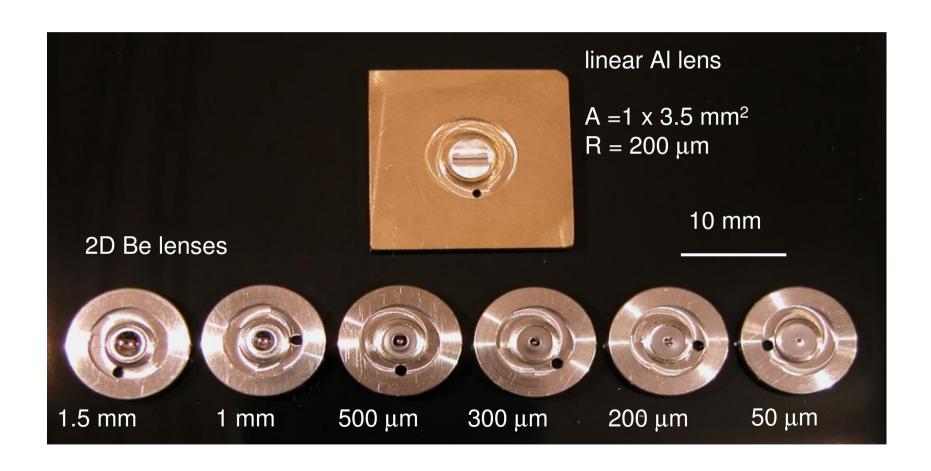
REFRACTIVE X-RAY LENSES NEW DEVELOPMENTS

BRUNO LENGELER

PHYSICS DEPARTMENT RWTH AACHEN UNIVERSITY

and RXOPTICS

Rotational parabolic and linear parabolic x-ray lenses from RXOPTICS



Linear Be lenses (cylinder paraboloids)

length 2.5mm

R=500µm

R=1500µm



A. HISTORY OF DEVELOPMENT OF REFRACTIVE X-RAY LENSES

1. W.C.Roentgen (1896)

found no refraction of x-rays by matter, concluded that there are **no refractive lenses for x-rays**, this statement is still found in most textbooks of optics.

2. P.Kirkpatrick, A.V.Baez (1948)

on search for x-ray optics:
realized the importance of low refraction and of strong absorption
that hamper the fabrication of refractive x-ray lenses
mentioned the possibility to stack lenses in order to reduce the focal length,
gave up the idea of refractive lenses in favor of crossed mirrors which
carry their names (K-B mirrors).

3. R.Gähler, J.Kalus, W.Mampe (1980)

Test of a **refractive neutron lens** developed for the search of a neutron charge:

2 biconcave cylinder lenses stacked behind one another with circular profile (radius 2.4mm), made of quartz, resulting in a focal length of 2.5m for cold neutrons of 20 A wavelength.

The possibility to transfer the concept to x-rays is not mentioned.

N.B.: materials for neutron lenses should have a large coherent cross-section and small incoherent and absorption cross-sections.

4. S.Suehiro, H.Miyaji, H.Hayashi (1991)

considered **one spherical biconcave lens of high Z material** (Au, Pt, W) no follow-up of idea

5. B.X.Yang (1993)

considered **one parabolic lens of low Z material** saw problems in manufacturing this lens proposed Fresnel lenses instead of refractive lenses

6. T.Tomie (1994-1997)

proposed row of drilled holes in a straight line

=> stacking of individual lenses reduces focal length alignment problem is solved

patent: Japan 1994 USA, Germany 1995 USA 1997

presented at XRM conference Wuerzburg (August 1996)

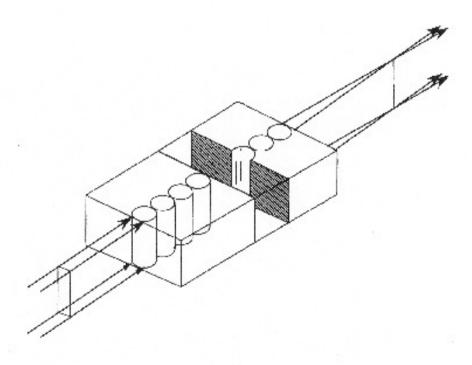
X-RAY LENS

U.S. Pat. No. 5,684,852, Date of Patent Nov. 4, 1997

Inventor: Toshihisa TOMIE
Electrotechnical Laboratory,
Agency of Industrial Science and Technology,
Ministry of International Trade and Industry

Related U.S.Application Data: Devision of Ser.No.389,503, Feb. 16, 1995, Pat. No. 5,594,773

Foreign Application Priority Data Feb. 18, 1994 Japan



BUT:

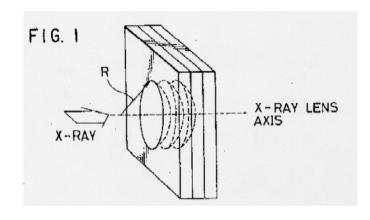
- * idea was not tested by author
- * linear lens with strong spherical aberration
- * rough cylinder surfaces
- * it is difficult to control the form fidelity:
 drilling of a hole deforms the adjacent hole

The stacking of individual lenses is explicitely excluded in the patent as not feasible!

Patent: X-ray lens

T. Tomie

U.S. Patent 1997 # 5,684,852



. . .

(3) The total focal distance f_T can be reduced to f/N by cascading N X-ray lenses of long focal distance f, as shown in Fig. 1. In this configuration, however, many unit X-ray lenses have to be arranged after fabricating the individual unit X-ray lenses. The thickness of each unit X-ray lens has to be very thin to avoid strong absorption of X-rays, making each unit X-ray lens very fragile and difficult to handle. Moreover, aligning the optical axes of all unit X-ray lenses along the X-ray lens axis with high precision would be extremely difficult. Hence, arranging many X-ray lenses in the configuration shown in Fig. 1 is practically impossible.

. . .

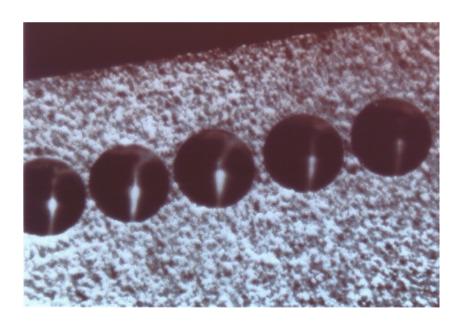
7. A. Snigirev, I. Snigireva, V. Kohn, B. Lengeler (1996)

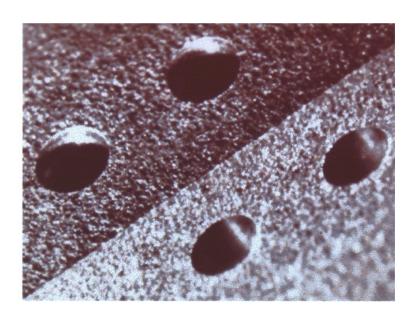
Nature 384, 49 (1996)

received 19 June 1996, accepted 19 September 1996.

first realisation and test of refracting x-ray lens (linear row of drilled holes in Al and later in Be)

- => showed feasibility of these lenses
- => not suitable as an optical device





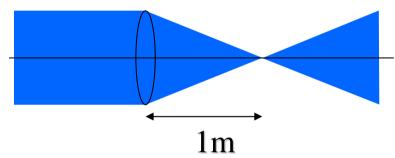
beryllium lenses with holes 0.5 mm in diameter

