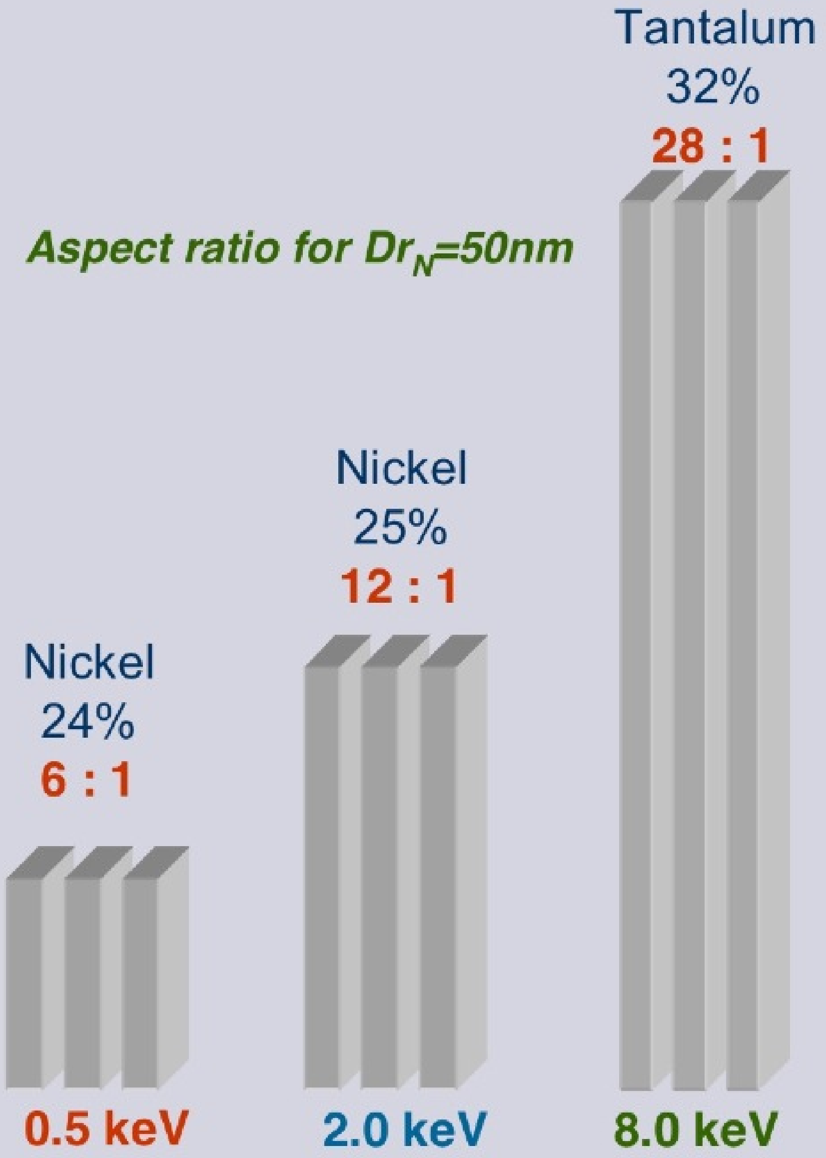
- Focal length: $f_m = \frac{D\,\Delta r}{m\,\lambda}$

- Depth of focus: $DOF = \pm\,\frac{2\,\Delta r^2}{m\,\lambda}$

Fresnel zone plates

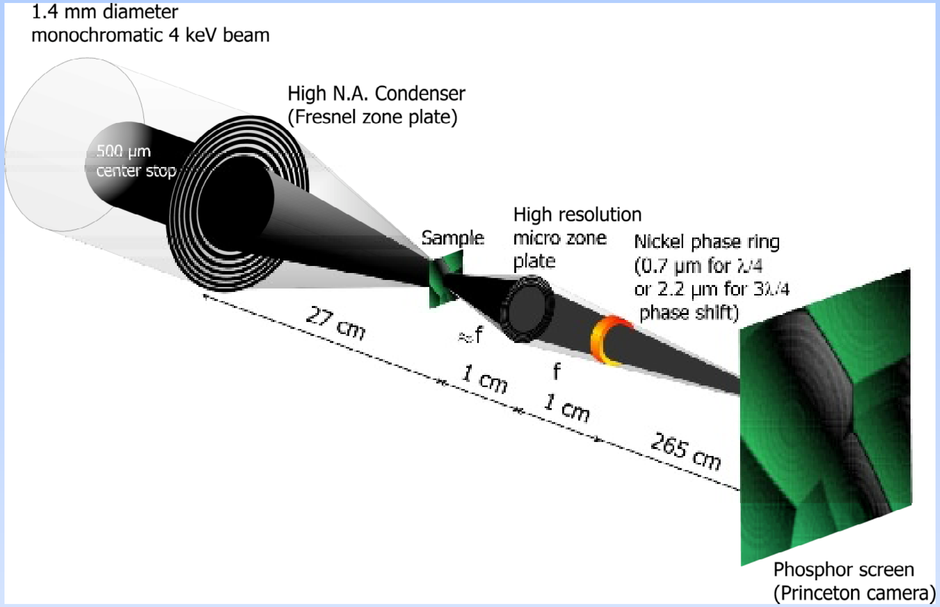


Material	t (μm)	ε (%)
E=0.5keV		
Ge	0.28	16
Ni	0.25	24
E=2.0keV		
Ni	0.60	25
Au	0.45	24
E=8.0keV		
Ta	1.70	32
W	1.50	33

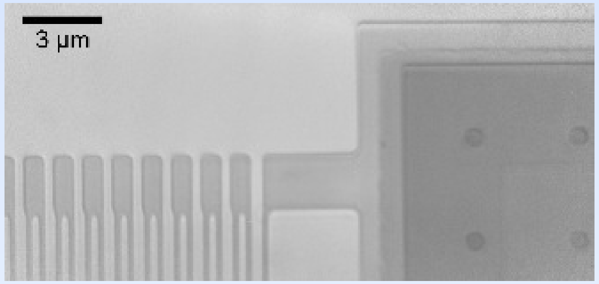


Towards sliced ML in transmission (J Maser et al)

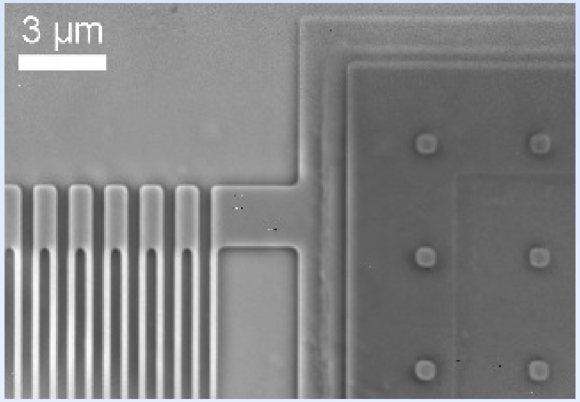
Zernike Phase Contrast Microscopy at ID21



Full field microscope: FZP's
60 nm spatial resolution at 4 keV
Zernike PhC with phase ring



