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Front Matter: Volume 7077

, "Front Matter: Volume 7077," Proc. SPIE 7077, Advances in X-Ray/EUV Optics and Components III, 707701 (6 October 2008); doi: 10.1117/12.812350

SPIE.

Event: Optical Engineering + Applications, 2008, San Diego, California, United States

PROCEEDINGS OF SPIE

Advances in X-Ray/EUV Optics and Components III

**Ali M. Khounsary
Christian Morawe
Shunji Goto**
Editors

**11–13 August 2008
San Diego, California, USA**

Sponsored and Published by
SPIE

Volume 7077

Proceedings of SPIE, 0277-786X, v. 7077

SPIE is an international society advancing an interdisciplinary approach to the science and application of light.

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Please use the following format to cite material from this book:

Author(s), "Title of Paper," in *Advances in X-Ray/EUV Optics and Components III*, edited by Ali M. Khounsary, Christian Morawe, Shunji Goto, Proceedings of SPIE Vol. 7077 (SPIE, Bellingham, WA, 2008) Article CID Number.

ISSN 0277-786X

ISBN 9780819472977

Published by

SPIE

P.O. Box 10, Bellingham, Washington 98227-0010 USA

Telephone +1 360 676 3290 (Pacific Time) · Fax +1 360 647 1445

SPIE.org

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Printed in the United States of America.

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Focus on X-Ray Focusing Workshop

Ali M. Khounsary, Argonne National Laboratory (United States)
Christian Morawe, European Synchrotron Radiation Facility (France)
Shunji Goto, Japan Synchrotron Radiation Research Institute (Japan)

Focus on X-Ray Focusing Workshop

August 13, 2008

"Focus on X-Ray Focusing" was the title of a one-day workshop during Optics + Photonics 2008 held on August 13, 2008 in conjunction with the Advances in X-Ray/EUV Optics and Components III conference. The aim of the Workshop was to provide the audience with a comprehensive introduction and up-to-date information on various X-ray focusing techniques covering theory, development, implementation, progress and applications. The Workshop consisted of ten presentations by some of the renowned practitioners in the field, each describing one of the focusing techniques and its challenges, limitations, and prospects.

Workshop Chairs:

Ali Khounsary, Argonne National Laboratory

Christian Morawe, European Synchrotron Radiation Facility (France)

Shunji Goto, Japan Synchrotron Radiation Research Institute (Japan)

Introduction to x-ray focusing, Franz Pfeiffer, Swiss Light Source, Paul Scherrer Institute and Ecole Polytechnique Fédérale de Lausanne (Switzerland)

X-ray focusing with Kirkpatrick-Baez optics, K. Yamauchi, Osaka Univ. (Japan)

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Diffraction focusing by zone plates, Michael Feser, Xradia, Inc.

13th August 2008

Introduction to X-Ray Focusing

Franz Pfeiffer

Swiss Light Source, Paul Scherrer Institut
& École Polytechnique Fédérale de Lausanne

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SLS

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Christian David, Joan Vila, Konstantins Jefimovs, Vitaliy Guzenko, Harun Solak, Sankha Sarkar,
Laboratory for Micro- and Nanotechnology,
Paul Scherrer Institut, CH

Outline

1. X-ray focusing – Why
2. Methods overview – focus on X-ray waveguides
3. Recent advances at Paul Scherrer Institut
4. 'Super-Resolution' coherent X-ray microscopy
& characterization of focused wave-fields

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

X-ray Focusing – Why

full-field microscope

- + fast
- only x-ray transmission signal

scanning microscope

- + many detection schemes possible (elemental & chemical specificity, crystalline ordering, ...)
- requires coherent illumination

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Small-Angle X-Ray Scattering (SAXS) & Scanning Microscopy

Principle:

- Raster scan a focused x-ray beam & record diffraction patterns

Length Scales:

- raster scanning gives access to length scales between 1 micron – 10 nm
- SAXS gives access to length scales between 1 nm – 1 micron

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Small-Angle X-Ray Scattering (SAXS) & Scanning Microscopy

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Outline

1. X-ray focusing – Why
2. **Methods overview – focus on X-ray waveguides**
3. Recent advances at Paul Scherrer Institut
4. 'Super-Resolution' coherent X-ray microscopy
& characterization of focused wave-fields

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X-Ray Focusing - HOWTO

Take a lens: Yes, but ...

Compound refractive lenses [C. Schroer & W. Jark]

Reflective optics

Kirkpatrick-Baez optics [K. Yamamauchi]

Curved reflective multilayers [C. Morawe]

Bragg-reflective optics [F. Foerster]

Capillary optics

Monocapillary optics [L. Pina]

Polycapillary optics [C.A. Mc Donald]

Diffractive optics

Frensel Zone Plates [M. Feser]

Multilayer Laue Lenses [G.B. Stephenson]

...and Waveguides

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X-ray waveguides

X-rays

$$\lambda \sim 0.1 \text{ nm}, d \sim 50 \text{ nm}$$

Transmission
 $\ll 1 \%$

Y. P. Feng et al., Phys. Rev. Lett. 71, 537 (1993);
S. Lagomarsino et al., Appl. Phys. Lett. 71, 2557 (1997);
S. Di Fonza et al., Nature 403, 638 (2000).

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Resonant beam coupling waveguides

F. Pfeiffer et al., Physical Review B 62(24), 16939-16943 (2000)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Field distribution in 1D resonant beam coupling waveguides

F. Pfeiffer et al., Physical Review B 62(24), 16939-16943 (2000)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

2D Waveguides

F. Pfeiffer, et al., Science **297**, 205 (2002)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Farfield pattern of modes in 2D x-ray waveguides

F. Pfeiffer, et al., Science **297**, 205 (2002)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Waveguide based 'Holo-Microscopy' – slide from T. Salditt

ESRF data, 10.4 keV

C. Fuhse et al., Appl. Phys. Lett. 85 (2004) & C. Fuhse et al., Phys. Rev. Lett. (2006)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Outline

1. X-ray focusing – Why
2. Methods overview – focus on X-ray waveguides
3. Recent advances at Paul Scherrer Institut
 - > Fresnel Zone Plate fabrication by EUV inference lithography
 - > Zone-doubling technique for ultra-high resolution FZP
4. 'Super-Resolution' coherent X-ray microscopy
& characterization of focused wave-fields

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Spatial resolution limit in x-ray microscopy

e-beam lithography limits

Isolated
10 — 20 nm lines

Dense
10 — 20 nm lines

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Possible solutions

W. L. Chao et al. NATURE 435 (2005) 1210-1213

Figure 2 | An illustration of the overlay nanofabrication technique for micro zone plate fabrication.

Figure 3 | Scanning electron micrograph of a zone plate with 15 nm outermost zone. Shown in the inset is a more detailed view of the outermost zone. The zonal period, as indicated by the two black lines, is measured to be 30 nm. The zone placement accuracy is measured to be 1.7 nm.

H. C. Kang et al. PRL96 (2006) 127401

The Swiss alternatives:

- FZP fabrication by EUV inference lithography
- Zone-doubled FZP

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FZP fabrication by EUV inference lithography (H. Solak)

work by Sankha S. Sarkar, Yasin Ekinci, and Harun Solak @ PSI

Solak HH, Ekinci Y, Kaser P, Park S

Photon-beam lithography reaches 12.5 nm half-pitch resolution. JOURNAL OF VACUUM SCIENCE & TECHNOLOGY B 25, 91 (2007)

Both semitransparent window and pinhole

Advantages in relation to e-beam lithography:

- Parallel writing
- No Proximity effect
- No Finite pixel size

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FZP fabrication by EUV lithography

work by Sankha S. Sarkar, Yasin Ekinci, and Harun Solak @ PSI

Pinhole Diameter ~ 300 nm

Distance from mask ~ 600 μm

N = 30, D ~ 32 μm

$\Delta r_N \sim 260$ nm

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Zone-doubling for high resolution FZP (slides J. Vila)

work by J. Vila, K. Jefimovs, and C. David @ PSI

Manufacturing advantages:

No alignment required

One single EBL exposure

FZP pattern generation is simple and reproducible!

K. Jefimovs et al., Phys. Rev. Lett. 99, 264801 (2007)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Zone-doubling for high resolution FZP (slides J. Vila)

1) Substrate preparation and e-beam lithography (0.25 duty cycle at the outer regions)

2) Cr mask dry etching in a Cl_2/CO_2 plasma

3) Silicon RIE in a $\text{CHF}_3/\text{SF}_6/\text{O}_2$ plasma

4) Iridium coating by atomic layer deposition

Low refractive index material → Silicon

High refractive index material → Iridium

ALD Deposition @ University of Helsinki
T. Pilvi & M. Ritala

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Zone-doubling for high resolution FZP (slides J. Vila)

FZP with $D = 100\mu\text{m}$, $\Delta r = 20\text{ nm}$

FZP with $D = 100\mu\text{m}$, $\Delta r = 15\text{ nm}$

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Zone-doubling for high resolution FZP (slides J. Vila)

FZP with $D = 100\mu\text{m}$, $\Delta r = 20\text{ nm}$

FZP with $D = 100\mu\text{m}$, $\Delta r = 15\text{ nm}$

20 μm diameter central stops made of 1.5 μm thick layer of Pt by FIB induced deposition.

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STXM @ PoLLux beamline of SLS (*J. Raabe & G. Tzvetkov*)

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STXM @ PoLLux beamline of SLS (*J. Raabe & G. Tzvetkov*)

**15 nm lines/spacings
(30 nm period)**

1.0 keV
photon
energy

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'Super-Resolution' Coherent Scanning X-Ray Microscopy

Principles:

- In standard scanning x-ray microscopy, resolution is limited to probe size
- Collect coherent diffraction patterns while scanning the spot (Ptychography)
- Phase and amplitude of the object can be retrieved with **enhanced resolution**

P. Thibault, M. Dierolf, A. Menzel, O. Bunk, C. David, F. Pfeiffer, SCIENCE 321, 379-382 (2008)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Ptychographic phase retrieval

- W. Hoppe, Acta Cryst. A 25, 508 (1969).
- R. Hegerl, W. Hoppe, Ber. Bunsen-Ges. Phys. Chemie 74, 1148 (1970).

