REFRACTIVE X-RAY LENSES NEW DEVELOPMENTS

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(Grenoble, July 2010)

A. 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



- * 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!**²



Design of refractive x-ray lenses

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

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

To obtain a **small focal length**:

- i) small radius of curvature R: typical: R = 50 to 1500µm
- ii) high density of lens material
- iii) profile must be parabolic: no spherical aberration

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

Lens surfaces must be paraboloids of rotation



parameters for Be lenses:

- R = 50 to $1500 \mu m$
- $2R_0 = 0.45$ to 2.5mm
- d below 30µm

Rotational parabolic and linear parabolic lenses



Linear Be lenses (cylinder paraboloids)

length 2.5mm

R=500µm

R=1500µm



SEM image of linear Be lens (R=500µm)



Refractive x-ray lenses available at Aachen University

material: Be 6 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

• small radii for imaging and focusing large radii for prefocusing

iv). 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

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



A few examples: for 1m focal length by lenses with R=50µm

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 N	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

tacking of different lenses
$$\frac{1}{f} = \sum_{j=1}^{j=1} \frac{1}{f}$$

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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 $3*R=200\mu m$ and $1*R=1000\mu m$ gives f=9.000m

More flexibility by lenses with larger R!

v). Lens material must be mechanically, thermally and chemically stable

vi). low Z lens material:

mass absorption cofficient $~\mu$ / ρ \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! ¹⁴

Cylinder parabolic (1D) lenses from ESRF-Russia and from TU Dresden

material: Si

technology: microfabrication (e-beam lithography, etching)

performance: stapling is done by microfabrication

bilenses possible

very small radii possible, (down to 1µm) => very small focal length

only 1D-lenses

Nanofocusing Lenses (NFL)



Silicon bilens (ESRF-Russia)



B. Properties of refractive x-ray lenses

In the following we consider mainly Be, Al and Ni

1. Energy range

Be : about 5 to 40keV

d guaranteed below 50µm, typically 30µm

Al : about 30 to 80 keV

d guaranteed below $30\mu m$, typically $22\mu m$

Ni: about 80 to 150 keV

d guaranteed below $20\mu m$, typically $10-16\mu m$

2. Comparison parabolic versus spherical lens



spherical lenses are inappropriate for imaging!

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!

3. 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

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density : 1.85 g/cm<sup>3</sup>
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melting point : 1287 °C

recrystallisation: about 600°C

main supplier: 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) 22

Small angle scattering of different lens materials

Be single crystal	5	*	10^{4}	σ_{Th}/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	1000-100	00			
sapphire Al ₂ O ₃	2				

Lens material: metals versus resists

	metals			resists	
	Be	Al	Ni	PMMA, Kapton, SU-8,	
radiation damage		none		yes	
heat conductivity (W/m.K)	200	237	91	ca 0.2	
melting point (°C)	1277	660	1453	ca 200	
SAXS	low to medium		um	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 !

4. Aperture of paraboloid of rotation:

- * no spherical aberration
- * focusing in full plane
 - => excellent imaging optics
- * radius R and aperture $2R_0$ are decoupled





Effective lens aperture D_{eff}

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



$$D_{eff} = 2R_0 \sqrt{\left[1 - \exp\left(-a_p\right)\right]/a_p}$$
$$a_p = \mu N z_0 = \frac{1}{2} \mu L_{st}$$

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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 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 δ = 2.359 10⁻⁶ and μ = 0.4903/cm f = 423.9mm

Z ₀	$2R_0$	D _{eff}	Т
(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 (thickness d) between apices on transmission of lensstack

Transmission = $exp(-\mu Nd)$

Example : Be lenses $R=50\mu m$, $d=30\mu m$

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%



5. 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**)



about 300°C!

6. Insensitivity of lenses to surface roughness and contamination (compared to mirrors)



Damping of intensity due to surface roughness $\sigma \sim \exp[-Q^2 \sigma^2]$ 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 $\delta = 1.4 \ 10^{-4} \ A^{-1}$ at N = 100 and $\lambda = 1A$ A lens is about 1000 times less sensitive to σ than a mirror!