Absorption Contrast

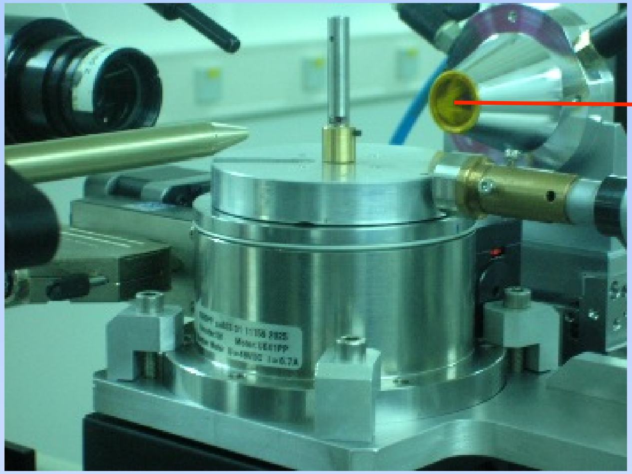


Zernike Phase Contrast

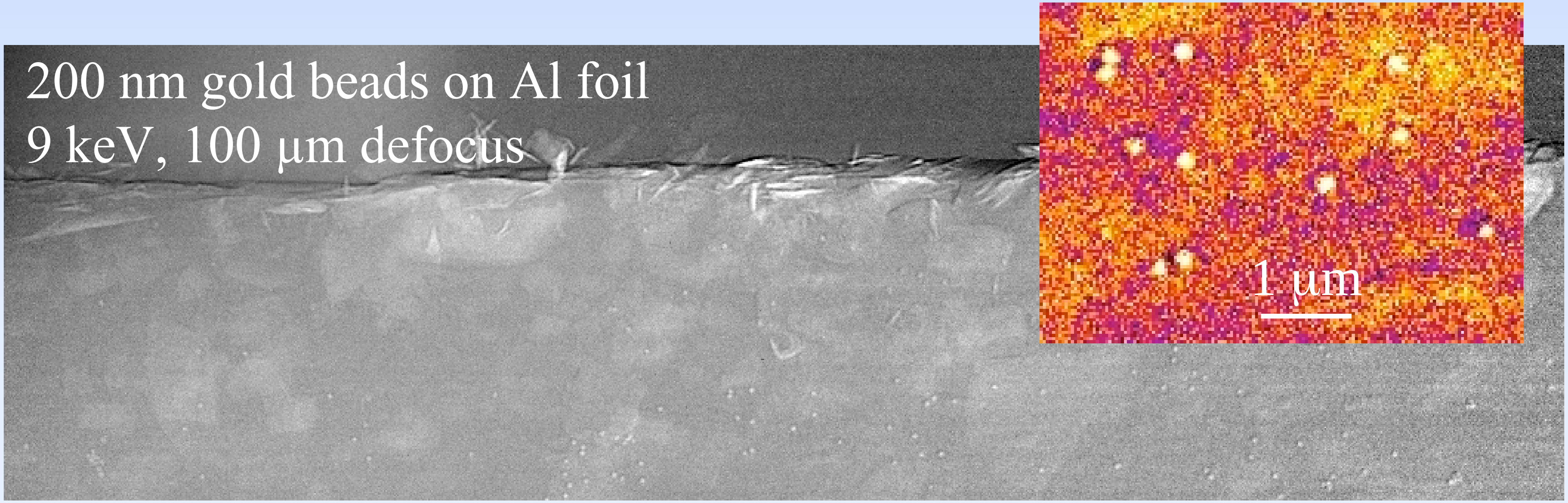
Serpentine resistor
from Sematech
225 nm Cu lines

U. Neuhaeusler, W. Ludwig, ID21; G. Schneider, D.Hambach

Full-field Microscopy



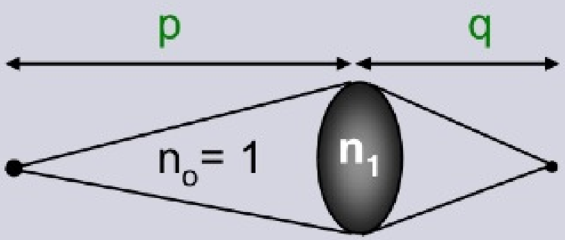
Fresnel Zone Plate
50 nm outermost zone width
34 nm pixelsize @ 9 keV
58 mm focal distance



200 nm gold beads on Al foil
9 keV, 100 µm defocus

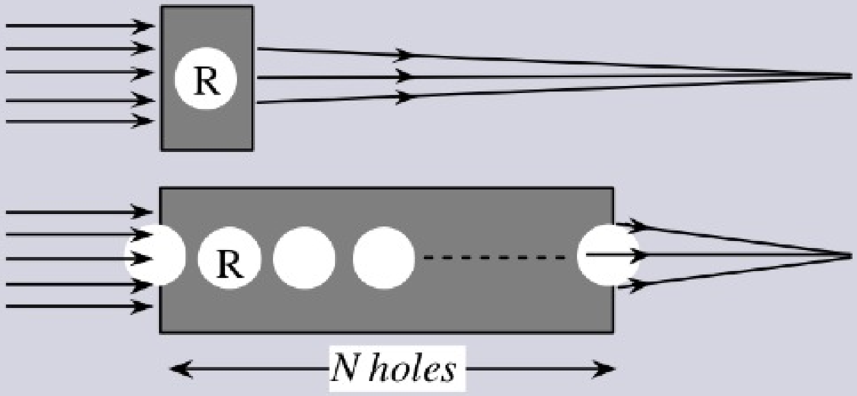
W Ludwig, G Johnson, P Cloetens

Compound refractive lenses



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

Gaussian lens formula : $\frac{1}{F} = \frac{1}{p} + \frac{1}{q}$



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

$$\boxed{\frac{1}{F} = N\frac{2\delta}{R}} \propto \lambda^2$$

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

$\Downarrow$

$n_1 < 1$: 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

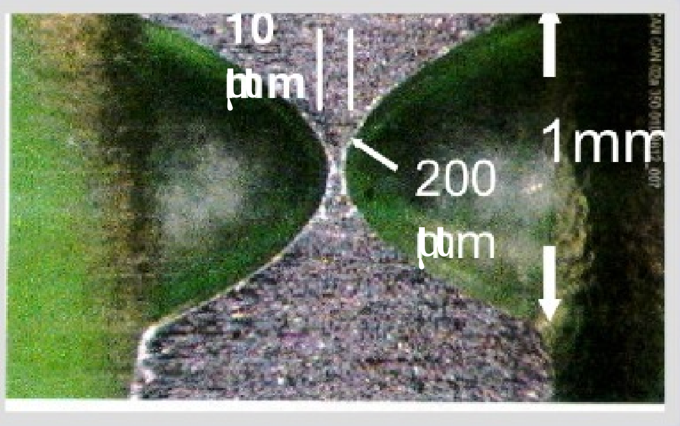
Advantages
- simplicity and low cost
- low sensitivity to heat load

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

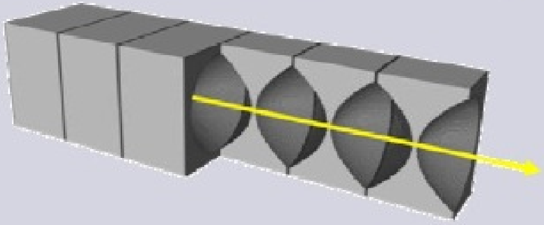
A. Snigirev et al. Nature, 384 (1996)