LETTERS TO NATURE

Resolution beyond the 'information limit' in transmission electron microscopy

P. D. Nellist, B. C. McCallum
& J. M. Rodenburg

Cavendish Laboratory, University of Cambridge, Madingley Road,

Title: Conventional resolution of transmission electron microscopy is orders of magnitude larger than the wavelength of the electrons used. Aberrations of the objective lens corrupt spatial information on length scales below a limit known as the point resolution. Methods to correct for lens aberrations¹⁻³ require knowledge of the phase of the waves which make up the image (this constitutes the 'phase problem'). Beyond the point resolution, information can still be transferred by the microscope, but partial coherence of the scattered beams imposes an ultimate limit (the 'information limit') on the resolution of the transferred image information. Here we discuss this limit.

P.D. Nellist et al., *Nature* **374**, 630 (1995)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

A test case: far-field phase retrieval with laser light

M. Dierolf et al., Europhysics News 39, 22 (2008)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

'Super-Resolution' Scanning X-Ray Transmission Microscopy

Experiment at cSAXS beamline, Swiss Light Source, energy 6.8 keV

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'Super-Resolution' Scanning X-Ray Transmission Microscopy

'conventional' STXM analysis of $> 10^4$ diffraction patterns yields 'low-resolution' absorption & phase-contrast images

P. Thibault, et al., SCIENCE 321, 379-382 (2008)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

'Super-Resolution' Scanning X-Ray Transmission Microscopy

STXM
image

5 μm

0

P. Thibault, et al., SCIENCE 321, 379-382 (2008)

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Simultaneous reconstruction of probe & specimen

probe

P. Thibault, et al., SCIENCE 321, 379-382 (2008)

in focus

propagation distance (mm)

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'Super-Resolution' X-Ray Microscopy

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Near future: High-resolution x-ray imaging of cells ?

Email: franz.pfeiffer@psi.ch - Web: <http://people.epfl.ch/franz.pfeiffer>

Summary

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SWISS LIGHT SOURCE

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Swiss Light Source

PAUL SCHERRER INSTITUT
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Christian David, Joan Vila, Konstantins Jefimovs, Vitaliy Guzenko, Harun Solak, Sankha Sarkar,
*Laboratory for Micro- and Nanotechnology,
 Paul Scherrer Institut, CH*

X-ray focusing with Kirkpatrick-Baez optics

K. Yamauchi
Osaka University

Contents

1. Introduction
2. Accuracy criteria to realize 50nm-level focusing
3. Fabrication technology (@Osaka University)
4. Recent achievements
5. Challenges to realize sub-10nm focusing in hard X-rays
6. Other topics including XFEL optic
7. Summary

Advantages of KB mirror optic

Fresnel zone plate

Refractive lens

K-B mirror optics

Achromatic optic (total reflection mirrors)

High efficiency >90%

Large aperture $\approx 500\mu\text{m}$

Long working distance 10mm $\sim$ 500mm

Recent achievement in 50nm-level focusing

90nm x 90nm, 45nm focusing were achieved at **ESRF** by a graded multilayer coating and a fine bending system

Efficient sub 100 nm focusing of hard x rays

O. Hignette, P. Cloetens, G. Rostaing, P. Bernard and C. Morawe
Rev. Sci. Instrum. 76, 063709 (2005)

75nm x 85nm focusing was achieved at **APS** by an optical figuring and a differential deposition.

Short focal length Kirkpatrick-Baez mirrors for a hard x-ray nanoprobe

W. Liu, G. Ice, J. Tischler, A. Khounsary, C. Liu, L. Assoufid
and A. Macrander, Rev. Sci. Instrum. 76 (11), 2005, p.113701

36nm x 48nm, 25nm focusing were achieved by **SPring-8** and **Osaka Univ.** group by EEM, P-CVM, MSI and RADSI.

H. Mimura et al., Hard X-ray Diffraction-Limited Nanofocusing with Kirkpatrick-Baez Mirrors,
Japanese Journal of Applied Physics Part 2, 44 (18), L539-L542 (2005).

H. Mimura et al., Efficient focusing of hard x rays to 25 nm by a total reflection mirror, Applied
Physics Letters 90, 051903 (2007)

Ch. Morawe et al,
Proc. SPIE 6317 (2006)

Required accuracy for nano-focusing under D-limited condition

Kirkpatrick-Baez mirrors

Elliptical mirror $\times 2$

Diffraction-limited focusing

40mm < Ls < 50mm

100mm < Ls < 150mm

Designed profile (ellipse)

Beam profile

© Plasma CVM (chemical vaporization machining)

→ Rough figuring (Rapid figuring with sub-10nm (P-V) accuracy)

K. Yamamura et al., Rev. Sci. Instrum. 71 (2000), 4627

© EEM (elastic emission machining)

→ Final figuring and smoothing (Fine figuring and atomic smoothness)

K. Yamauchi et al., Rev. Sci. Instrum. 73 (2002), 4028

© MSI (microstitching interferometry)

→ Figure tester with spatial resolution close to $1\mu\text{m}$

K. Yamauchi et al., Rev. Sci. Instrum. 74 (2003), 2894

© RADSI (relative-angle determinable stitching interferometry)

→ Figure tester for steeply curved ellipse of large NA mirror

H. Mimura et al., Rev. Sci. Instrum. 76 (2005), 045102

J-tec URL <http://www.j-tec.co.jp>

Plasma CVM (Chemical Vaporization Machining)

*A chemical removal process utilizing reactive species generated in the **atmospheric pressure plasma***

- *High density reactive species $\Rightarrow$ High removal rate*
- *Chemical reaction $\Rightarrow$ No damage on the surface*
- *Non contact $\Rightarrow$ Insensitive against external disturbanc*

Material	Removal rate ($\mu\text{m}/\text{min}$)
Fused silica	170
Silicon	94
Molybdenum	36
Tungsten	32
Silicon carbide	6.4
Diamond	2.5

Plasma CVM (continued)

Atmospheric pressure plasma

Plasma is localized around the electrode

High spatial resolution figuring is possible without mask

Pipe electrode is utilized for

high spatial resolution figuring

Rotary electrode is utilized for

high-efficiency machining

Figuring by Numerically Controlled Plasma CVM

isc-type rotary electrode

Plasma

or

Shape of the removal spot

Removal shape

Removal spot

Convolution

$\otimes$

wellin ti

Removal depth is proportional to dwellin time so that figuring is controlled by scanning speed.

EEM (Elastic Emission Machining)

The ultra-fine particle is supplied to the work surface by ultrapure water flow

In EEM, chemical reaction between sol surfaces is utilized.

Atom removal occurs selectively at the top site of the work surface

SiO_2 powder particle

Bump site is preferentially removed

Atomically flat surface can be obtained

Removal mechanism is verified to be chemical by first-principles molecular dynamics simulation

Surfaces smoothing properties in EEM

P-V 1.603 nm
RMS 0.183 nm

Pre-machined surface

P-V 0.910 nm
RMS 0.100 nm

EEM surface (Removal depth:2nm)

The roughness of about 2nm(P-V) can be flattened with the removal of 2nm thickness.

The bump site is selectively removed.

PSD analysis

P-V 0.769 nm
RMS 0.087 nm

EEM surface (Removal depth:2nm)

STM image of EEM processed surface

Distribution of atom
classified for every
atomic layer

1st layer (0.034%)

STM image (100nm x 100nm)

2nd layer (1.4%)

3rd layer (16.0%)

4th layer (47.0%)

5th layer (30.7%)

6th layer (4.0%)

Others (0.1%)

EE pattern S M ima e
(20nm x 20nm)

95% of the EEM processed
surface is constructed with
only 3 atomic layers.

Typical figuring properties using EEM

Sub-30nm focusing

Designed configuration

< Wave-optically expected beam profile >

Focusing performance

Beam waist structures

Focal point

100 μ m

Measured profile

Calculated profile
using measured shape

Tunability of beam size and photon flux

Photon flux:

$$5 \times 10^9 \sim 10^{12} (1/s)$$

Installed in a new hutch of BL29

emonstration of oomin performance

Mouse cell tubulin was stained with nanocrystals of CdSe/ZnS.

“Hard-X-ray sub-10nm focusing By KB mirrors”

Kirkpatrick-Baez mirror pair

To realize Sub-10nm focusing K-B mirrors

X-ray energy : 20keV
Focal length : 150mm
Acceptance width : 1.1mm
Incidence angle : 11.1mrad

7nm

MIS with RADSI and EEM can prepare
surface figure within 1~2nm (P-V) error

Required accuracy

@20keV Mirror length: 100mm, Focal length: 150mm

Figure error of 1nm is not allowable in this case

Off-line figure testing might be impossible?

Multi-layer technology is needed to realize large NA

Graded multi-layer

Reflectivity

Not only figure error but also thickness deviation of the multilayer induce the wavefront phase error.

At-wavelength phase-retrieval interferometry

Phase retrieval properties

On focal plane

Intensity is changed to experimental value.

Phase is kept to be recovered value.

On mirror surface

Intensity is changed to theoretical value.

Phase is kept to be recovered value.

Wavefront errors

Intensity profile

Iteration 10

Wavefront errors

Intensity profile

Iteration 1000

New knife-edge method

Conventional knife-edge method

New method

Details of the new knife-edge method

Scanner is acting as a phase object

Phase is shifted with $\lambda/2$

Microroughness at the bridge

A-A' profile

A demonstration of at-wavelength measurement (30nm-focusing mirror was employed)

FIG. 3. Results of measuring intensity profiles in the focal plane and phase retrieval calculations. **A** Intensity profiles in the focal plane. The black line is the profile measured by scanning the microbridge, while the red line was obtained using phase retrieval calculations for determining the mirror surface profile. The plot interval is 1 nm. **b** Single logarithmic plot of graph in a. **c** Ideal profile of x-ray mirror. **d** Comparison of measured and reconstructed figure error profiles.