B. Strategy for refractive x-ray lenses

> have been considered as not feasible for a long time

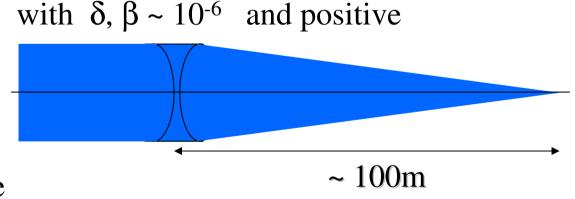
> visible light: index of refraction $n = 1 + \delta$ with $\delta \sim 0.5$ for glass

- * refraction strong
- * absorption weak
- * focal length short
- * focusing lens convex



> **x-rays**: $n = 1 - \delta + i\beta$

- * refraction weak
- * absorption strong
- * focal length long
- * focusing lens concave



"There are no refractive lenses for x-rays!" W.C.Roentgen **BUT:** refraction is not zero and absorption is not infinite!

Substantial improvement in design and manufacturing made at Aachen University and at RXOPTICS

- * parabolic form is a must!
- * **biconcave** form is a must!
- * stacking of many individual lenses in a row with µm precision was achieved
- * low Z material
- * in the mean time more than 4000 lenses (rotationally parabolic and cylinder parabolic) made in Be, Al and Ni have been delivered to 11 synchrotron radiation sources in 9 countries.
- * 1996-2010: more than 600 publications on and with refractive x-ray lenses
- * at ESRF: about 50% of beamlines equipped with refractive x-ray lenses.
- * others took over the concept: SPring8, ANKA, Kurchatov.

Design of refractive x-ray lenses

lensmaker formula:
$$\frac{1}{f} = (1-n)\frac{2}{R}$$
 or $f = \frac{R}{2\delta}$

$$\delta = 2.70(\lambda^2 \rho Z / A)10^{-6}$$

λ in Angstrom
 ρ in g/cm³
 Z atomic number
 A atomic mass in g

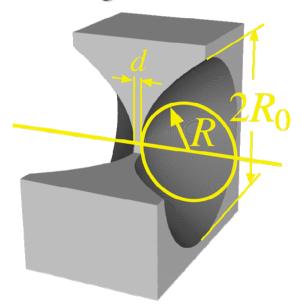
To obtain a **small focal length**:

small radius of curvature R: down to 50µm high density of lens material

Requirements on refractive x-ray lenses

i). Lens surfaces must be parabolic

single 2D-lens



parameters for Be lenses:

 $R = 50 \text{ to } 1500 \mu \text{m}$

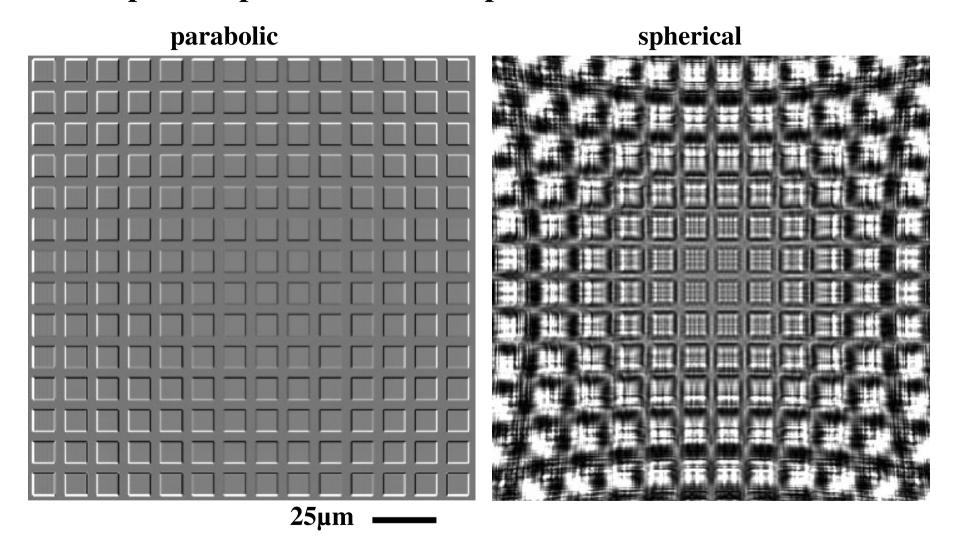
 $2R_0 = 0.45$ to 2.5mm

d below 30µm

parabolic profile: no spherical aberration focusing in full plane

=> excellent imaging optics

Comparison parabolic versus spherical lens

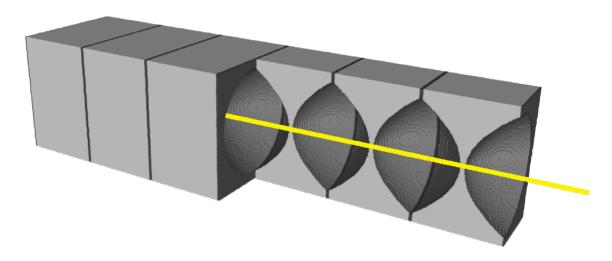


spherical lenses are inappropriate for imaging!

ii). stacking many lenses in a row

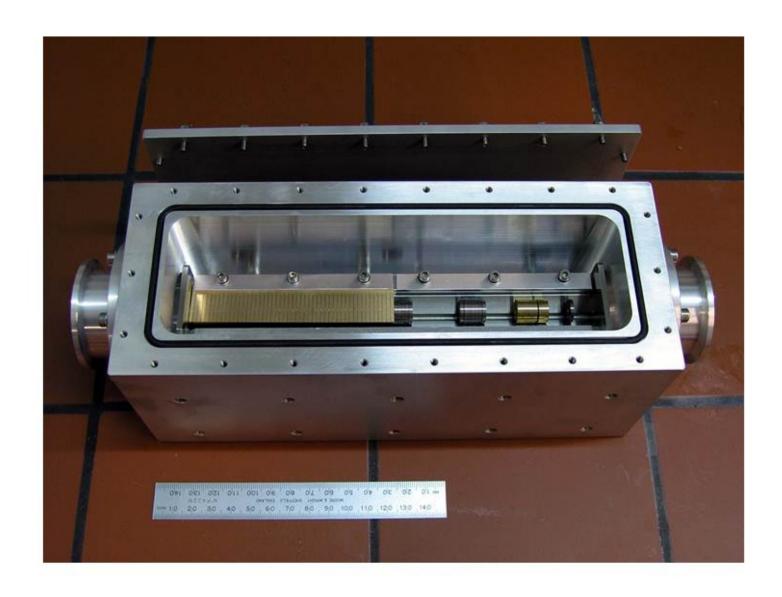
$$f = R / 2\delta N$$
 (thin - lens)

variable number of lenses : N = 1 to about 300 Precision of stacking: better than 1µm



typical: f = 0.2m - 10m

LENS CASING by RXOPTICS (can be integrated in vacuum of beam line)