Typical value of surface roughness of our lenses: 0.1µm

For
$$1 = 1A$$

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

7. 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)



European Synchrotron Radiation Facility

8. Thick lenses

* if $L \ll f$ (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



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



=> 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)



Adiabatically focusing lenses (PRL 94, 054802(2005))

the refractive power per unit length increases along the lens!



Adiabatically focusing lenses

 $2R_{0i}$ and $2R_{0f}$ entrance and exit diameter

focal length $f = \frac{R_{0i}}{\sqrt{4\delta \ln \frac{R_{0i}}{R_{0f}}}}$ length of stack $L = \frac{\sqrt{\pi}R_{0i}}{\sqrt{4\delta}} \Phi(\ln \frac{R_{0i}}{R_{0f}})$

effective aperture

$$D_{eff} = 2R_{0i} \sqrt{\frac{1}{a_p} (1 - exp(-a_p))}$$
$$a_p = \mu L/2$$

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Two examples of adiabatically focusing lenses

1. rotationally parabolic Be lenses at 17 keV $(2\delta = 2.359 \ 10^{-6})$

$$R_{i} = 1500 \mu m, R_{0i} = 1224.7 \mu m$$

$$R_{f} = 50 \mu m, R_{0f} = 223.6 \mu m$$

$$=> f = 432.4 mm$$

$$L = 983.2 mm$$

$$D_{eff} = 498.9 \mu m$$

$$d_{tr} = 52.2 nm$$

compared to $d_{tr} = 42.0$ nm for thick lens with maximum length:

 $R = 50 \mu m$, N=162, L=324mm, f=205.9mm, D_{eff} = 295.0 \mu m

not worth the effort!

2. Cylinder paraboloids (C.Schroer TU Dresden)

a. adiabatically focusing **diamond** lenses at 27.6keV

$$R_{0i} = 9.43 \mu m$$

$$R_{0f} = 50 nm \quad (R_f = 25 nm)$$

$$N = 1166$$

$$d_{tr} = 4.7 nm \quad (11.6 \mu m \text{ behind stack}) \quad diffraction limit$$
ompared to

b. nanofocusing **diamond** lens with constant aperture

N = 200 $R = 1.31 \mu m$ $R_0 = 7.2 \mu m$ $d_{tr} = 14.2 nm$ diffraction limit

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9. 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: NO RACING HORSES BUT EXCELLENT WORKING HORSES !

C. 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

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

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) see below: d_{tr} about 50nm

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



A. Snigirev et al



14.4125eV 39 Be lenses R = 1500μm

f = 11.718mgeometric aperture: 2.5mm

(A. Chumakov ESRF)



Intensity profile in the horizontal: ID18

well fitted by a Gaussian with 239 µm FWHM



Prefocusing with linear lenses Be, Al and Ni

- R = 50 to 1500mm length 2.5 mm
- * collecting more intensity
- * for making spot on sample more circular (on storage rings)





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, $R = 300 \mu m$

X-ray High Resolution Diffraction Using Refractive Lenses



E = 28 keV AI CRL, N = 112, F = 1.3 m

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

Resolution is limited by angular source size: s/L ~ 1 µrad

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

M. Drakopoulos, A. Snigirev, I. Snigireva, J. Schilling, Applied Physics Letters, 86, 0141024 2005.

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



Example: ID13 at ESRF

Be lens:
$$R = 50\mu m$$
, $N = 162$, $f = 205.9mm$,
 $D_{eff} = 295\mu m$, $d_{tr} = 42nm$

 $L_1 = 100m, L_2 = 206.3mm$

geometric image of source

$$\mathbf{S'} = \mathbf{S} \frac{\mathbf{L}_2}{\mathbf{L}_1}$$

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

diffraction limited in the vertical !

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

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

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

Example: low-betha undulator at ESRF

1. Be lenses, 17 keV, N = 162, f = 205.9mm, $d_{tr} = 42nm$ L₁ = 100 m, L₂ = 0.2063 m

2	
L	•

	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 = 3.854 m, $D_{eff} = 1048 \mu m$

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

lateral (horizontal) coherence length at position of lens 2: 295µm

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.

MANY THANKS

to

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