To avoid local-minimum problem

Calculated and measured intensity profiles on A-A' line.
(50 μm upstream from the focal point)

On-line compensation of wavefront

Focusing mirror with phase error

In-situ phase compensation

Sub-10nm

Piezo-electric
phase compensator

Focusing mirror with phase

Designing 1 (reduction of required accuracy)

$$\text{Phase error} = 2kd \sin \theta$$

d : Shape error
 θ : Glancing angle
 $k=2\pi/\lambda$: Wave number

An example

Glancing angle of
10nm-level focusing mirror: 7 [mrad]
Glancing angle of
active mirror: 1.0[mrad]

★ Required figure accuracy of the 10nm-level focusing mirror is 0.7nm (PV).

★ Glancing angle of the active mirror is 7 times smaller than that of the focusing mirror

Required figure accuracy of the active mirror becomes 4.9nm (PV).

Designing 2 (How many waves should be generated by the active mirror?)

Spatial wavelength of the figure error and the position of the satellite peak are wave-optically correlated.

Shape error profiles

Phase compensator

Optical interferometer

Optical configuration of 10nm-level focusing with AM

Compensator (AM: Active mirror)

Achieved beam size at Spring-8

400mm-long mirror for XFEL

WD: 350m

Conclusion

1. Mirror optic has many advantages against the other optics, such as long WD, no chromatic aberration (in case of total reflection mirror), high focusing-efficiency, and relatively large aperture.
2. Diffraction-limited focusing performance has been already realized in mirror focusing.
3. Sub-50nm focusing mirrors become ordinary devices.
4. Phase retrieval interferometry will become a possible technique for the mirror surface testing.
5. Active mirror can control wavefront phase with 0.1λ -level accuracy.
6. 10nm-level X-ray beams will be realized in the near future.

Acknowledgement

Co-workers

. Mimura^a, . Numoto^a, S. Matsuyama^a, S. Iwanda^a, . Kimura^a, . Sano^a, K. Yamamura^a,
. Ishino^b, M. Tabashi^c, K. Yamashita^b, and . Ishikawa^{b,c}

a: Osaka University b: RIKEN /SPRING-8

c: ASRI/SPRING-8

This research was supported by

A Grant-in-Aid for Specially Promoted Research 18002009, 2006 from the Ministry of Education,
Sports, Culture, Science and Technology, Japan,

A 21st Century COE Research, Center for Atomistic Fabrication Technology, 2003 from the
Ministry of Education, Sports, Culture, Science and Technology, Japan,

A Global COE Research, Center for Atomically Controlled Fabrication Technology, 2008 from the
Ministry of Education, Sports, Culture, Science and Technology, Japan,

The use of BL29XU of the SPRING-8 was supported by RIKEN.

Hard X-ray focusing with curved reflective multilayers

Ch. Morawe, ESRF (France)

Outline:

- Basic focusing considerations
- Theoretical models
- Multilayer properties
- Technological options
- Experimental progress
- Summary

Basic considerations

Diffraction limit $D_{FWHM} = C \frac{\lambda}{NA}$

Numerical aperture $NA = n \cdot \sin \varepsilon$

Straight aperture $C = 0.44$

Source size limit $D = \frac{q}{p} \cdot S$

Further physical limitations

- Volume diffraction
- Scattering

Technological limitations

- Non-trivial design
- Fabrication accuracy
- Alignment

Basic considerations

Ellipse geometry

$$2\varepsilon = \varphi_q(\theta_2) - \varphi_q(\theta_1)$$

$$\sin \varphi_q = \frac{p \cdot \sin 2\theta}{2 \cdot c}$$

$$NA = n \cdot \sin \varepsilon$$

Correct only for flat aperture !

Basic considerations

Simple approximation:

a) Total reflection mirror

$$\sin \varepsilon \approx \frac{1}{4} \sin \theta_c = \frac{\sqrt{2 \cdot \delta}}{4} = \frac{\lambda}{4} \sqrt{\frac{r_0 \rho_e}{\pi}}$$

$$\Rightarrow D_{FWHM} \approx 1.76 \cdot \sqrt{\frac{\pi}{r_0 \rho_e}}$$

$$D_{FWHM} \approx 25 \text{ nm (Pt)}$$

b) Multilayer mirror

$$\sin \varepsilon = \frac{1}{4 \cdot c} (p_2 \cdot \sin 2\theta_2 - p_1 \cdot \sin 2\theta_1)$$

$$\sin 2\theta \approx 2 \cdot \sin \theta \approx \frac{\lambda}{\Lambda}, p \approx 2 \cdot c$$

$$\Rightarrow \sin \varepsilon \approx \frac{\lambda}{2} (1/\Lambda_2 - 1/\Lambda_1)$$

$$\Rightarrow D_{FWHM} \approx \frac{0.88}{1/\Lambda_2 - 1/\Lambda_1}$$

$$D_{FWHM} \approx 5 \text{ nm}$$

No explicit energy dependence !

Theoretical models

Geometrical phase shift due to height/slope errors

$$\varphi_h = \frac{4\pi \cdot \sigma \cdot \sin \theta}{\lambda} \quad \sigma: \text{height error}$$

Reasonable values for ML mirrors with 80% flux in focal spot

$$\sigma_{RMS} \leq \frac{\lambda}{27 \cdot \sin \theta} \approx \frac{\Lambda}{13.5} \Rightarrow \sigma_{RMS} \ll 1 \text{ nm}$$

O. Hignette et al, SPIE 4501 (2001)

Similar results from wave optical simulations

H. Yumoto et al, SPIE 6317 (2006)

Challenge for fabrication and metrology !

Theoretical models

Geometrical ray tracing

- Analytical approach
- Parabolic/elliptic shape
- $ML \cong$ Two-interface slab
- Linear approximation for refraction
- Simple expressions for caustic and beam intersections

Goals

- Caustic shape
- Beam intersections
- Chromatic behavior

J-P. Guigay et al, Opt. Express 16, 12050 (2008)

Theoretical models

Caustic and beam intersections

- x and y diverge at grazing incidence
- Refraction and penetration amplify the effect
- Reduced aberration for increased angles of incidence
- Order of magnitude:

$$\Delta x \leq 1000 \text{ nm}$$

$$\Delta y \leq 10 \text{ nm}$$

Theoretical models

Exact ray tracing

- Snell's law
- No approximations
- Good agreement with analytical model
- Linear approach for refraction fails near critical angle
- Agreement ESRF – Osaka

H. Mimura et al, SPIE 67050L (2007)

Theoretical models

Chromaticity

$$\delta \sim \frac{1}{E^2} \rightarrow \left| \frac{df}{dE} \right| = 2 \cdot \frac{\Delta f}{E}$$

$$D_{tot}^2 = D_{diff}^2 + D_{chrom}^2 \stackrel{!}{\leq} 2 \cdot D_{diff}^2$$

	FZP	CRL	RML
$\frac{dE}{E}$	$\frac{D_{tot}^2}{1.76 \cdot f \cdot \lambda}$	$\frac{D_{tot}^2}{3.52 \cdot f \cdot \lambda}$	$\frac{D_{tot}^2}{3.52 \cdot \Delta f \cdot \lambda}$

Secondary effect on focus !

Theoretical models

Are we already doing better ?

- ML design via corrected Bragg law

$$\Lambda = \frac{\lambda}{2\sqrt{n^2 - \cos^2 \theta}} \left(\approx \frac{\lambda}{2 \cdot \sin \theta} \right)$$

- Refraction implicitly considered
- ML interface shapes **not elliptic** (except for surface layer)
- Difficult analytical access
- Aberrations reduced/suppressed ?

Need for wave optical calculations !

Theoretical models

Alternative approach

- ML design via numerical simulation (Osaka University)
- ML ray tracing and optical path optimization
- Equivalent to corrected Bragg equation (?)

$$\Lambda = \frac{\lambda}{2\sqrt{n^2 - \cos^2 \theta}}$$

H. Mimura et al, SPIE 67050L (2007)

Theoretical models

Results of numerical optimization

H. Mimura et al, SPIE 67050L (2007)

Single deep reflection

Multiple shallow reflections

Focal spot blurring < 1 nm !

Theoretical models

Wave optical simulations

- Exist for CRLs, FZPs, and MLLs
 - predict diffraction limited focusing
 - spot size down to nm dimensions
- Not yet available for reflecting MLs
- Space for future investigation

PhD project ESRF/Univ.Göttingen

PhD Thesis Student (m/f)

Subject: “Wave optical simulations for x-ray nano-focusing optics”

Place of Work: ESRF Grenoble (France) / University of Göttingen (Germany)

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Ref. CFR320 - Deadline for returning application forms: 30 September 2008

Multilayer properties

Numerical calculations: Dynamical theory of flat MLs

- Bragg peaks and fringes due to interference
- Positions depend on E and Λ
- Intensities depend on $\Delta\rho$, N , σ ...

Corrected Bragg equation

$$m \cdot \lambda = 2 \cdot \Lambda \cdot \sqrt{n_2^2 - n_1^2 \cos^2 \theta}$$

For $\theta \gg \theta_c \rightarrow m \cdot \lambda \approx 2 \cdot \Lambda \cdot \sin \theta$

L.G. Parratt, Phys. Rev. 95, 359 (1954)

Multilayer properties

Materials choice – Basic rules:

1. Select low-Z spacer material with lowest absorption (β_{spacer})
2. Select high-Z absorber material with highest reflectivity with spacer ($\delta_{\text{abs}} - \delta_{\text{spacer}}$)
3. In case of multiple choices select high-Z material with lowest absorption (β_{abs})
4. Make sure that both materials can form stable and sharp interfaces (lower d-spacing limit)

Computational search algorithms

- Soft X-rays: A.E. Rosenbluth (1988)
- Hard X-rays: K. Vestli (1995)

K. Vestli et al, Rev. Sci. Instr. 67, 3356 (1996)

Multilayer properties

Period number N:

Peak versus integrated reflectivity:

- R_{peak} increases with N up to extinction
- $\Delta E/E$ decreases $\sim 1/N$ in kinematical range
- R_{int} is maximum before extinction

High and low resolution MLs

Optimize N according to needs !