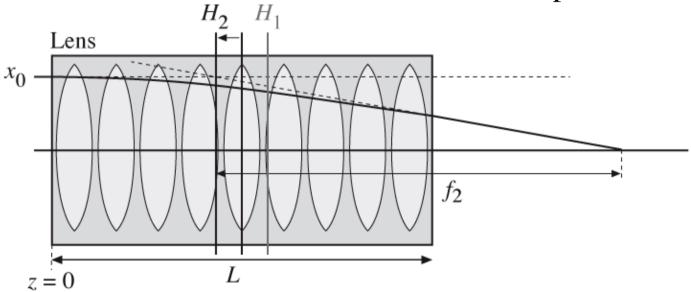
iii). Thick lenses

- * if L << f (thin lens): $f_0 = R / 2\delta N$
- * if L comparable to f : rays are bent towards optical axis inside lens

$$r(z) = R_0 \cos \kappa z$$

$$\kappa = \sqrt{\frac{2\delta}{RF}}$$

refracting power/length
F: thickness of lens
platelet



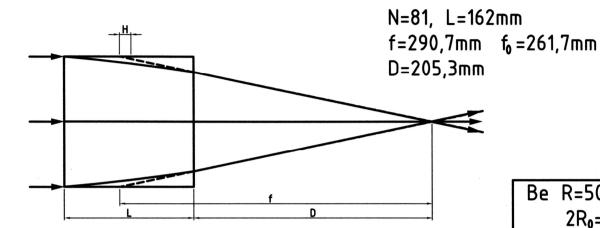
Focal length of thick lens

$$f = f_0 \frac{\sqrt{L/f_0}}{\sin\sqrt{L/f_0}}$$

L length of lens stack $(N*2z_0)$

 $f_0 = R / 2\delta N$ thin lens approximation

Minimal focal length achievable with Be, $R = 50\mu m$ at 17 keV

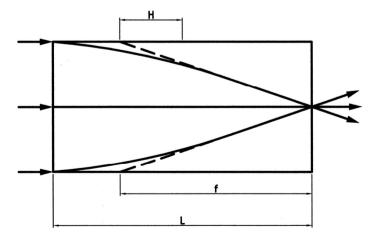


Be R=50 μ m

2R₀=447 μ m

17 keV, 0,7295A

2 δ =2,359 10⁻⁶ μ =0,4903/cm



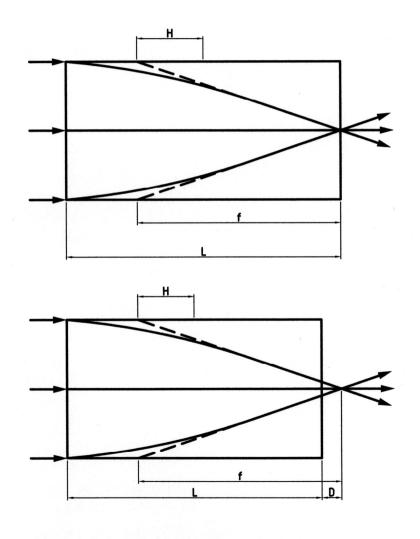
N=162, L=324mm f=205,9mm, f_0 =130,8mm D_{eff} =295,0 μ m d_{tr} =42,0nm (diffraction limit)

=> effective aperture : 295μm

=> best lateral resolution: 42nm (diffraction limit)

For lenses with constant refracting power:

number of lenses in the stack can be reduced slightly without loss of performance (the last lenses do not refract any more)



$$D_{\text{eff}}$$
=295,0 μ m d_{tr} =42,0nm

N=150, L=300mm f=207,2mm f=141,3mm D=23,5mm
$$D_{eff}$$
=287,7 μ m d_{tr} =43,3nm

iv). **Lens material** must be mechanically, thermally and chemically stable:

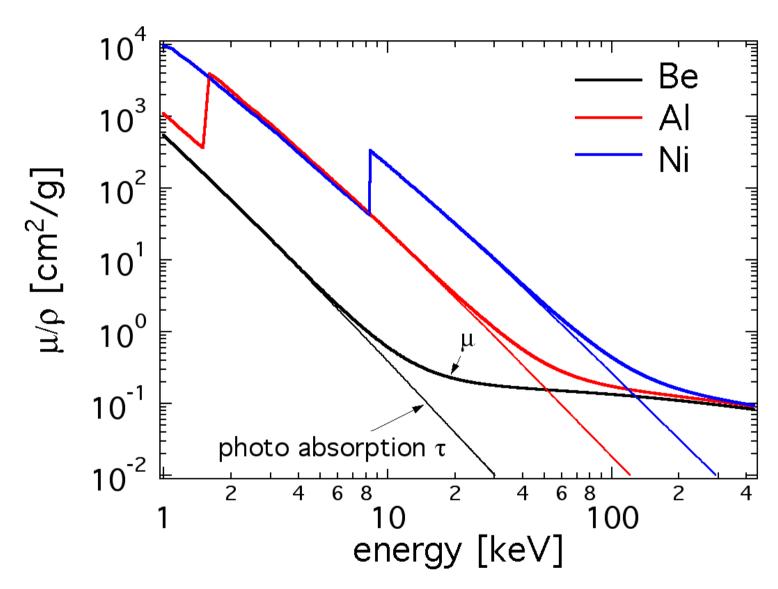
metals are the best choice! (no radiation damage) plastic is destroyed more or less fast in the x-ray beam!

v). low Z lens material:

mass absorption cofficient $\mu / \rho \sim Z^3 / E^3$

candidates: Be, B, C, Al, Si, Ni

Attenuation of x-rays in typical lens materials



Ultimately, **Compton scattering** limits transmission at high x-ray energies!

Refractive x-ray lenses available at RXOPTICS

material: Be 2 to 40 keV
 Al 40 to 80 keV
 Ni 80 to 150 keV

- profile: rotationally parabolic (2D) cylinder parabolic (1D)
- radii R at apex and geometric aperture $2R_0$

 $R = 50, 100, 200, 300, 500, 1000, 1500 \mu m$ $2R_0 = 450, 632, 894, 1095, 1414, 2000, 2450 \mu m$

length of 1D-lenses: 2.5mm lenses with R = 2000, 2500, 3000, 4000, 5000 and 5800 μ m (mainly for XFEL) are also available now.

• small radii for imaging and focusing large radii for prefocusing and for parallelisation.

A few examples: for 1m focal length by lenses with $R=50\mu m$

E (keV)	material	2δ (10-6)	N	f (m)
12.4	Be	4.4341	11	1.025
17	Be	2.3591	21	1.009
40	Be	0.4261	117	1.003
40	Al	0.6746	74	1.002
80	Al	0.1687	296	1.002
80	Ni	0.5515	91	0.996

How close can you adjust the focal length f (e.g. at 10 keV)?