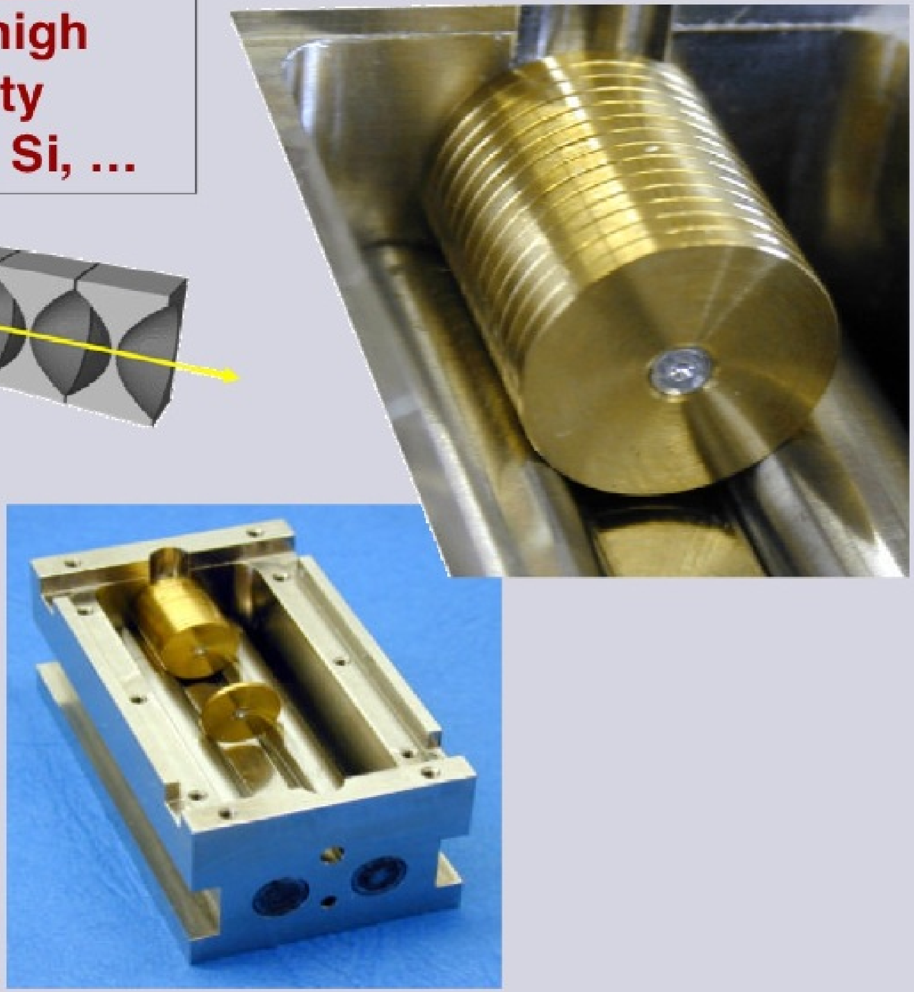
http://www.institut2b.physik.rwth-aachen.de/xray/applets/crlcalc.html

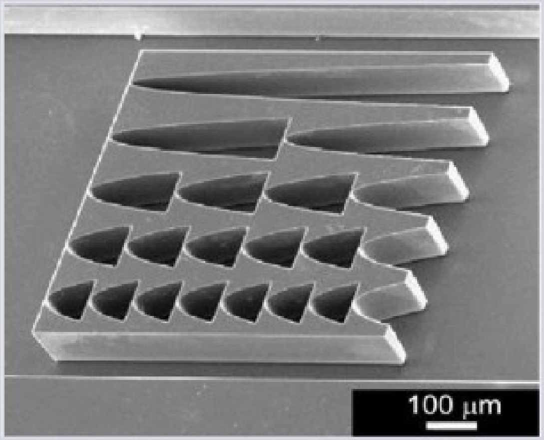
Compound refractive lenses



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



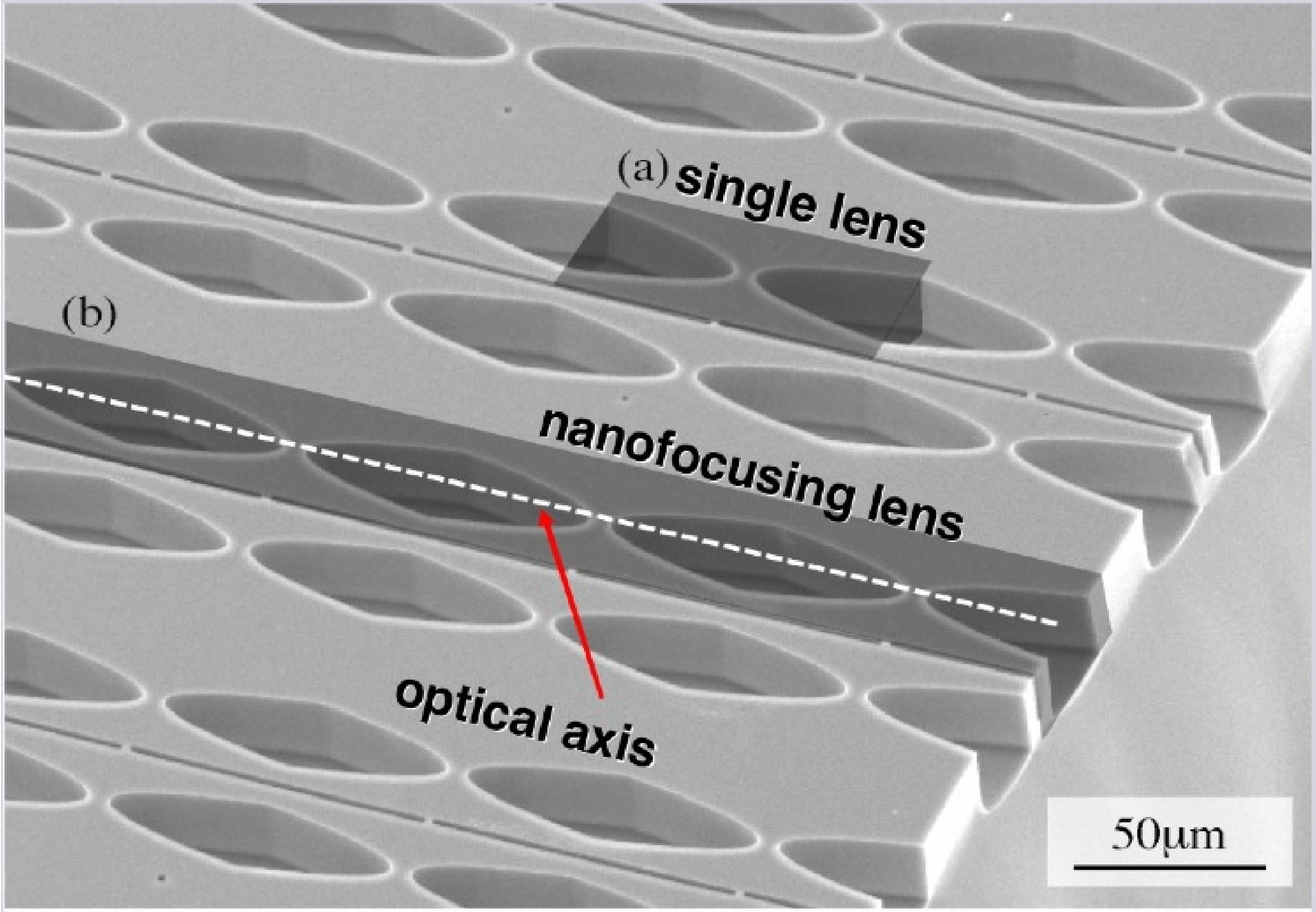




C. David et al.
PSI, Villigen, Switzerland

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

Compound refractive lenses



extreme curvature:

R = 1μm - 3μm

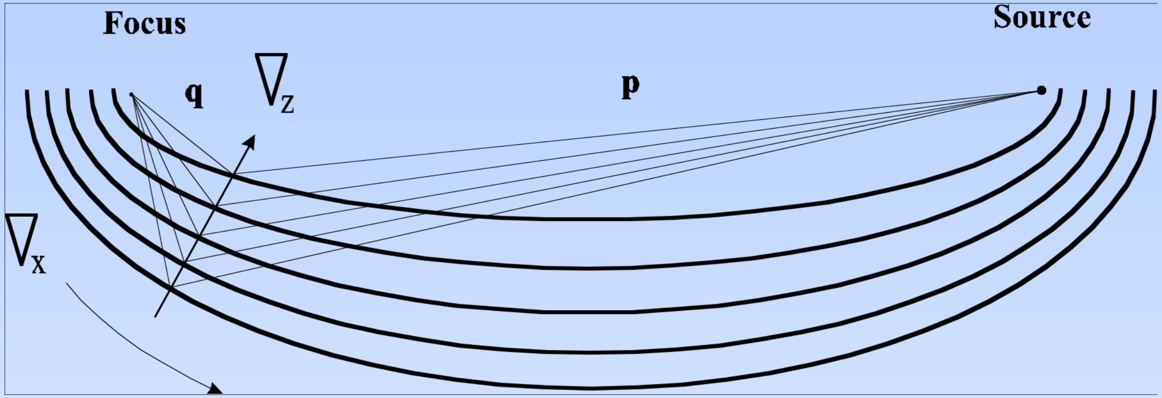
N = 50 - 100

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

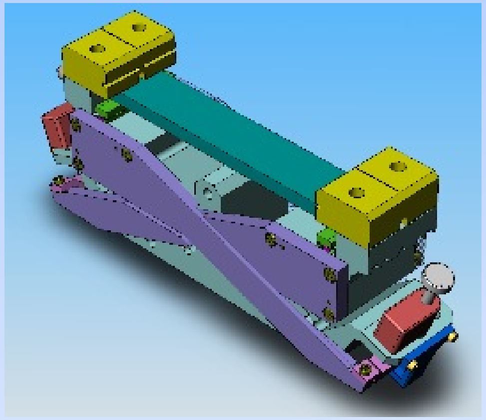
C. Schroer *et al*, *Applied Physics Letters*, 82(9), 2003

Bent graded multilayers in KB geometry

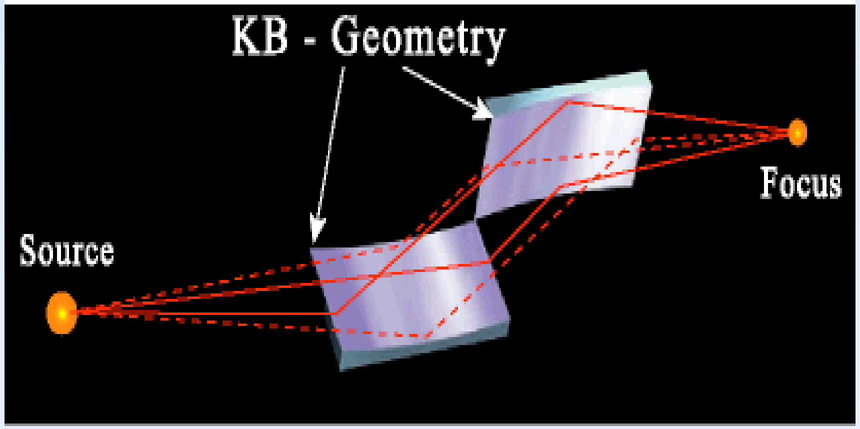
Bent graded multilayer



Reflective Optics



KB-geometry



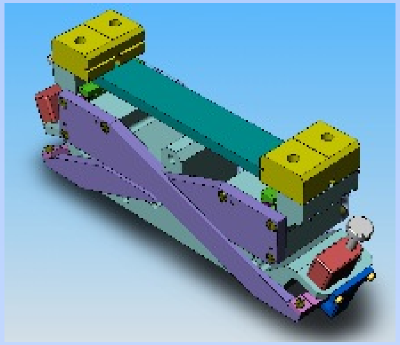
Kirkpatrick-Baez focusing

O. Hignette
Ch. Morawe

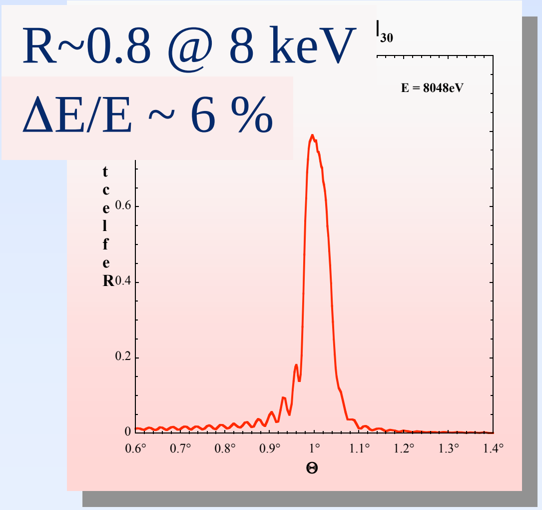
Industrial partners

- WavePrecision (USA)
- Winlight (F)
- SESO (F)
- ZEISS (D)

Optics
Group
ESRF



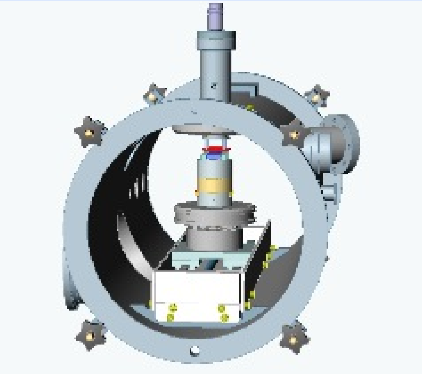
Opto-mechanical studies
Bender design
Technical Services



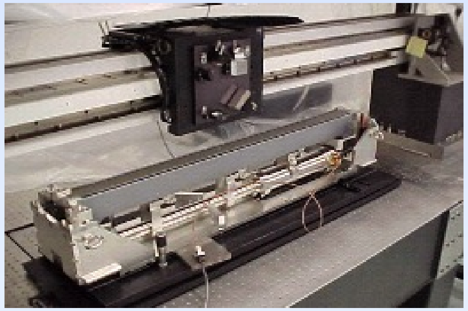
R~0.8 @ 8 keV

ΔE/E ~ 6 %

ESRF Multilayer Facility



Finishing Method



ESRF Metrology lab

Bent graded multilayers in KB geometry

Advantages

Multilayer Efficiency
  reflectivity towards 1
‘Achromatic’
  large bandwidth possible (6-7 %)
  scan energy easily
Large *NA*
Optics can be tuned to source geometry / actual wavefront