Multilayer properties

Filling factor $\Gamma = t_{\text{abs}}/\Lambda$

- Harmonics suppression
- Reflectivity enhancement

Optimum Γ for large N

A.V. Vinogradov et al, Appl. Opt. 16, 89 (1977)

$$\tan(\pi \cdot \Gamma_{\text{opt}}) \approx \pi \cdot \left(\Gamma_{\text{opt}} + \frac{\beta_{\text{abs}}}{\beta_{\text{abs}} - \beta_{\text{spacer}}} \right)$$

Best Γ drops with growing N !

Multilayer properties

Non-periodic stacks:

- Ni/B₄C structure
- $R(\theta) = \text{const}$ over 20% bandwidth

Ch. Morawe et al, Nucl. Instr. and Meth. A 493, 189 (2002)

Theoretical design

Experimental result

Multilayer properties

Multilayer properties

Multilayer properties

Energy dispersion:

Bragg equation:
$$E(\theta) = \frac{h \cdot c}{2\Lambda \sqrt{n^2(E) - \cos^2 \theta}}$$

Elliptic mirror:
$$\sin^2 \theta = \frac{b^2}{p \cdot q} \quad p + q = 2 \cdot a$$

$$E(q) = \frac{h \cdot c}{2\Lambda \sqrt{n^2(E) - 1 + \frac{b^2}{(2a - q)q}}}$$

Dispersion “along ML mirror”

Multilayer properties

Intensity profiles: (Kirkpatrick-Baez multilayer optics on ESRF BM05)

Ch. Morawe et al, SPIE 5537, 115 (2004)

Technological options

Deposition techniques	Vacuum	Particle energy	Deposition rate	Deposition area
Thermal evaporation	HV (UHV)	Low	Low	Small
E-beam evaporation	UHV	Low	Low	Small
Magnetron sputtering	HV (+Gas)	High	High	Large
DECR sputtering	HV (+Gas)	High	Low	Medium
Ion beam sputtering	UHV (+Gas)	Very high	High	Medium
Pulsed laser deposition	HV	Very high	High	Medium

- **Characteristics may vary depending on equipment and application**
- **Magnetron sputtering most widely used for X-ray multilayer fabrication**
- **High particle energy favors very thin and uniform layers**

Technological options

Large area coatings (uniform, gradient)

- Relative motion source - substrate
- Masking techniques

$$t(\vec{r}) = \int \varphi(\vec{r}, \vec{r}') \frac{d\vec{r}'}{v(\vec{r}')}$$

Technological options

Technology and engineering

Curved MLs

- Figured substrates or bending techniques ?
- Surface finishing (deterministic polishing/etching/coating)

Stability and stress

- Intrinsic stress after coating
- Thermal and radiation load (white beam)
- Sample environment (vacuum/He/N₂)

Metrology

- Ex-situ techniques reaching limits
- On-line metrology (intensity, phase retrieval)
- Phase correction elements

Several solutions commercially available!

APS profile coating

ESRF bender

Osaka EEM technology

Experimental progress

Example: $[\text{W/B}_4\text{C}]_{25}$ ML @ 24550 eV

Experimental progress

ESRF focusing experiment

- Full undulator spectrum
- $P = 150$ m, $Q = 77$ mm
- Vertical line focus
- Raw data **45 nm** FWHM @ $100\text{ }\mu\text{m}$ aperture

Ch. Morawe et al, Proc. SPIE 6317 (2006)

Experimental progress

ID19: low β @ 150 m, $E = 15...24$ keV
 86×83 nm²: 2×10^{11} ph/s @ 80 mA

ID22: high β @ 60 m, slit source, $E = 17$ keV
 76×84 nm²: 10^9 ph/s @ 200 mA
 150×100 nm²: 10^{12} ph/s @ 200 mA

3×10^5 ph/s/mA/nm²

ESRF BLs since 1998:

23 focusing devices

37 ML coatings

P. Cloetens, O. Hignette, ESRF

Summary

ML - KB “Data Sheet”

Energy range	5...100 keV
Peak reflectivity	50...90% (per reflection)
Energy bandpass	0.5...20%
Minimum focal spot size	≈ 5 nm (expected diffraction limit) < 50 nm (proof of feasibility) < 100 nm (routine operation)
Focal distance	50...1000 mm
BL layout	Beam deflection (horizontal + vertical)
Alignment	Pre-alignment + on-line (recommended)
Available technologies	Static (fixed energy) Dynamic (tunable energy)
Principal curved ML developers	ESRF, Univ.Osaka/Spring-8, (APS)
Synchrotron optics (no MLs)	Irelec (France), JTEC (Japan), SESO (France), Xradia (USA), Zeiss (Germany)
Lab optics or coatings only	AXO (Germany), Incoatec (Germany), Osmic/Rigaku (USA), WinlightX (France), Xenocs (France)

Refractive x-ray lenses for hard x-ray microscopy

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Hard X-Ray Microscopy & Tomography

Full-field microscopy:

- transmission imaging
- contrast generated by attenuation and refraction
- large 3D-images of the sample (tomography)

Scanning microscopy:

- scan sample with nanobeam (< 100 nm lateral size)
- different contrasts:

- fluorescence
- absorption (XAS)
- diffraction (SAXS, WAXS, CXDI)

...

- scanning: relatively slow

- tomography: local inner structure of sample

K

Optics for Hard X-Rays

Full-field microscopy:

- objective lens for imaging free of aberrations
- condensor lens to concentrate x-rays on sample

Scanning microscopy:

- generate an intensive x-ray microbeam

Variety of x-ray optics available today:

- Fresnel zone plates and multilayer Laue lenses

- refractive lenses [Snigrev, et al., Nature **384**, 49 (1996)]

- curved/bent mirrors and multilayers

- capillaries

- wave guides (mode filter)

- crystal optics

...

Refraction

$$n = 1 - \delta + i\beta, \quad \delta > 0$$

Vacuum optically denser than matter!

$$\delta = \frac{1}{2\pi} N_A r_0 \lambda^2 \rho \frac{Z + f'}{A}$$

specific refraction:

independent of material
(away from absorption edges)

very weak

Absorption

$$n = 1 - \delta + i\beta, \quad \delta > 0$$

Lambert-Beer Law:

$$I(x) = I_0 e^{-\mu x}$$

attenuation coefficient μ :

$$\mu = \frac{4\pi\beta}{\lambda}$$

2 main contributions:

photo absorption $\tau \sim Z^3/E^3$

Compton scattering μ_c

For comparison: $\mu_{\text{glas}} = 10^{-7} \text{ cm}^{-1}$
for visible light

Refractive X-Ray Lenses

single lens

stack of lenses:
compound refractive lens (CRL)

$$R = 50 \mu\text{m} - 1000 \mu\text{m}$$

$$d = 10 \mu\text{m} - 30 \mu\text{m}$$

$$2R_0 = 450 \mu\text{m} - 1000 \mu\text{m}$$

variable number of lenses: $N = 10 \dots 300$

parabolic profile: no spherical aberration

→ true imaging optic

Parabolic Refractive X-Ray Lenses

Bruno Lengeler
RWTH Aachen

Full-Field Imaging

lenses used as
objective lens in full
field microscope

image distance

$$L_2 = \frac{L}{L_1}$$

numerical aperture

$$NA = \frac{D}{2L}$$

Full-Field Imaging

For comparison:
spherical lens

(simulation)

imaging parameters:

$$\begin{aligned} E &= 12\text{keV} \\ N &= 91 \text{ (Be)} \\ f &= 495 \text{ mm} \\ m &= 10\times \end{aligned}$$

spatial resolution:

$$\sim 100 \text{ nm}$$

Full-Field Imaging: Spatial Resolution

expected resolution: 84 nm

line profile

(SPIE 2002)

→ resolution of x-ray optical setup: $105 \text{ nm} \pm 30 \text{ nm}$

Hard X-Ray Microbeam

source

Focus size and shape determined by:

source size
magnification L_2/L_1
diffraction limit
aberrations

L_1

lens

Flux in focus determined by:

brilliance
source size

focusing cross-section of lens

$$L_2 = \frac{L_1 f}{L_1 - f}$$

microbeam
on sample

ERL

High brilliance:

High flux per phase space volume

Ideal for nanobeams:

small source:

small geometric image
(diffraction limited focusing)

small divergence:

optic captures large fraction
of emitted radiation

Be-Lens with $R = 50\mu\text{m}$: Microbeam

vertical focus

200nm

ESRF ID10

$$E = 8 \text{ keV}$$

$$N = 31$$

$$L_1 = 60.8 \text{ m}$$

$$f = 156 \text{ mm}$$

$$\text{gain} \sim 10^5$$

$$\text{flux: } 3 \cdot 10^{11} \text{ ph/s}$$

mono: Si 111

expected focus size: 170 nm

horiz focus: $1.14 \mu\text{m}$
(horizontal slits at 0.3mm gap)

focus source size and stability limited!