R	200µm	300µm	500µm	1000µm
4	7.334 m	11.001 m	18.334 m	36.668 m
3	9.778 m	14.667 m	24.446 m	48.891 m
2	14.667 m	22.001 m	36.668 m	73.336 m

stacking of different lenses

$$\frac{1}{f} = \sum_{j} \frac{1}{f_{i}}$$

for $f=8m : 3*R=200\mu m$ and $1*R=300\mu m : f=8.000$

for f=9m : $3*R=200\mu m$ and $1*R=1000\mu m$: f=9.167m

if possible and needed: choose E=9.908keV then 3*R=200µm and 1*R=1000µm gives f=9.000m

C. Properties of refractive x-ray lenses

In the following we consider mainly Be, Al and Ni

1. Energy range

Be: about 2 to 40keV

d guaranteed below 50µm, typically 30µm

Al: about 30 to 80 keV

d guaranteed below 30µm, typically 22µm

Ni: about 80 to 150 keV

d guaranteed below 20µm, typically 10-16µm

2. Material properties

Beryllium

manufactured by powder metallurgy
contains up to 1wt% of BeO
contains many grain boundaries
=> small angle x-ray scattering
results in background radiation

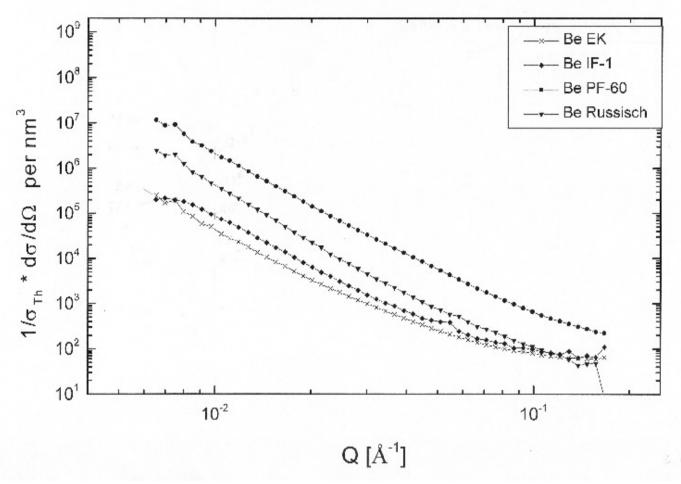
density: 1.85 g/cm³

melting point: 1287 °C

recrystallisation: about 600°C (depending on quality)

main supplier: MATERION- BRUSH-WELLMAN

Small-angle x-ray scattering in different types of Be



PF-60 is standard Be from BW
IF-1 has 20 times less SAXS than PF-60
only 2 times more SAXS than single crystal (EK)

BERYLLIUM from Materion-Brush-Wellman

wt-ppm	BeO	Fe
PF60, I70H	<7000	600 -700
O30H	4000	900
IF1	100 - 600	250

IF1 only available as 0.5mm plates!

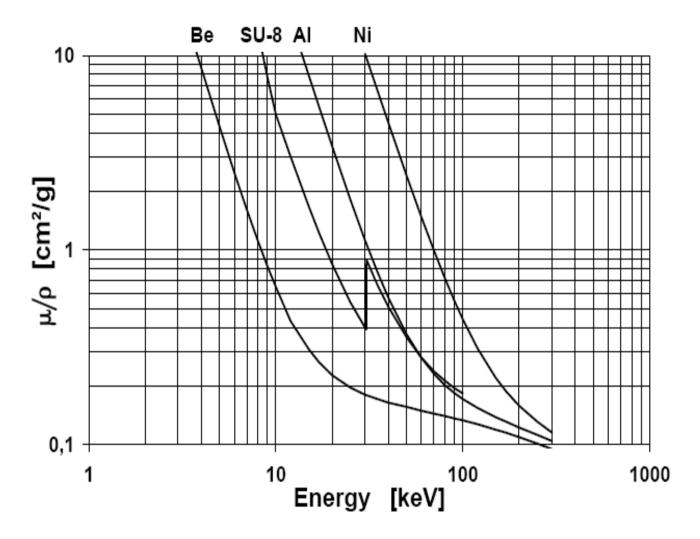
Small angle scattering of different lens materials

Be single crystal	5 *	* 10^4 σ_{Th}	$/\text{nm}^{3}$ at 0.0565°
Be IF-1	10		or $Q=10^{-2}/A$
Be PF-60	238		
Be Russian	47		
Al 5N	90		
B HCStarck	20		
diamond	14		
PMMA	2		
Teflon CF ₂	770		
Pyro-graphite	200		
glassy carbon	1000-1000	0	
sapphire Al ₂ O ₃	2		

Lens material: metals versus resists

	metals Be Al	S Ni	resists PMMA, Kapton, SU-8,
radiation damage	none		yes
heat conductivity (W/m.K)	200 237	91	ca 0.2
melting point (℃)	1277 660	1453	ca 200
SAXS	low to medium		low to high
density	1.85 2.7	8.9	ca 1.1
form	1D and 2D		only 1D
R_{min}	50μm		10μm
kinoform	no		yes

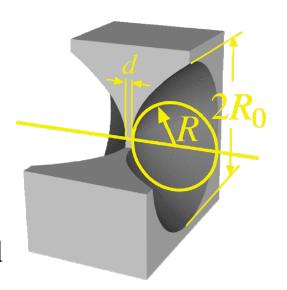
X-ray absorption in SU-8



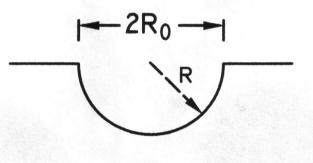
SU-8 contains 1 atom of Sb per formula unit!
SU-8: no advantage compared to Be and Al!

3. Aperture of paraboloid of rotation:

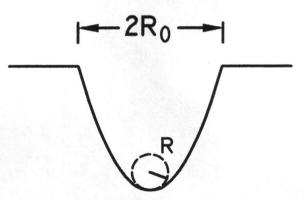
- * no spherical aberration
- * focusing in full plane
 - => excellent imaging optics
- * radius R and aperture 2R₀ are decoupled



spherical lens:



parabolic lens:

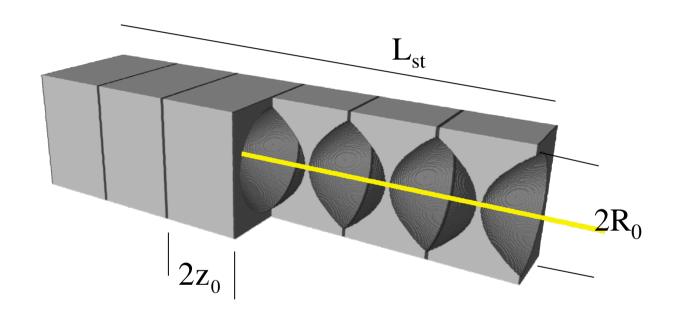


 $R_0 \le R$

R₀ and R independent

Effective lens aperture D_{eff}

Absorption reduces the effective aperture below the value of the geometric aperture $2R_0$



$$D_{eff} = 2R_0 \sqrt{\left[1 - \exp(-a_p)\right]/a_p}$$

$$a_p = \mu N z_0 = \frac{1}{2} \mu L_{st}$$

Transmission T versus effective aperture D_{eff} (A_{eff})

transmission T: fraction of transmitted intensity compared to intensity falling on geometric aperture πR_0^2

$$T = \frac{1}{\pi R_0^2} \int_0^{R_0} \exp(-\mu N 2z) = \frac{1}{2a_p} [1 - \exp(-2a_p)]$$

$$a_p = \mu N R_0^2 / 2R = \mu N z_0$$

effective aperture \mathbf{D}_{eff} reduced by absorption compared to geometric aperture $2R_0$

$$D_{eff} = 2R_0 \sqrt{[1 - exp(-a_p)/a_p]}$$

Example: Be stack with N = 50, R = 50 μ m at 17 keV $2\delta = 2.359 \ 10^{-6}$ and $\mu = 0.4903/\text{cm}$ f = 423.9mm

Z_0	$2R_0$	$\mathbf{D}_{\mathbf{eff}}$	T
(µm)	(µm)	(µm)	
500	447.2	339.5	37.3%
1000	632.5	386.2	20.2%
	100	98.5	94.1%

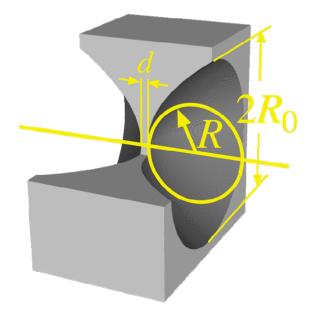
The **effective aperture** is the relevant parameter for characterizing the transmission of refractive lenses!