Drawbacks

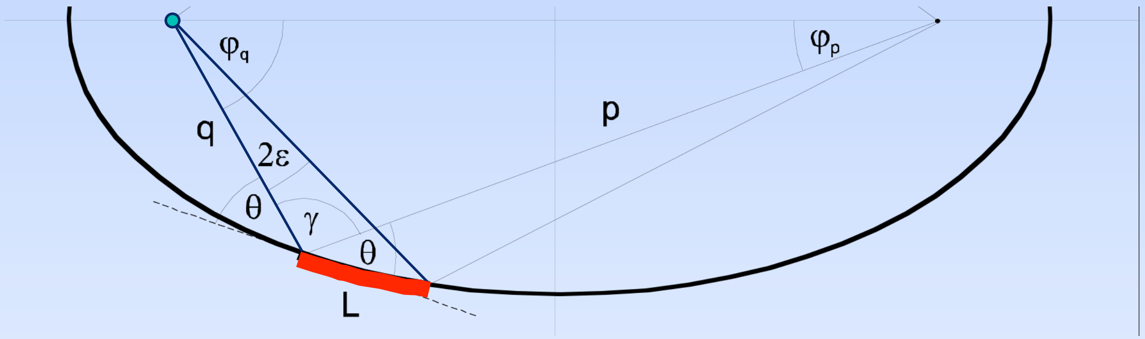
Mirror quality!
Not ‘install and go’: more for dedicated end-stations
(Not in-line optics)
Angular sensitivity (temperature drift …)

Ultimate limits in KB focusing

- Source size: ‘long’ beamline, secondary source, X-FEL
- Mirror quality: technological issue, finishing method (cf. Osaka)

- Multilayer: are volume diffraction effects a limitation?
- Diffraction

$$s_{FWHM} = 0.44 \cdot \frac{\lambda}{NA}$$



Mirror (critical angle)

$$s \approx \frac{1.76 \cdot \lambda}{\sqrt{2 \cdot \delta}} = 1.76\sqrt{\frac{\pi}{r_0 \cdot N \cdot Z}}$$

Pt: s ~ 25 nm

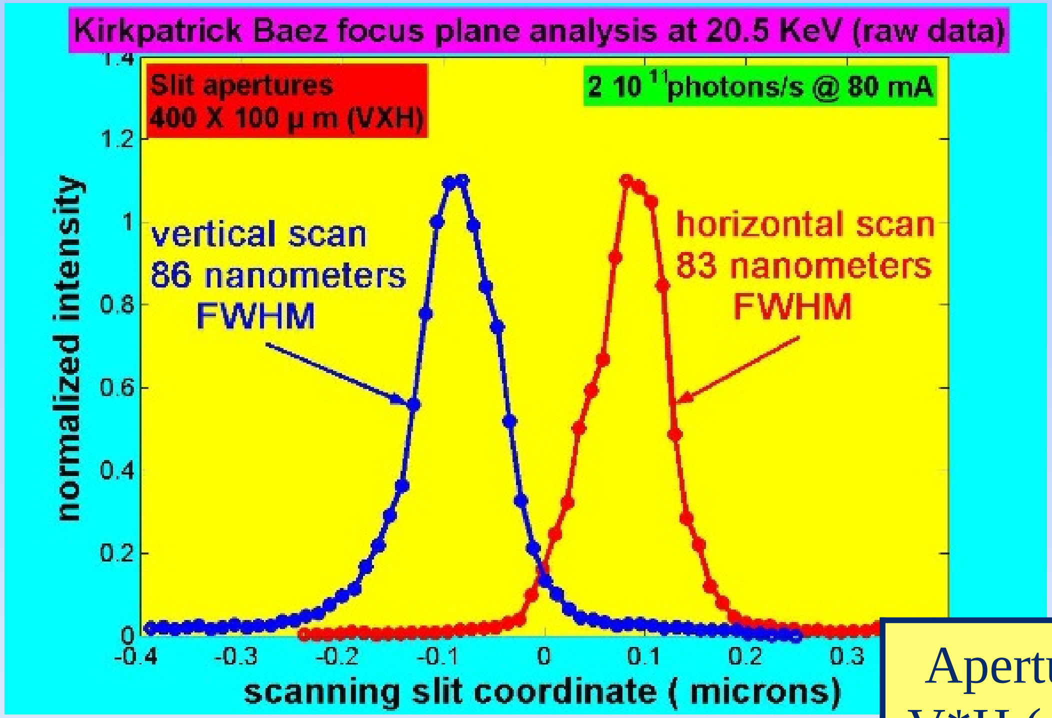
Multilayer (d-spacing Λ, gradient)

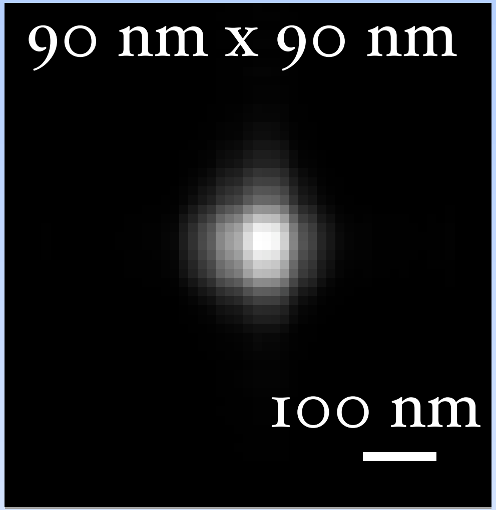
$$s \approx \frac{1.76\, q\, \Lambda}{L}$$

$\Lambda = 2$ nm $\Rightarrow$ s ~ 4 nm

Mainly technological limitations

KB Focusing





Aperture V*H (μm)	Spot fwhm V*H (nm)	Flux ($\Delta E/E=10^{-2}$) ph/s @ 80 mA
200 x 50	118 x 109	$5\ 10^{10}$
400 x 100	86 x 83	$2\ 10^{11}$
600 x 160	116 x 90	$4.5\ 10^{11}$