Rotationally Parabolic Refractive X-Ray Lenses

State-of-art (Be, Al):

$R = 50 \text{ } \mu\text{m}$ [tested for focusing $< 200 \text{ nm}$ (source size limited)]

$R = 200 \text{ } \mu\text{m}$ [tested for full field imaging with $\sim 100 \text{ nm}$ resolution]

$R = 300 \text{ } \mu\text{m}$, $500 \mu\text{m}$, and $1000 \mu\text{m}$ [not tested, yet]

Lenses with $R = 1500 \text{ } \mu\text{m}$ under development

Energy range: 5 - 150 keV and higher

Application:

hard x-ray full-field microscope (tomography)

microbeam analysis, e. g., micro-fluorescence, XANES, SAXS
also in tomography

coherent (micro-)diffraction

beam conditioning (moderate focusing, collimation)
(white beam compatible)

→ Optics for X-FEL

Microprobe Example: SAXS-Tomography

Probe nanoscale structure on a virtual section through a sample

Sample:

nondestructive probe of the interior of sample

define virtual slice

obtain SAXS cross section at each location on section

APL **88**, 164102 (2006)

injection molded PE

Collab.: N. Stribeck, Univ. Hamburg

SAXS-Tomography

$$I_{\vec{q}}(r)$$

101 projections with
70 steps each
80μm step size

SAXS-Tomography

Reconstruction:

attenuation

diffraction

integral
scattering
along rotation
axis

$$q_r = 0$$

SAXS-Tomography

Sample is fibre textured:
scattering cross section

scattering cross section

inhomogeneous
nanostructure

In each pixel:
full scattering
cross section
(rotationally
symmetric)

APL **88**, 164102 (2006)

interpretation:
Stribeck, et al.
Macromol. Chem.
Phys. 207, 1139 (2006)

Effective Aperture and Diffraction Limit

Numerical aperture:

D_{eff} limited by:

geometric aperture $2R_0$
attenuation inside lens material
(includes Compton scattering)

→ low Z lens material

$$NA = \sin \alpha = \frac{D_{\text{eff}}}{2L_2}$$

Diffraction limit:

$$d_t = 0.75 \cdot \frac{\lambda}{2NA}$$

Numerical Aperture

large f : aperture dominated by attenuation

$$D_{\text{eff}} = 4\sqrt{\frac{f\delta}{\mu}} \propto \sqrt{f}$$

- reduce μ/δ (low Z lens material)
- $NA = D_{\text{eff}}/2f \propto 1/\sqrt{f}$: reduce focal size to minimum

Nanofocusing Lenses (NFLs)

nanolens

strong lens
curvature:
 $R = 1\mu\text{m} - 5\mu\text{m}$
 $N = 35 - 140$

lens made of Si by e-beam litho-
graphy and deep reactive ion etching!

APL **82**, 1485 (2003)

Crossed Nanofocusing Lenses

Setup at ID13 of ESRF

Focusing with NFLs

Si lens: $E = 21\text{keV}$, $L_1 = 47\text{m}$

source:

ID13 low- β invac. undulator

source size: $150 \times 60\mu\text{m}^2$

vertical focus: 55nm

horizontal focus: 47nm

$f = 10.7\text{mm}$

demagnification:
 $\sim 2400 \times 4400$

flux: $1.7 \cdot 10^8\text{ph/s}$

APL **87**, 124103 (2005)

2D Scanning Mode: X-Ray Fluorescence

Arabidopsis thaliana

Fluorescence map

pollen

~ 100 nm resolution

$E = 15.25 \text{ keV}$

High-Resolution Fluorescence Microtomography

Arabidopsis thaliana

trichome (leaf hair)

tip of trichome
(freeze dried)

Energy:
24.3keV
focus size:
80nm x 120nm
pixel size:
100nm

Nano-Diffraction

User experiment at ID13 carried out with prototype (Feb 2008)

M. Hanke, et al., APL **92**, 193109 (2008)

Scan single SiGe/Si(001)-islands

(a) Ge fluorescence

(b) light micrograph

Beam parameters:

$E = 15.25 \text{ keV}$

beam size: $200 \times 200 \text{ nm}^2$

flux: $> 10^9 \text{ ph/s}$

facet-rods

Map of diffuse scattering around
Si(004)-reflection

Limits of Focusing with NFLs

Diffraction limit:

$$N = 100$$

$$I \geq 0.084$$

$$R = 0.5 - 50 \mu\text{m}$$

NA limited by

$$\sqrt{2\delta}$$

adapt aperture to
converging beam (AFLs):

$$\text{focus} < 5 \text{ nm}$$

[PRL **94**, 054802 (2005)]

Further improvement of focus size with diffractive optics (e. g., MML).

Nanoprobe: Coherent Nanodiffraction

$$E = 15.25 \text{ keV}$$

$$\lambda = 0.813 \text{ \AA}$$

beam size:
< 150 x 150 nm
(amplitude)

Wave Front in Diffraction Limited Focus

divergence angle:
numerical aperture

$$d_t = \frac{2\sqrt{2\ln 2}}{\pi} \frac{\lambda}{2NA} \approx 0.75 \frac{\lambda}{2NA}$$

Gaussian limited plane wave

CXDI

XPCS, XFCS

Coherence in Focus

Focus size (amplitude):

$$b_{\text{ampl}} = \sqrt{2b_{\text{geo}}^2 + 2d_t^2}$$

lateral coherence length:

$$l_t = 2d_t \sqrt{1 + \frac{d_t^2}{b_{\text{geo}}^2}}$$

So Far: No Ideal Lens...

Shape errors:

Underetching & proximity effect:

Roughness:

deviation from parabola

~ 10 mrad

tilted side wall

Shape of Wave Front in Focus

Main result:

beam flat in central
speckle

In general:

Speckle size in focal
plane can not be finer
than diffraction limit!

Test Object: Gold Particle on Si₃N₄-Membrane

size < 100 nm

Diffraction Pattern of Gold Nanoparticle

sample-detector distance:
1250 mm (in air)

detector:
FReLoN 4K
50 μ m pixel size

exposure time:
10 x 60 s

intensity on sample:
3300 ph/s/nm²

integral dose in beam:
10¹¹ ph > 2 month in fla
coherent beam

compared to 10¹² ph/pulse
at XFEL

Reconstruction

reconstruction by HIO

shrink-wrap

phase left free to evolve

200 reconstructions

(9 converged to wrong solutions)

averaged solution:

reconstructions:

left-handed

right-handed

Conclusion

Refractive x-ray lenses:

- high resolution imaging optics

- small numerical apertures ($\sim$ mrad): aspherical lens is parabolic

- spatial resolution ~ 100 nm and below for different designs

- diffraction limits (theoretical):

 - < 20 nm for regular design (all lenses same size)

 - ~ 5 nm for adiabatically focusing lenses

 - (lens size adapted to converging beam)

- diffraction limited focusing: high degree of coherence in focus

Applications:

- full-field microscopy

- scanning microscopy (fluorescence, diffraction, absorption)

- coherent diffraction imaging

- combine scanning microscopy with coherent diffraction imaging:

- ptychography [Thibault, et al., Science **321**, 379-382 (2008)]

Kinoform x-ray lens arrays

Werner Jark

Sincrotrone Trieste
Basovizza (TS), Italy

werner.jark@elettra.trieste.it

[www.elettra.trieste.it/experiments/
beamlines/microfluo/index.html](http://www.elettra.trieste.it/experiments/beamlines/microfluo/index.html)

[www.elettra.trieste.it/organisation/
experiments/laboratories/
multilayer_technology/index.html](http://www.elettra.trieste.it/organisation/experiments/laboratories/multilayer_technology/index.html)

Modern Developments in X-Ray and Neutron Optics
Series: Springer Series in Optical Sciences , Vol. 137
Erko, A.; Idir, M.; Krist, Th.; Michette, A.G. (Eds.)
2008, XXIII, 533 p. 299 illus., 5 in color.
With series ad on (virtual) p. 535, 536.. Hardcover

ISBN: 978-3-540-74560-0

Read on CLESSIDRA kinoform lenses
on pages 331-351

What happens here?

μ XFA@ELETTRA
BL 10.1L

Cu wire
100 μ m

my hair
60 μ m

Cu
30 μ m

my hair
60 μ m

3.7 keV

my hair
70 μ m

11.8 keV

transparent hairs can deviate (i.e. refract) x-rays

Refraction and reflection: x-rays

- Snell's law: $n \sin \alpha = \text{const}$ $\cos \phi_0 = (1 - \delta) \cos \phi_1$
- Beam deviation at one interface: $\Delta = \phi_0 - \phi_1$
- with $\cos \Delta = 1$, $\sin \Delta = \Delta$: $\cos \phi_1 = \cos \phi_0 + \Delta \sin \phi_0$ then $\Delta = \delta / \tan \phi_0$

Refraction in transmitted x-rays

$n > 1.0$: convex
lens focuses

$n < 1.0$: concave
lens focuses

We just heard it!

Refraction in transmitted x-rays

Back to hair and Cu wire

Refraction in transmitted x-rays

Equation for parabola

$$2R(x-x_0)=y^2$$

focal length

$$f=R/(2\delta)$$

Parabolic material increase

Gaussian transmission function

$$T=\exp(-y^2/(2\delta fL))$$

with L =attenuation length

Refraction in transmitted x-rays

$$T = \exp(-y^2 / (2\delta f L))$$

Optimum aperture

$$A = 2\sqrt{2\delta f L}$$

Average T is 75%

Figure of merit
 of materials
 $\sqrt{\delta L}$

Refraction in transmitted x-rays

Figure of merit $\sqrt{\delta L}$

Difficult Li, very interesting
Be, but also plexiglass
(pmma)

Compton scattering
dominates at higher
energies (>30 keV):
all materials have similar
figure of merit

Can we “lighten” the lens?

A.J. Fresnel made the CONVEX lenses for the Cordouan lighthouse in 1822 lighter

Strategy: Refraction only on
transmission through inclined surfaces

remove “useless” rectangular blocks

increase angles by refraction – reflection – refraction in prisms

To “lighten” a CONCAVE lens

History:

Prior to introduction of compound refractive lenses (CRL)!

Suehiro et al, Nature 352, 385 (1991): proposal in letter without picture (lathe)

Michette, Nature 353, 510 (1991): critically comments the idea

Yang, NIM A328, 578 (1993): more detailed elaboration

T. Tomie, “X-ray lens”,
Japanese patent 6-045288
(18 Febr 1994)

Covering compound
refractive lenses and
“lightened” lenses

Never made this way

First realisation:
Aristov et al, APL 77,
4058 (2000)

To "lighten" a CONCAVE lens

Without restrictions?

Tomie 1994

Restrictions

Do not distort the passing wave
Keep planes of equal phase continuous

Make use of longitudinal field periodicity:

**Remove blocks, which shift phase by
integer multiple of 2π !**

$$D = m\lambda/\delta$$

m = integer multiple

λ = wavelength

δ = refractive index

Reduced material!!