Influence of material between apices on transmission of lensstack (thickness d)

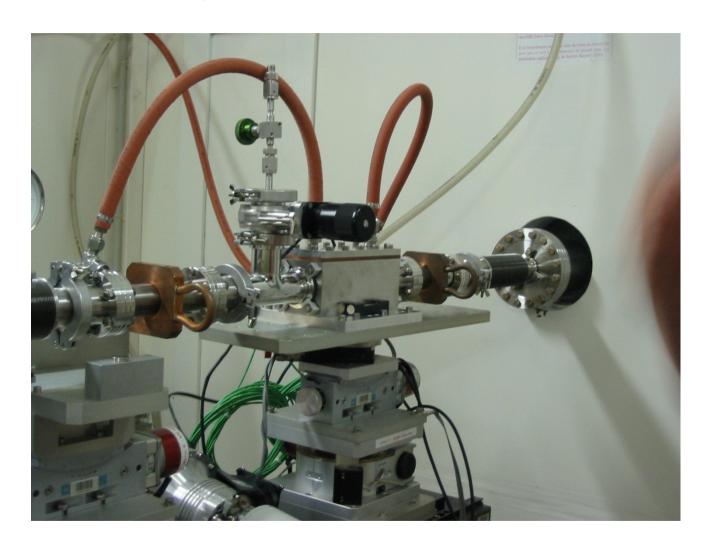
Transmission = $\exp(-\mu Nd)$

Example: Be lenses R=50µm, d=30µm

- 1. 12keV, μ=0.8196/cm, N=22, f=0.480m transmission: 94.7%
- 2. 17keV, μ=0.4903/cm N=42, f=0.505m transmission: 94.0%

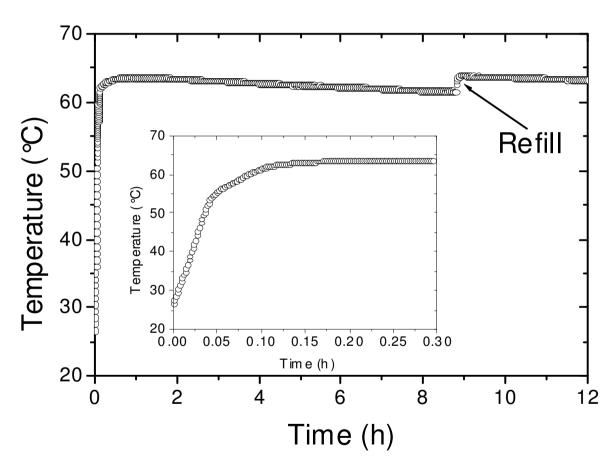


4. Thermal stability in the beam



Water cooled beryllium lens at ESRF (ID10)

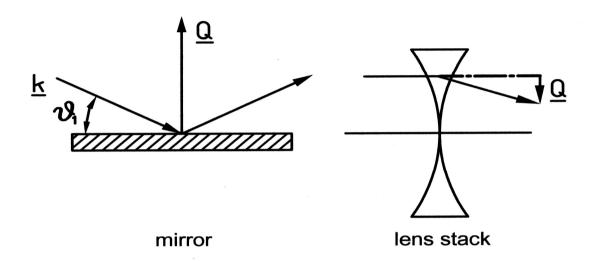
Temperature - time profile in white beam at ID10 ESRF ca. 100 W/mm² & total 40 W (**Be lens**)



In Be lenses the temperature should not exceed about 300°C!

5. Insensitivity of lenses to surface roughness and contamination

(compared to mirrors)



Damping of intensity due to surface roughness σ : $\sim \exp[-Q^2 \sigma^2]$ with **momentum transfer Q** = $2k \sin \theta_1 \cong 2k \theta_1$

mirror
$$Q = 1.4 \ 10^{-1} \ A^{-1}$$

at
$$\theta_1 = 0.6^{\circ}$$
 and $\lambda = 1A$

lens stack
$$Q = N^{1/2} k \delta = 1.4 \cdot 10^{-4} A^{-1}$$
 at $N = 100$ and $\lambda = 1A$

A lens is about 1000 times less sensitive to σ than a mirror!

www.rxoptics.de

Typical value of surface roughness of our lenses: 0.1µm

For
$$l = 1A$$

 $N = 100$
 $Q = 1.4 \ 10-4 \ / A$
 $exp(-Q^2s^2) = 0.981$

This is tolerable!

6. Chromatic aberration

refractive x-ray lenses show strong chromatic aberration

$$f = R/2\delta N$$

$$\delta = 2.70 * 10^{-6} * \lambda^2 \rho Z/A$$

Changing the energy at fixed focal length implies changing the number of lenses in the stack!

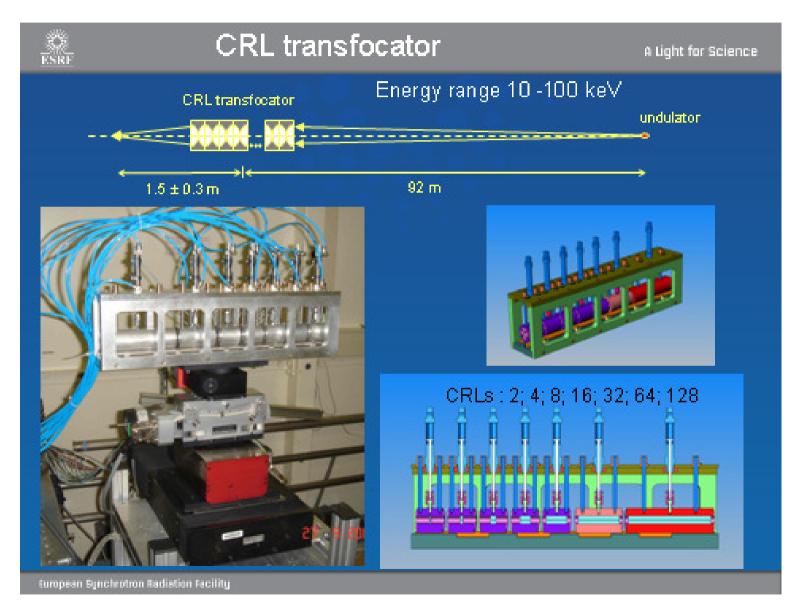
solution: TRANSFOCATOR developed at ESRF

flexible change of f

in air and in vacuum

new type of monochromator

TRANSFOCATOR (ESRF development)



7. Handling and adjustment

a. refractive lenses are robust and compact:

easily installed and removed in its own lens casing or in the vacuum of the beam line

b. focus stays on axis:

fast adjustment (typically in 15 minutes)
relatively insensitive to misorientation
to vibrations
no need for readjusment of the beam-line components
downstream

c. comfortable working distance between optics and sample

REFRACTIVE LENSES: EXCELLENT WORKING HORSES!