X-ray energy: 20.5 keV

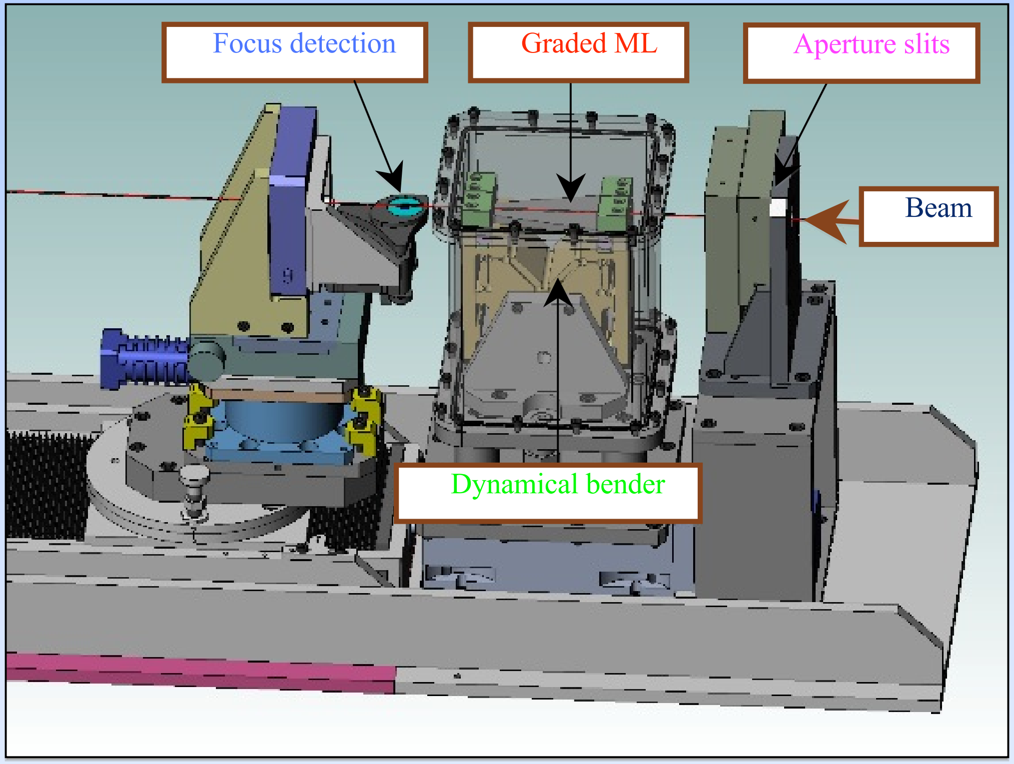
Distance source-KB *p* = 150 m

Gain > $3\ 10^{6}$!!

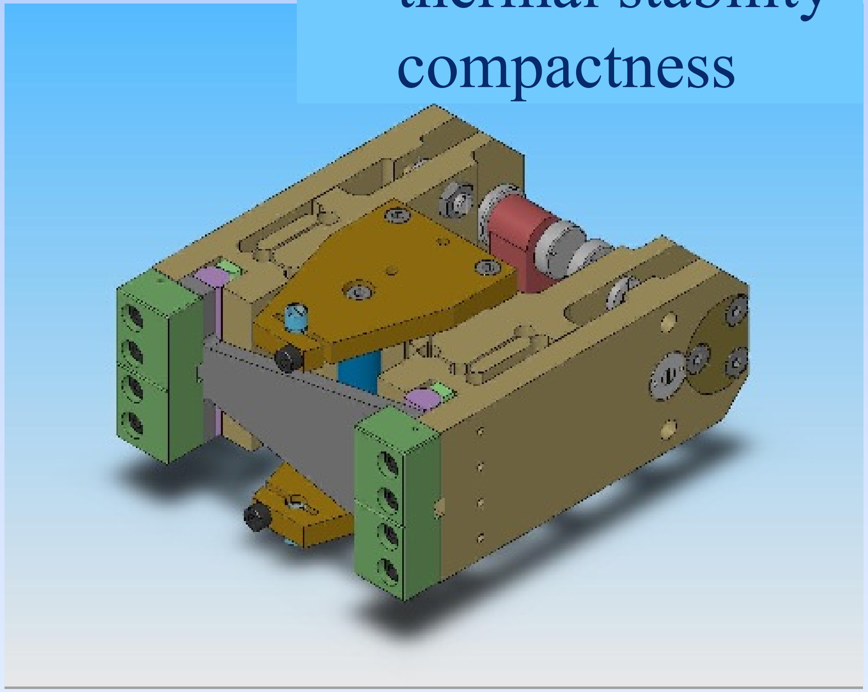
O Hignette, P Cloetens, G Rostaing, P Bernard, C Morawe Rev. Sci. Instr. 76, 063709-1 (2005)

Ultimate limits in KB focusing

Line focus measurement on ID19



Invar mirror bender:
thermal stability
compactness

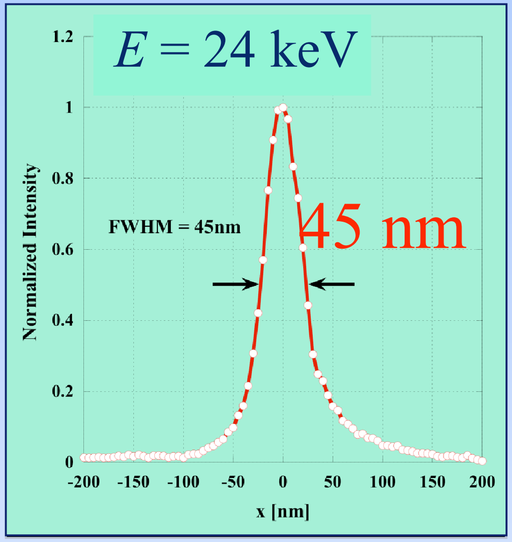


Energy 24 keV
Focal length 80 mm
Incidence angle 5.5 mrd
Vertical 25 µm FWHM source at 150 m