First prototype: Aristov et al, APL 77, 4058 (2000)

These are kinoform lenses produced by lithography

$D = m\lambda / \delta \approx 40 \mu\text{m}$
For Si@12 keV

later: Evans-Lutterodt et al, Opt. Express 11, 919 (2003), Nöhammer et al, JSR 10, 168 (2003), Nazmov et al, NIM B217, 409 (2004), Alianelli et al, SPIE Proc. 6705, art. no. 670507 (2007) (single in Si)

Reduced material!!

Dimensions: make very good use of chip mass production techniques

BUT lithography can only shape linear structures

Need crossed lens pair for bi-dimensional focusing:
match height and aperture!!

$$D = m\lambda / \delta \approx 40 \mu\text{m}$$

For Si@12 keV

Still room for other shapes

Looks good!!

But: More flux than center segment could provide?
Better spot size than diffraction limit of center segment?

- YES, both
- More flux,
● large focus
- NO, both

Fig. 1. An SEM micrograph of a 100- μ m-deep silicon planar parabolic lens with minimized absorption. The lens aperture $A = 150 \mu\text{m}$, the number of unit lenses $p = 5$, the maximum phase variation number $M = 2$, the focal length $F = 80 \text{ cm}$ at the design wavelength $\lambda_0 = 0.071 \text{ nm}$ ($E_0 = 17.48 \text{ keV}$).

Evans-Lutterodt et al, Opt. Express 11 (2003)
Alianelli et al, SPIE Proc. 6705 (2007)

IMT@FZK

Problem: small outer zones

Evans-Lutterodt et al, PRL 99
(13), 134801 (2007)

Transmission: integrated $\leq 30\%$
i.e. $\leq 45\%$ of expectation
Drops off when segment height $< 2 \mu\text{m}$

Nevertheless in single lens
smallest focus $\approx 320 \text{ nm fwhm}$
(Stein et al, JVST B26, 122 (2008))
private communication Evans-Lutterodt:
more recently $< 320 \text{ nm}$

Alternative “lightening”

How to keep segment HEIGHT large?

Keep it constant!

Then simplify and compact the lens

and make it

It needs a name

Looks good: CLESSIDRA

Jark et al,
JSR 11,
248 (2004)

Jark et al,
JSR 13,
239 (2006)

As before
 $D \approx 40 \mu\text{m}$

for
SU8@8 keV
Si@12 keV

CLESSIDRA

Obviously would be a linear
transmission grating

in sufficiently spatially
coherent incident beam

Spatially incoherent incident beam

CLESSIDRA is an array of
tiny refracting prisms

Deflection angle in prism

$$\Delta = \frac{2\delta}{\tan \phi} = \delta \frac{b}{h}$$

Distance to refractive focus

$$\Delta f = h$$
$$f_{ref} = \frac{h}{\Delta} = \frac{h \tan \phi}{2\delta} = \frac{h^2}{\delta b}$$

Can focus size be <h?

NO!

We can use perfect prisms!

Rapidly alignable x-ray optics

Put it

Radiography

You see the
cressidra shape

Adjust
tilt, yaw, roll
for sharp
shadow

SYRMEP beamline, 12 keV photon energy, CCD camera 9 μ m pixel

Rapidly alignable x-ray optics

Radiography

You are done.

Now you can go
to refractive
focus position.

SYRMEP beamline, 12 keV photon energy, CCD camera 9 μ m pixel

Rapidly alignable x-ray optics

10 m downstream
from lens
with $h=12.83 \mu\text{m}$

Gain in focus is 6x
Size is $110 \mu\text{m}$ ($\approx$ hair)
lens efficiency = 25%

Same performance with
120 mm long mirror
with slope error $<0.5''$!

hair

See also Jark et al,
JSR 15, 411 (2008)

SYRMEP beamline, 19.5 keV photon energy, CCD camera $9 \mu\text{m}$ pixel

Rapidly alignable x-ray optics

with better beam coherence and high resolution CCD

$h=25.7 \mu\text{m}$

border

in focus

(2.16 m)

center

0.67 mm

BM05(MOTB)@ESRF, 8 keV photon energy, CCD 0.65 μm pixel

Rapidly alignable x-ray optics

hairs

Is the focus size not correlated with prism height h ??

Spatially COHERENT incident beam

In near field or Fresnel regime an object with periodic transmission function is re-imaged at the Talbot distances

$$D_{Tal,k/l} = \frac{kh^2}{l\lambda}$$

*k, l integers,
1/l demagnification
factor*

Now phase continuity required

$$b = D = \frac{m\lambda}{\delta}$$

fixing the refractive focal length to

$$f = \frac{h^2}{\delta b} = \frac{h^2}{m\lambda} = D_{Tal,k=1/l=m}$$

Operation restricted to discrete wavelengths

Some numbers

**$f=1 \text{ m@ } 8 \text{ keV}$
 $(\lambda=0.155 \text{ nm})$**

$$f = \frac{h^2}{m\lambda}$$

$m=1$

h independent of material: $h=12.45 \mu\text{m}$

**Needs lithography (we have one of few
beamlines for deep x-ray lithography
(DXRL) at ELETTRA)**

in resists (pmma or SU8) $b=36.7 \mu\text{m}$

**Crossed
hairs**

Some numbers

Spatially coherently
illuminated area:
 $A=0.44\lambda q/s$

8 keV or $\lambda=0.155$ nm

SYRMEP ($q=23$ m, $s=90$ μm): $A=17$ μm

BM05 ($q=53$ m, $s=85$ μm): $A=42$ μm

ID22 ($q=40$ m, $s=30$ μm): $A=91$ μm

$q=100$ m, $s=23$ μm : $A=300$ μm

Quality control: transmission

Contact radiography

filling: 50%

pmma
 $h=25.7 \mu\text{m}$
 $m=2, N=29$

**$L_{\text{exp}}=1.612 \text{ mm}$
at 8.5 keV**

**T variation in
last row
0.25 ---- 1.0**

expected average transmission

**damping:
CCD cross talk**

Quality control: refraction efficiency

Slit scan (0.1 mm):
Flux integrated over
50 μ m in focus

Relative efficiency
>50%
out to border

Rounding in
connected
prism tips

average transmission from before

Optimising the wavelength

pmma, $m=2$
 $h=25.7 \mu\text{m}$

illuminating 1.0 mm centered: 40 rows

Lens with **curved** prisms

Peak width $4.0 \mu\text{m}$

**“Coherent”
illumination**

**8.0 keV
detuned**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645 \mu\text{m}$ equivalent pixel**

Optimising the wavelength

pmma, $m=2$
 $h=25.7 \mu\text{m}$

illuminating 1.0 mm centered: 40 rows

Distance scan: full aperture 1.5 mm

Peak width $4.0 \mu\text{m}$

**“Coherent”
illumination**

**8.0 keV
detuned**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645 \mu\text{m}$ equivalent pixel**

Optimising the wavelength

pmma, $m=2$
 $h=25.7 \mu\text{m}$

illuminating 1.0 mm centered: 40 rows

Distance scan: full aperture 1.5 mm

simulation

**“Coherent”
illumination**

**8.0 keV
detuned**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645 \mu\text{m}$ equivalent pixel**

Optimising the wavelength

pmma, $m=2$
 $h=25.7 \mu\text{m}$

illuminating 1.0 mm centered: 40 rows

Another run (1 year later):

New monochromator

New E calibration

Vibrations:

Larger virtual source

→ Wider peaks (expect $6.5 \mu\text{m}$)

→ reduced spatial coherence
(from $42 \mu\text{m}$ to $21 \mu\text{m} < h$)

Peak width $7.3 \mu\text{m}$

**“Coherent”
illumination**

**8.0 keV
detuned**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645 \mu\text{m}$ equivalent pixel**

Optimising the wavelength

pmma, $m=2$
 $h=25.7 \mu\text{m}$

illuminating 1.0 mm centered: 40 rows

Peak width $6.6 \mu\text{m}$

**“Coherent”
illumination**

**7.9 keV
better
tuned**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645 \mu\text{m}$ equivalent pixel**

Optimising the wavelength

pmma, $m=2$
 $h=25.7 \mu\text{m}$

illuminating 1.0 mm centered: 40 rows

Lens with **curved** prisms

Peak width $5.7 \mu\text{m}$

**“Coherent”
illumination**

**7.7 keV
best
tune**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645 \mu\text{m}$ equivalent pixel**

Optimising the wavelength

pmma, $m=2$
 $h=25.7 \mu\text{m}$

illuminating 1.0 mm centered: 40 rows

Lens with **perfect** prisms

Peak width $6.5 \mu\text{m}$

**“Coherent”
illumination**

**7.9 keV
best
tune**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645 \mu\text{m}$ equivalent pixel**

Optimising the wavelength

pmma, $m=2$
 $h=25.7 \mu\text{m}$

illuminating 1.0 mm centered: 40 rows

Focusing horizontal source size

Lens with **perfect** prisms

reduced spatial coherence
(from $21 \mu\text{m}$ to $13 \mu\text{m} < h$)

**Focus size is $12.5 \mu\text{m} =$
expected demagnified source image
But also $h/2$**

**7.9 keV
best
tune**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645 \mu\text{m}$ equivalent pixel**

Refraction efficiency

vertical focusing: illuminating $65\text{ }\mu\text{m}$ (3 rows) at $270\text{ }\mu\text{m}$ off-axis

**“Coherent”
illumination**

**7.9 keV
best
tune**

**MOTB@ESRF (BM05-beamline)
CCD with $0.645\text{ }\mu\text{m}$ equivalent pixel**

Refraction efficiency

vertical focusing: illuminating $25\text{ }\mu\text{m}$ (1 row) at $270\text{ }\mu\text{m}$ off-axis

“Coherent”
illumination

7.9 keV
best
tune

MOTB@ESRF (BM05-beamline)
CCD with $0.645\text{ }\mu\text{m}$ equivalent pixel

Refraction efficiency

vertical focusing: illuminating $25\text{ }\mu\text{m}$ (1 row) at $270\text{ }\mu\text{m}$ off-axis

Outlook

De Caro and Jark, JSR 15, 176 (2008):

Found an analytical solution for the intensity distribution in focal plane for completely spatially coherent illumination!