D. Applications of refractive x-ray lenses

refractive x-ray lenses can be used like glass lenses are used for visible light

but

the numerical aperture N.A. is very small typically 10^{-4} to 10^{-3}

New and improved x-ray techniques

1. **Imaging**: x-ray microscopy: 2D image

x-ray tomography: 3D reconstruction

in absorption and phase contrast

monitor of source in storage ring

test of optical components upstream from lens

2. Focusing: diffraction,

spectroscopy.....

with high lateral resolution

in the sub 100 nm range (50 nm were

reached)

3. Coherent photon flux:

X-ray diffraction

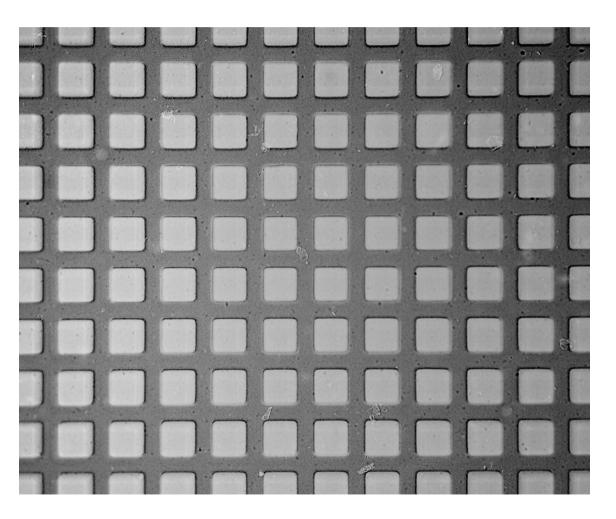
speckle spectroscopy

1. High resolution x-ray microscopy

Example: Ni mesh 12.7µm period

parabolic refractive Be lens N = 91, $R = 200 \mu m$

f = 495 mm at 12 keV



magnification: 10

detector: high

resolution film

NO DISTORTION!

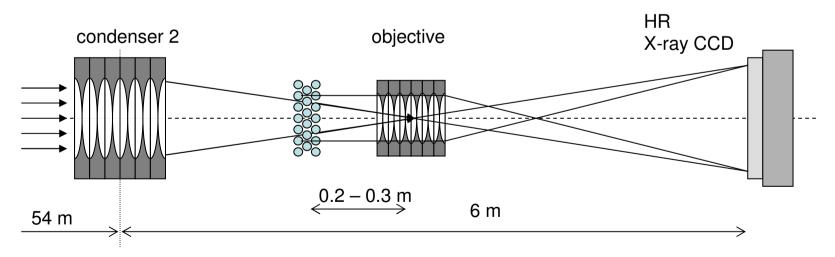
High-resolution x-ray microscopy

illumination of object from behind via **prefocusing lens** (condenser 2) in order to adjust beam size on sample

objective with small focal length and low distortion

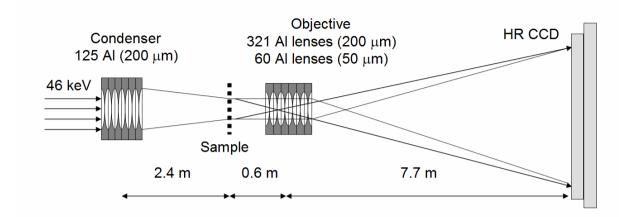
(rotationally parabolic) d_{tr} down to about 50nm

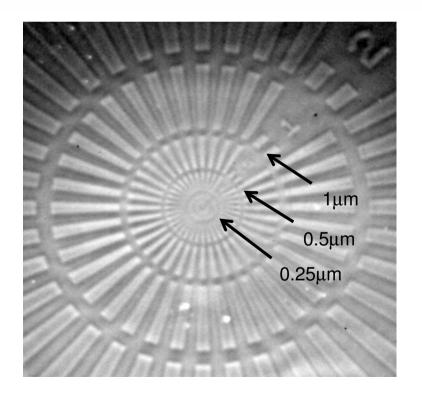
large magnification in order to relieve requirements on CCD camera (object slightly outside focus)



A. Snigirev et al

High Energy X-ray Microscopy at ID15 Al lenses





Siemens star Ta 0.5 μm

E = 46 keV

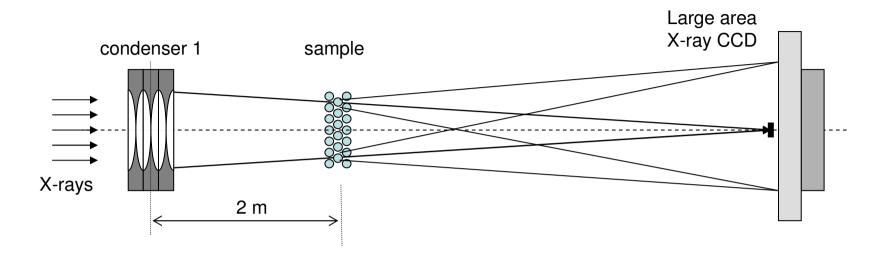
M. Di Michiel

M. Scheel

A. Snigirev

I. Snigireva

Microscopy in diffraction mode

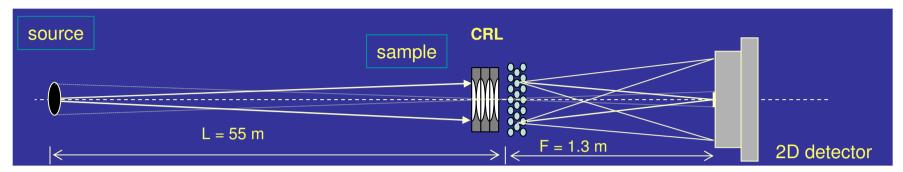


Be
$$N = 19$$
, $R = 300 \mu m$

The same place on the sample can be investigated in imaging mode diffraction mode

(like in electron-microscopy)

X-ray High Resolution Diffraction Using Refractive Lenses



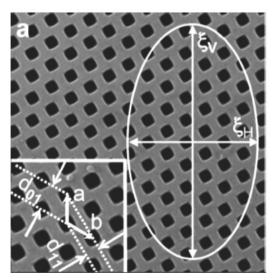
E = 28 keVAl CRL, N = 112, F = 1.3 m

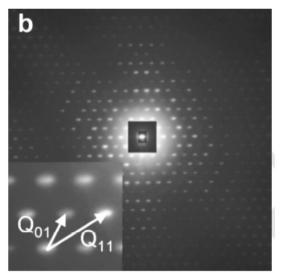
CCD resolution $2 \mu m$ pixel $/ \Theta = d$

Resolution is limited by angular source size: $s/L \sim 1 \mu rad$

Momentum transfer Resolution: 10⁻⁴ nm⁻¹

Si photonic crystal a=b=4.2 μ m d₀₁=3.6 μ m d₁₁=2.1 μ m





Lattice vectors $g_{01} = 1.75 \cdot 10^{-3} \text{ nm}^{-1}$ $g_{11} = 3 \cdot 10^{-3} \text{ nm}^{-1}$