Ultimate limits in KB focusing

Line Focus

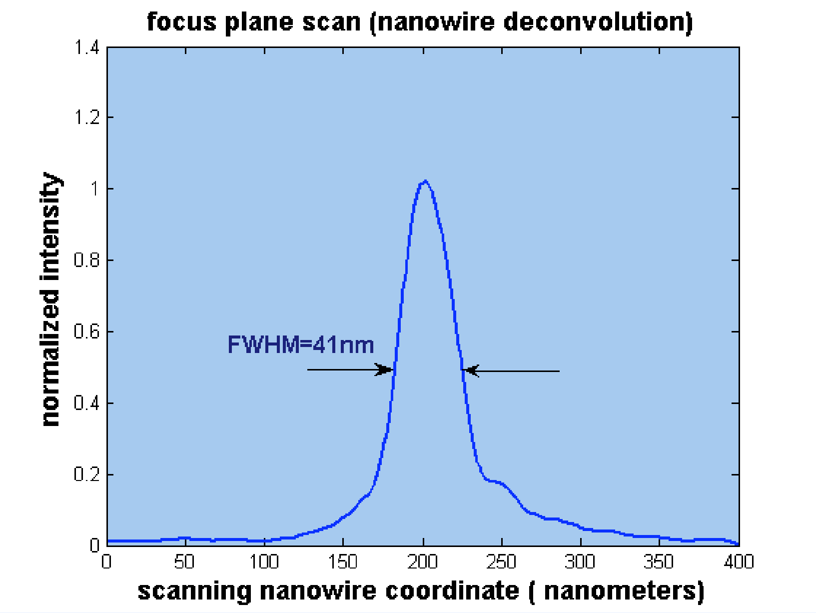
Line width: 45 nm



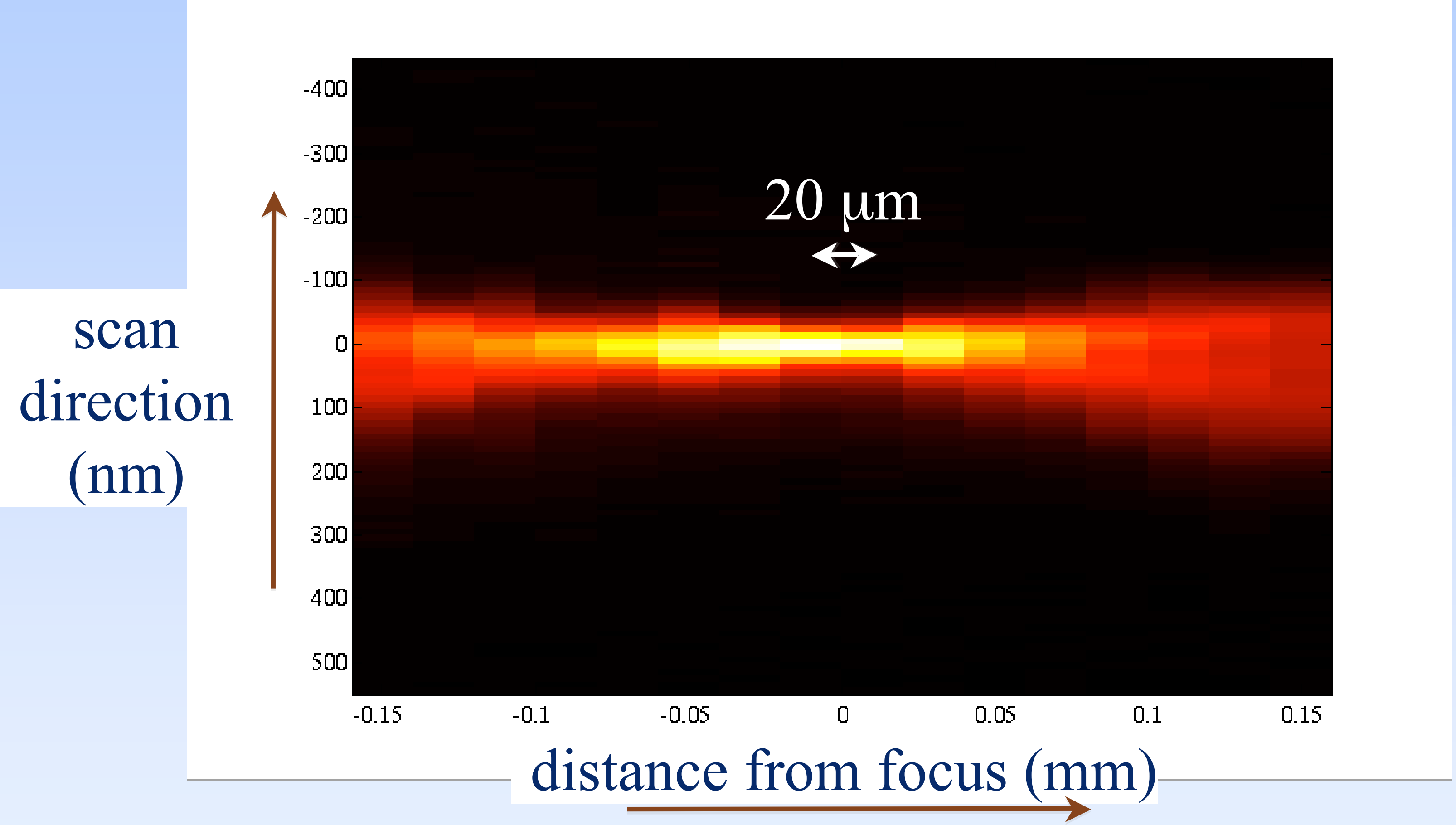
O. Hignette
P. Cloetens
Ch. Morawe
W. Ludwig
P. Bernard
R. Mokso