Diffraction limited focus size identical for CLESSIDRA and concave parabolic lenses of same aperture: for aberrations corrected prisms
AND for perfect prisms with $m=1$ and $m=2$!

Reduction of maximum : 5%

Reduction of maximum : 20%

Outlook

De Caro and Jark, JSR 15, 176 (2008):

Perfect prisms introduce periodic wavefield distortion into transmitted field. Peak-valley amplitude of distortion is $\lambda/8$ for $m=1$ and $\lambda/4$ for $m=2$! The Rayleigh criterion for diffraction limited optics allows $<\lambda/4$ distortion!

Moderate loss of intensity into well localised secondary diffraction peaks. To be blocked with pinholes upstream of focus.

Reduction of maximum : 5%

Reduction of maximum : 20%

Outlook: new concepts

B. Cederstroem et al, JSR 12, 340 (2005)

hair

More absorbing,
less distorting,
larger peak
separation, shorter
focal length

problems in tips

Outlook: disconnect

IAF, Fraunhofer, Freiburg

IMT @ FZK, Karlsruhe

get better tips with reduced rigidity

Outlook: depth/aperture match

This was for $h=12.83\text{ }\mu\text{m}$

Aperture is $140 \cdot h$
Etching depth is only $25 \cdot h$

Conservative outlook

Take $A=25 \cdot h$ matched to depth of $25 \cdot h$, as shown before

Stack $M=2$ or Cederstroem prism array

$h=12 \mu\text{m}$: $A=0.3 \text{ mm}$ and $f=0.46 \text{ m}$ @ 8 keV ($\lambda=0.155 \text{ nm}$).

Spatial resolution limit $r=0.88 \cdot \lambda \cdot f/A$ $r=210 \text{ nm!}$

!!needs spatially coherent beam, e.g. $q=100 \text{ m}$ for $s=23 \mu\text{m}!!$

image could be 110 nm

Average transmission $>80\%/>60\%$ for one/bi-dimensional lens

More ambitious outlook

Take $A=50 \cdot h$ matched to depth of $50 \cdot h$

Stack $M=2$ or Cederstroem prism array

$h=6 \mu\text{m}$, $A=0.3 \text{ mm}$ and $f=0.116 \text{ m}$ @ 8 keV ($\lambda=0.155 \text{ nm}$).

Spatial resolution limit $r=0.88 \cdot \lambda \cdot f / A$ $r=53 \text{ nm!}$

!!needs spatially coherent beam, e.g. $q=100 \text{ m}$ for $s=23 \mu\text{m}!!$

image could be 27 nm

Average transmission $\approx 70\% / \approx 50\%$ for one-/bi-dimensional lens

Fresnel lens outlook

Evans-Lutterodt et al, PRL 99 (13),
134801 (2007)

Arrived already at
 $A=0.3$ mm, $f=0.1$ m
image size $\ll 320$ nm fwhm
Stein et al, JVST B26, 122 (2008),
priv. comm. Evans-Lutterodt

Only center segment is >6 μm !

Relative efficiency $\approx 25\%$
for segments ≈ 1.7 μm
and $<10\%$ for ≈ 0.7 μm

In Si better shape fidelity at smaller dimensions than in photoresist!
In turn etch depth limitation for RIE at 0.1 mm?

Fresnel lens outlook: adiabatic

Schroer et al, PRL 94, 054802 (2005)

Follow adiabatically shrinking beam size

In 1166 lenses

Lens thickness: $37.8 \mu\text{m} \dots 0.100 \mu\text{m}$

Lens aperture: $42.2 \mu\text{m} \dots 0.224 \mu\text{m}$

Outer segment: $4.5 \mu\text{m} \dots 0.024 \mu\text{m}$

focus $r=2.21 \text{ nm}$ @ 27.6 keV

Thank you for help

DXRL: Marco Matteucci, Frédéric Pérennès, Benedetta Marmiroli

IMM (Mainz): Laurence Singleton, Abdi Tunayar (EU action: EMERGE)

SYRMEP: Lucia Mancini, Giuliana Tromba, Luigi Rigon, Francesco Montanari, Ralf Hendrick Menk, Diego Dreossi

workshop: Gilio Sandrin, Ivan Cudin

IC-CNR: Liberato De Caro

ESRF-ID22: Jean Susini, Andrea Somogyi, Remi Tucoulou, Sylvain Bohic

ESRF-BM05: Anatoly Snigirev, Irina Snigireva

More about project:

<http://www.elettra.trieste.it/experiments/beamlines/microfluo/docs/clessidra.pdf>

Carolyn MacDonald

UAlbany Center for X-ray Optics

Outline

Structure of Polycapillaries

- Physics of Reflection, endin imits
- iver ence
- Ali nment and Characteri ation
 - Source Angle
 - ransmission vs Ener y
 - Spot Size
- Gain and iouville s heorem
- eflect Analysis
- Applications
 - MicroXR
 - XR
 - Astronomy
 - herapy

Polycapillary Optics

COLLIMATING

multifiber

50 mm x 50 mm output
250 mm input focal lenth
12 μ m channel

FOCUSING

monolithic

How do they work? Maxwell's equations for a non-magnetic insulator:

$$\left. \begin{aligned} \nabla \cdot \vec{E} &= 0 \\ \nabla \cdot \vec{B} &= 0 \\ \nabla \times \vec{E} &= -\frac{\partial \vec{B}}{\partial t} \\ \nabla \times \vec{B} &= \mu_0 \epsilon \frac{\partial \vec{E}}{\partial t} \end{aligned} \right\} \Rightarrow \begin{aligned} \frac{\partial^2 \vec{E}}{\partial t^2} &= \frac{1}{\mu_0 \epsilon} \nabla^2 \vec{E} \\ v &= \frac{1}{\sqrt{\mu_0 \epsilon}} = \frac{1}{\sqrt{\mu_0 \epsilon_0}} \sqrt{\frac{\epsilon_0}{\epsilon}} = \frac{c}{n} \end{aligned}$$

$\Rightarrow n, \text{index of refraction} = \sqrt{\frac{\epsilon}{\epsilon_0}}$

So, given an incident plane wave of the form:

$$\vec{E} = \vec{E}_0 e^{i\omega t}$$

we expect a response

$$\vec{D} = \epsilon \vec{E} = \epsilon_0 \vec{E} + \vec{P}$$

hence

$$\vec{D} = \epsilon \vec{E} = \epsilon_0 \vec{E} + \vec{P} \Rightarrow \epsilon = \epsilon_0 + \frac{\vec{P}}{\vec{E}}$$

needed to model
the response, $\vec{P}$
of the material
to the EM wave

Free Electrons in one dimension:

$$E = E_0 \cos \omega t$$

$$F = m\ddot{x} = qE = qE_0 \cos \omega t$$

$$x = x_0 \cos \omega t$$

$$\Rightarrow -m\omega^2 x_0 = qE_0 \Rightarrow x_0 = \frac{-qE_0}{m\omega^2}$$

$$P_0 = Nqx_0 = \frac{-Nq^2}{m\omega^2} E_0$$

$$\Rightarrow \epsilon = \epsilon_0 + \frac{-Nq^2}{m\omega^2} = \epsilon_0 \left(1 - \frac{\omega_p^2}{\omega^2} \right)$$

$$P = \quad x$$

number of
electrons per
volume

char e on an
electron

x displacement of
the electron due to
the electric field

$$\epsilon = \epsilon_0 \left(1 - \frac{\omega_p^2}{\omega^2} \right)$$

Index of Refraction

$$n = \sqrt{\frac{\epsilon}{\epsilon_0}} = \sqrt{1 - \frac{\omega_p^2}{\omega^2}} \approx 1 - \frac{1}{2} \frac{\omega_p^2}{\omega^2} = 1 - \delta$$

$$\Rightarrow \delta = \frac{1}{2} \left(\frac{30 \text{ eV}}{10 \text{ eV}} \right)^2 \approx 4.5 \times 10^{-6}$$

Consequences for normal incidence mirror:

$$R(\theta = 0) = \left(\frac{n_1 - n_2}{n_1 + n_2} \right)^2 = \frac{\delta^2}{2} \approx 10^{-11}$$

Total External Reflection

$$n_1 \sin(90 - \theta_1) = n_2 \sin(90 - \theta_2)$$

$$(1) \cos(\theta_1) = (1 - \delta) \cos(\theta_2)$$

$$(1) \cos(\theta_c) = (1 - \delta) \cos(0) \Rightarrow 1 - \frac{\theta_c^2}{2} \doteq 1 - \delta \Rightarrow \theta_c \doteq \sqrt{2\delta}$$

$$\theta_c = \frac{\omega_p}{\omega} \approx 3 \times 10^{-3} \quad \mathbf{R=1}$$

Damping

$$F = m\ddot{x} = -kx - b\dot{x} + qE$$

$$x = x_0 \cos(\omega t + \phi)$$

$$n = 1 - \delta - i\beta$$

$$R < 1$$

Reflectivity vs Angle

X rays in hollow capillary tubes:

Can collect

And redirect beams

To make a lens

How much bending is required for a lens?