2. Focusing

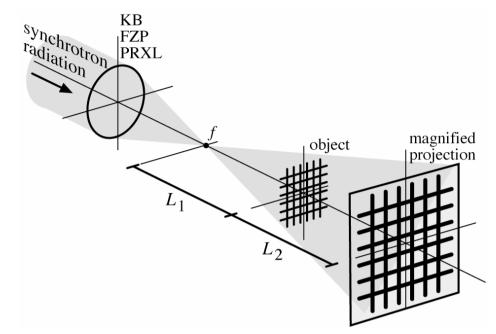
Microscopy

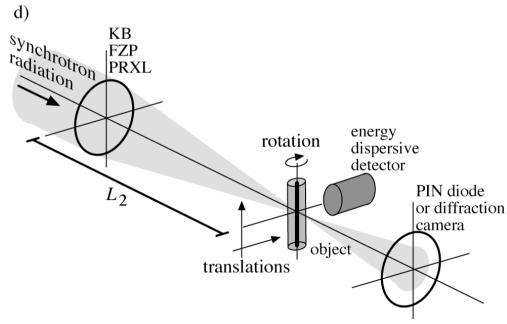
Object placed close to secondary source: => strong magnification

The smaller the focus, the sharper the image!

Spectroscopy, tomography

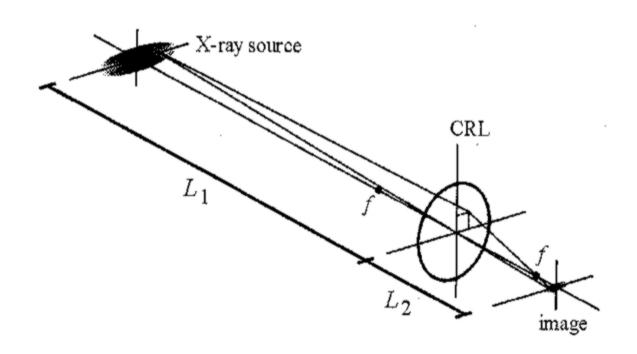
large depth of field scanning beam over sample (diffraction, SAXS, XAS, fluorescence...)





Small focus requires

- 1. small source
- 2. long distance L_1 source-lens
- 3. small focal length and large effective aperture of lens



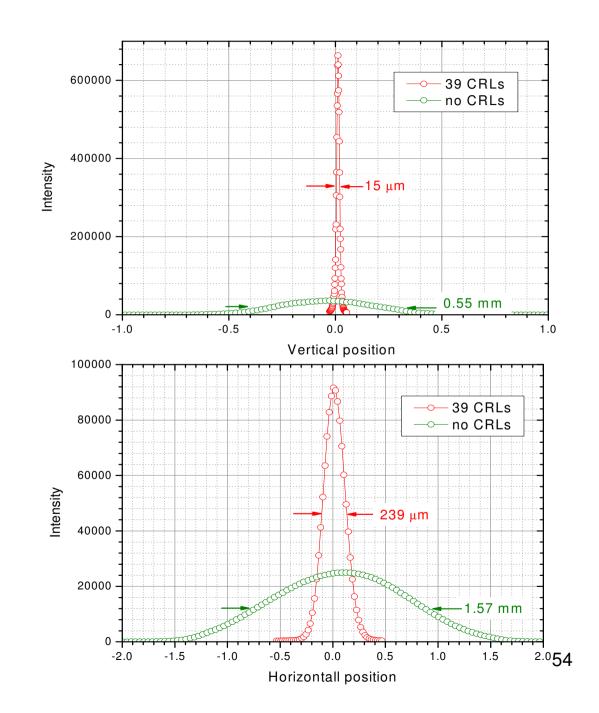
a. FOCUSING with rotationally parabolic Be lenses (R = 1500µm)

Image of the ID18 source at ESRF

14.4125eV 39 Be lenses R = 1500μm

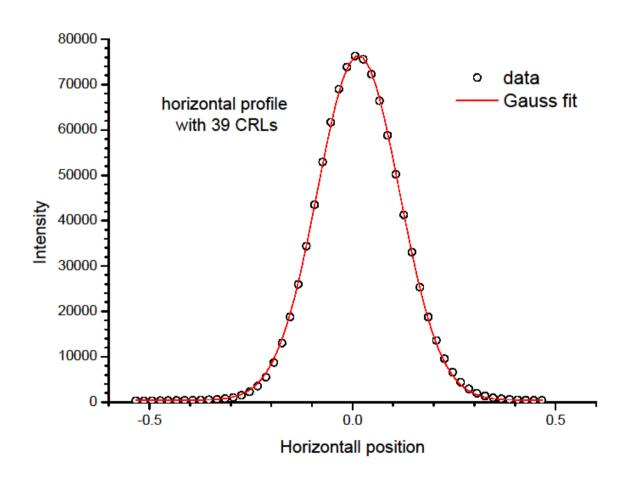
f = 11.718m geometric aperture: 2.5mm

(A. Chumakov ESRF)



Intensity profile in the horizontal: ID18

well fitted by a Gaussian with 239 μm FWHM (very low background in the wings)



b. Focusing with Be lens at energies as low as 2keV

ID12 at ESRF (A. Rogalev)

gain in intensity on sample at 2 keV:

factor 500 compared to situation without lens!

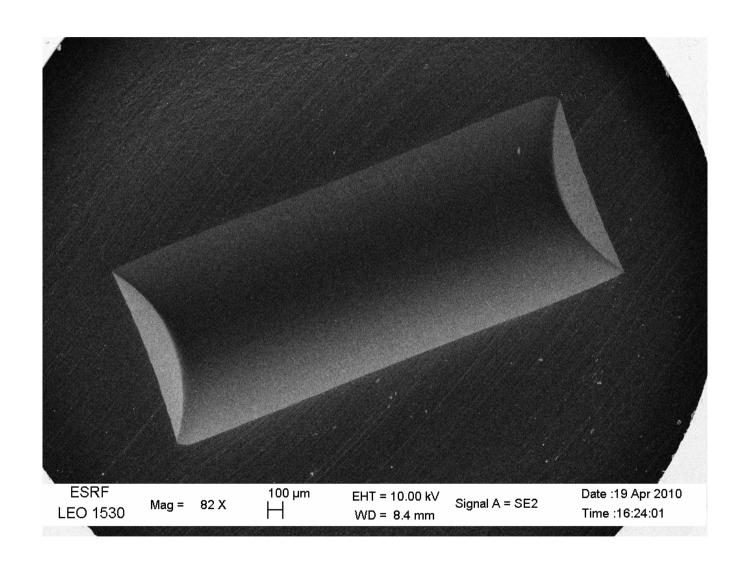
c. Prefocusing with linear lenses Be, Al and Ni

 $R = 200 \text{ to } 1000 \mu \text{m}$, length 2.5 mm

- * collecting more intensity
- * for making spot on sample more circular (on storage rings)