Deconvolution nano-wire

Line width: 41 nm

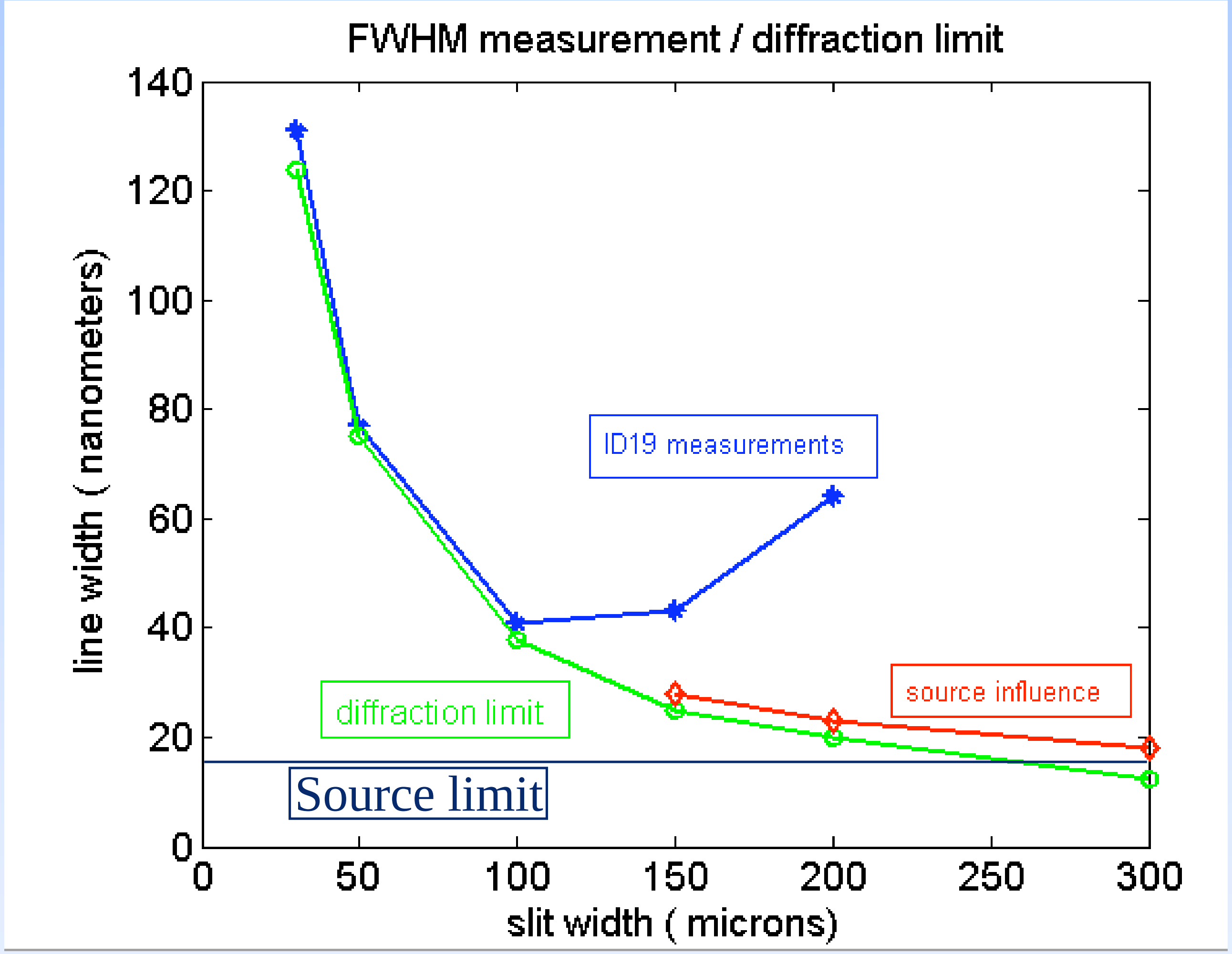


Depth of focus



Depth of focus is only ~ 20 µm for 40 nm focus @ 24 keV

Diffraction limit

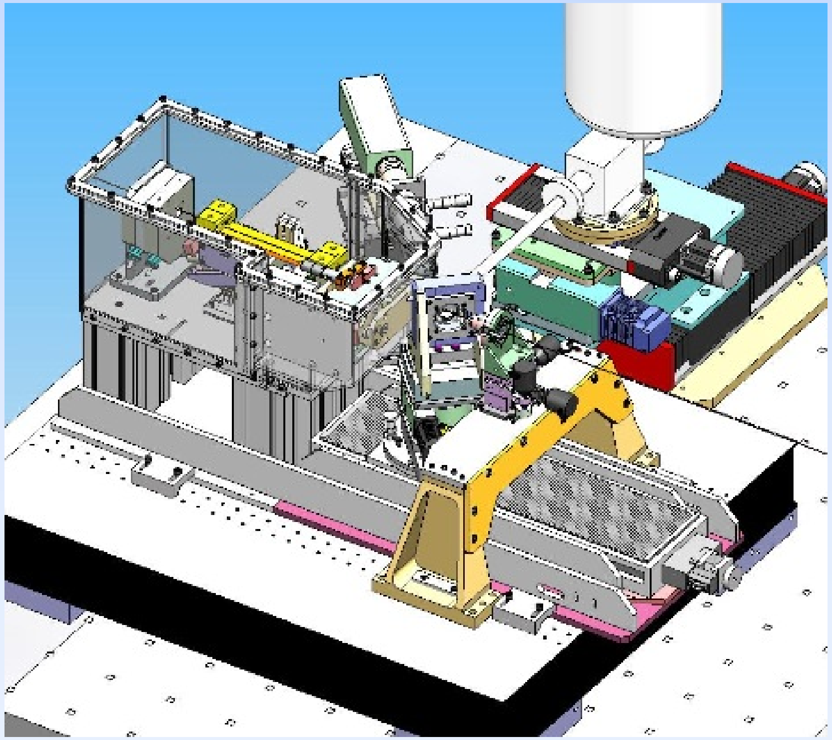


No volume diffraction effects visible at the 40 nm level

O. Hignette

Applications: Nano-Imaging Project

- Goal
  build an *end-station* (ID22NI)
  dedicated to *3D imaging*
  with routine ~ 50 nm spatial resolution
  combine micro-structure and micro-analysis



3 years project, started 2005
Pilot project

Nanochemical Imaging of Neurons

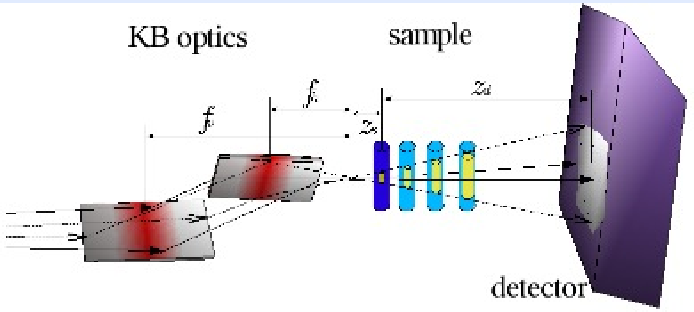
Role of iron in Parkinson's disease

Fe accumulates in
ultrastructures outside the nucleus

ID22NI

R Ortega, G Devès, A Carmona, CNAB, CNRS, Gradignan
S Bohic, P Cloetens

Zoom Tomography



Inside ϕ = 1 mm sample → **local tomography!**

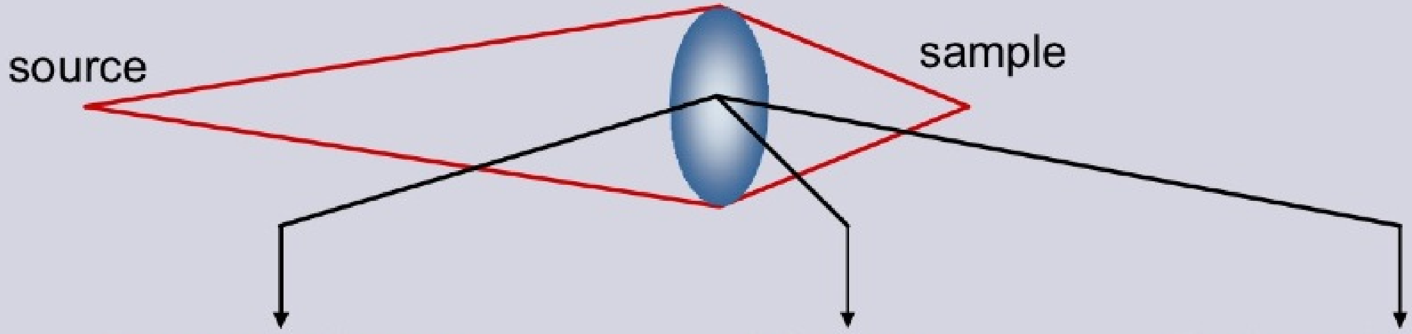
E = 20.5 keV

X-ray magnification ~ 80 (voxel size = 90 nm)

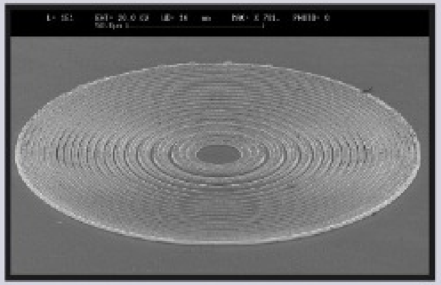
ID19

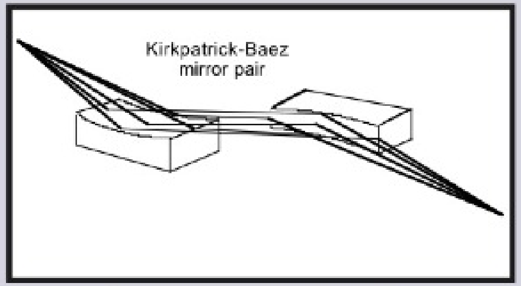
R Mokso, P Cloetens, E Maire, W Ludwig, JY Buffière

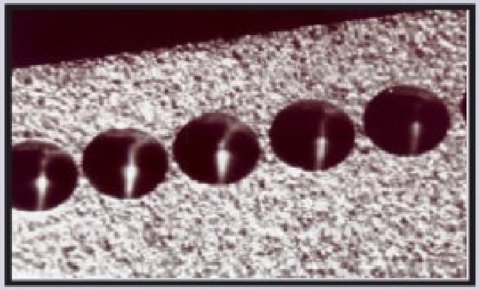
Comparison of optics



Zones plates | Mirrors | Refractive lenses







Energy ↗

• **Resolution**	+++	+++	+++
• **Achromaticity**	-(-)	+++	--(-)
• **Efficiency**	+	+++	++
• **Imaging (MTF)**	++++	-	+

J Susini

Conclusions

- **X-ray optics increasingly used in imaging setups**
  - **quality and availability remain limiting factors**
  - **continuously improving**

- **X-ray lenses for sub-micron imaging**
  - **be careful with simple numbers**
  - **KB multilayer optics: high efficiency, large *E* bandwidth**
  - **FZP's: low energies, objective lens in full-field microscopy**
  - **CRL's: high energies, diffraction**

- **Need for dedicated microscopes / end-stations**

- **Crucial for ESRF Upgrade Programme:**
  - **nanofocusing, imaging, …**