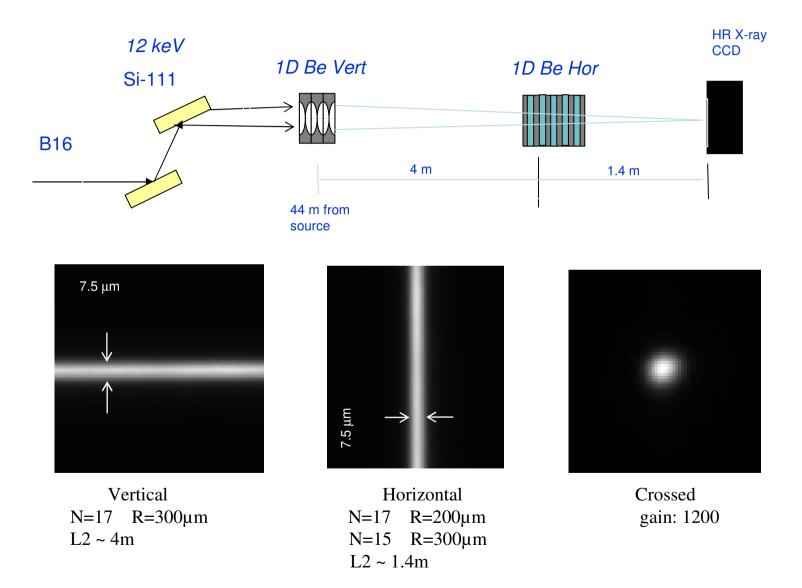
SEM image of linear Be lens (R=500µm)



Focusing with 2 independent linear lenses in cross-geometry

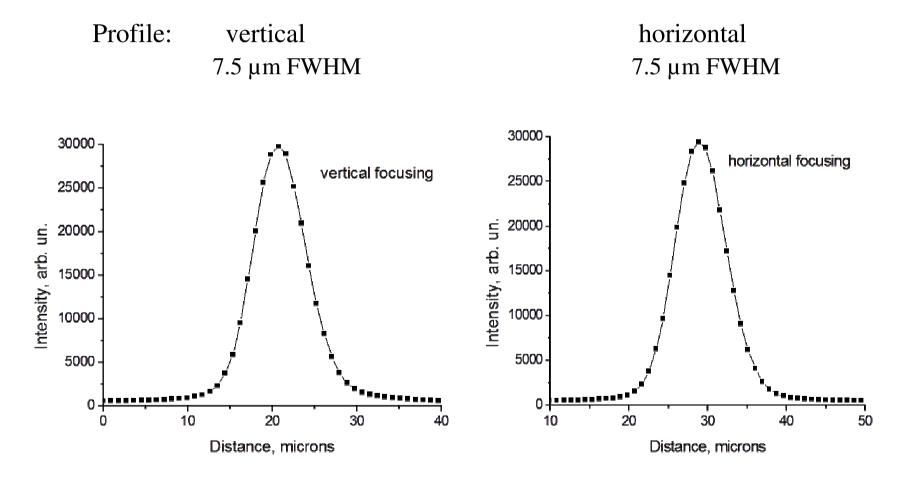
- Ratio of horizontal to vertical source size in storage rings: 20 and more
 - =>elongated spot on sample
- Generation of more circular spot size by astigmatic imaging of source via 2 independent linear lenses in cross geometry
- Example: experiment at DIAMOND Light Source by A. Snigirev et al with 1D Be from RXOPTICS

Astigmatic focusing with 2 crossed, linear Be lenses



Astigmatic focusing with 2 crossed, linear Be lenses

I & A Snigirev, I. Dolbnya, K. Sawhney Collaboration with Optics Group at DIAMOND



3. Coherent flux

- * diffraction of individual large molecules, nanoparticles
- * speckle spectroscopy

Illuminated area on sample must be smaller than the lateral coherence area at the sample position. Then all monochromatic photons are undistinguishable, i.e. they are in the same mode!

* coherent photon flux is a property of the brillance B of the source and of the degree of monochromaticity

$$F_{c} = B\lambda^{2} \frac{\Delta\lambda}{\lambda}$$

* the coherent flux can at best be conserved, it cannot be increased by a focusing optic.

Example: ID13 at ESRF

Be lens:
$$R = 50\mu m$$
, $N = 162$, $f = 205.9mm$,

$$D_{eff} = 295 \mu m, d_{tr} = 42 nm$$

$$L_1 = 100 \text{m}, L_2 = 206.3 \text{mm}$$

geometric image of source
$$S' = S \frac{L_2}{L_a}$$

FWHM	S (µm)	S' geom (nm)	S' incl diffr (nm)
horizontal	120	248	251
vertical	20	41	59

diffraction limited in the vertical!

Example: low-betha undulator at ESRF

1. Be lenses, 17 keV, N = 162, f = 205.9mm,
$$\mathbf{d_{tr}} = \mathbf{42nm}$$

 $\mathbf{L_1} = 100 \text{ m}, \ \mathbf{L_2} = 0.2063 \text{ m}$

2.

	Source size FWHM	Geometric image FWHM
horizontal	120µm	248 nm
vertical	20µm	41nm

Image is diffraction limited in the vertical:

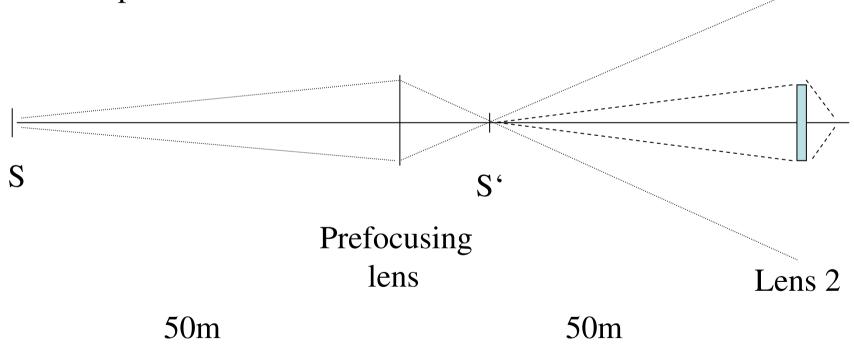
=> coherent illumination in the vertical

Not so in the horizontal!

3. remedy for horizontal direction

* insert a linear lens (prefocussing lens) which focuses only in the horizontal

* the secondary source S' must have a lateral coherence length at the postion of lens 2 which is equal to the effective aperture of lens2.



Prefocusing lens

Be linear: $R = 500\mu m$, N = 55, f = 3.854m, $D_{eff} = 1048\mu m$

Image S' at $b_1 = 4.168m$ behind horizontal lens

lateral (horizontal) coherence length at position of lens 2: $295 \mu m$

this is equal to D_{eff} of lens 2: only the coherent flux passes through lens 2, the rest is peeled off.

gain in flux (compared to no prefocusing): about factor 10.

Coherent Imaging (Ptychography)

(see talk by F. Seiboth, C. Schroer)

- * illuminate sample coherently in a small spot by means of Be-lenses
- * Scan this microfocus over sample with overlaping neighboring scans
- * take a diffraction image on each position
- * overlap of images allows for reconstruction of the object when each spot is illuminated coherently
- → Our Be lenses preserve coherence well enough to give a resolution which is 10 times better than the spot size!

MANY THANKS

To

my former students,

Anatoly and Irina Snigirev from ESRF

Christian Schroer and collaborators from TU Dresden

for many years of efficient and pleasant collaboration