Approximate outermost fiber of lens as circle of radius R

Capture angle

$$\phi = 2 \operatorname{atan}\left(\frac{r}{f}\right)$$

$$L \approx R\phi \Rightarrow R \approx \frac{L}{\phi}$$

$$f = 50 \text{ mm}, \quad r = 5 \text{ mm} \Rightarrow \phi = 11.5^\circ \quad L = 100 \text{ mm} \Rightarrow R \approx 1 \text{ m}$$

Cutoff Energy dependence on bend radius and channel size

$$\cos(\theta) = \frac{R}{R+c} \Rightarrow 1 - \frac{\theta^2}{2} \approx \frac{1}{1 + \frac{c}{R}} \approx 1 - \frac{c}{R}$$

$$\theta^2 \approx \frac{2c}{R} < \theta_c^2 = \left(\frac{\omega_{\text{plasma}}}{\omega} \right)^2 = \left(\frac{E_{\text{plasma}}}{E} \right)^2 \Rightarrow E < E_{\text{plasma}} \sqrt{\frac{R}{2c}}$$

$$c = 2 \mu\text{m} \quad R = 1 \text{ m} \quad E_{\text{plasma}} = 30 \text{ eV} \Rightarrow E < 15 \text{ keV}$$

Transmission

Beam Filtering

Characterization of Polycapillary Optics

Capture Angle,

$$\omega = 3^\circ$$

$\Omega = 2$ milliradian

transmission 9.9

Input focal length

Focusing Lens Output Spot Measurement

Focusing Optic Output

ocal spot size 0.8 mm

Image plate

Global Divergence=

$$\text{slope} = 2\beta = 0.55^\circ$$

FOCAL SPOT SIZE

Minimum due to Local Divergence 2a

Minimum size :

$$d = c + 2\alpha f = c + 1.3 \theta_c f$$

$$c = 5 \mu\text{m} \quad 2\alpha = 0.28^\circ \quad \begin{cases} f = 50 \text{ mm} \\ f = 5 \text{ mm} \end{cases} \quad \begin{aligned} &\Rightarrow d = 250 \mu\text{m} \\ &\Rightarrow d = 30 \mu\text{m} \end{aligned}$$

Rocking Curve collimating optic

Global divergence

Rocking Curve focusing optic

$$\text{Rocking Curve Width} = 0.83^\circ = 2\alpha + 2\beta = 0.28^\circ + 0.55^\circ$$

Relative to What?

To make the optic look good: compare to flux through a pinhole of diameter $<$ spot size compared to source at focal point:

$$F_{\text{no optic}} = \frac{P_{\text{source}}}{4\pi(f_{\text{in}} + L + f_{\text{out}})^2} \pi \left(\frac{d}{2} \right)^2 \quad F_{\text{Lens}} = \frac{P_{\text{source}}}{4\pi(f_{\text{in}})^2} \pi r_{\text{Lens}}^2 T$$

$$\text{Gain} = \frac{F_{\text{Lens}}}{F_{\text{no optic}}} = \left(\frac{f_{\text{in}} + L + f_{\text{out}}}{f_{\text{in}}} \right)^2 \left(\frac{r_{\text{Lens}}}{d/2} \right)^2 T$$

$$d = 200 \mu\text{m} \quad r_{\text{Lens}} = 5 \text{ mm} \quad f = 50 \text{ mm} \quad L = 100 \text{ mm} \Rightarrow \text{Gain} = 8000$$

Liouville's Theorem

Angle area product cannot decrease

$$A_f \Omega_f \geq A_o \Omega_o$$

Optics Defects

Optic defects

Bending:

λ_b 10 cm

parameter: bending radius, R

Waviness:

λ_r λ λ_b
parameter:

Modeled by random angle shifts of $\delta\theta$ after each bounce

Optic defects

$$\lambda \approx 1 \text{ } \mu\text{m}$$

Roughness:

parameters:

correlation length
rms height

Roughness
correlation

$$g(\Delta x) = \frac{1}{L} \int_0^L Z(x)Z(x + \Delta x)dx = \bar{Z}^2 e^{-\frac{|\Delta x|}{s}}$$

Simulation Analysis: Single Fiber

simulation with
 $w=0.15$ mrad and
 $R=120$ m.

transmission

0.5
0.0

Experiment
Simulation

10 30 50 70
Energy, eV

Simulation Analysis: Lens made from fibers

Optics Defect: Channel Blockage

39 mm input focal length
 7.8 mm output focal length
 30 μ m loss in length of lens

Application to Radiation Damage

Characteristics of Polycapillary Focusing Optics:

Type Beam Focusing, Polychromatic

Useful X-ray Energy Range Typically 0.1 - 30 keV

Collection Solid Angle Up to 20 degrees

Working Distance 2.5mm 5mm 10mm 20mm
50mm 100mm

Focused beam size
(Mo K α , FWHM, 17.4keV) 10um 18um 30um 45um
100um 180um

Gain (Compared to pinhole aperture
100 mm from source) 100x - 10000x

Applications: XRF

Spatial Resolution of MXR with 39 μm spot

Micro graph of Cu grid

254 μm

49 μm

Elemental Mapping

MXRF maps of a quartz phenocryst with small volcanic glass inclusions

E	EME	M	(p)
K		4.1	
i		1.5	
e		0.57	
n		0.28	
Pb		0.52	

Courtesy of Ning Gao, XOS

Applications: Protein Crystallography

iso yme pattern taken in
20 seconds with 2.8
rotatin anode, comparable to 30-35
min. without optic

linear R factor without optic 6.4
with optic on same sample: 6.9

Applications: Protein Crystallography

Source: 37 kV, 25 W

Optic: 5.8 mm input, 136 mm

Crystal size	Less than 200 μm
oscillation angle	1.5 deg (44 frames)
time / frame	60 min
PINdiode intensity	3×10^{-4}
resolution	2.0 Å
R-factor	5.2%

Applications: Protein Crystallography

5 min
1 mA
2.1

Applications: Powder Diffraction

Applications: Powder Diffraction

Optic Results	Relative diffracted beam Intensity	i sample at focus, plate at 66-75 mm	
		Peak width	Average Peak error
one	1	0.7	0.23
f 47	5	1.1	0.3
f 119	113	1.9	0.2

Applications: **m**SPECT/CT

E. Ritman et al., Mayo clinic

Applications: Orthovoltage Therapy

Will the spot size increase due to intervening “tissue”?

Applications: Orthovoltage Therapy

Beam hardening

Applications: Synchrotron Focusing and Astronomy

Table 1. Results for monolithic focusing optic.

X-Ray Ener y (eV)	Spot si e (mm)	rans- mission ()	measured Gain 350 μ m pinhole	calculated Gain 350 μ m pinhole	calculated Gain 90 μ m pinhole	calculated Gain 10 μ m pinhole
6	0.09	36	78	81	645	911
8	0.08	49	96	110	933	1359
10	0.09	39	83	87	624	842
12	0.09	39	74	87	654	903
white	0.17	42	11	89	243	266

References

- F.R. Sugiyo, Danhong Li, C.A. MacDonald, "Beam Collimation with Polycapillary X-ray Optics for High Contrast High Resolution Monochromatic Imaging," Med. Phys., **31**, p. 3288, 2004.
- C.A. MacDonald and W.M. Gibson, "Applications and Advances In Polycapillary Optics", X-ray Spectrometry, **32** (3), 2003, pp 258-268.
- C.A. MacDonald, W.M. Gibson, and W. Pepler, "X-Ray Optics for Better Diagnostic Imaging," Technology In Cancer Research And Treatment, 1, (2), April 2002, pp 111-118.
- Suparmi, Cari, Lei Wang, Hui Wang, W.M. Gibson, C.A. MacDonald, "Measurement and Analysis of Leaded glass Capillary Optic Performance for Hard X ray Applications," Journal of Applied Physics **90** (10), pp. 5363-8, 2001.
- F.A. Hofmann, W.M. Gibson, C.A. MacDonald, D.A. Carter, J.X. Ho, J.R. Ruble, "Polycapillary Optic – Source combinations for Protein Crystallography," Jour. Applied Crys. **34**, pp.330-335, 2001.
- C.A. MacDonald and W.M. Gibson, "An Introduction to X-ray and Neutron Optics," chapter 19 in M. Bass, ed., **Handbook of Optics, Volume III**, McGraw-Hill 2000.
- C.A. MacDonald and W.M. Gibson, "Polycapillary and Multichannel Plate X-ray Optics," chapter 30 in M. Bass, ed., **Handbook of Optics, Volume III**, McGraw-Hill 2000.
- J.B. Ullrich and C.A. MacDonald, "Electron Impact Sources," chapter 31 in M. Bass, ed., **Handbook of Optics, Volume III**, McGraw-Hill 2000.
- C.A. MacDonald and W.M. Gibson, "Applications Requirements Affecting Optics Selection," chapter 35 in M. Bass, ed., **Handbook of Optics, Volume III**, McGraw-Hill 2000.
- W.M. Gibson and C.A. MacDonald, "Summary of X-ray and Neutron Optics," chapter 37 in M. Bass, ed., **Handbook of Optics, Volume III**, McGraw-Hill 2000.
- C.A. MacDonald and S.D. Padiyar, "How Photons Work," cover article, Photonics Spectra, August 2000.
- C.A. MacDonald, S.M. Owens, and W.M. Gibson, "Polycapillary X-Ray Optics for Microdiffraction," Journal of Applied Crystallography, **32**, pp160-7, 1999.
- B.K. Rath, W.M. Gibson, Lei Wang, B.E. Homan and C.A. MacDonald, "Measurement and Analysis of Radiation Effects in Polycapillary X-ray Optics," Journal of Applied Physics, 83, no.12, pp. 7424-7435, June 15 1998.
- C.C. Abreu and C.A. MacDonald, "Beam Collimation, Focusing, Filtering and Imaging with Polycapillary X-ray and Neutron Optics," invited review article, Physica Medica, vol. XIII, N.3, 1997, pp. 79-89.
- F.A. Hofmann, C.A. Freinberg-Trufas, S.M. Owens, S.D. Padiyar, C.A. MacDonald, "Focusing of Synchrotron Radiation with Polycapillary Optics," Beam Interactions with Materials and Atoms: Nuclear Instruments and Methods B, vol. **133**, 1997, pp. 145-150.

FOCUSING OF X-RAYS USING CRYSTAL OPTICS

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Workshop: Focus on X-Ray Focusing, San Diego, August 13, 2008

X-Ray Bragg Reflection on a Flat Sample Crystal

Real space

$$n \lambda = 2 d_{hkl} \sin \theta_{hkl}$$

$\vec{S}_0$: incident beam wave vector
 $\vec{S}$: Bragg reflected beam wave vector
 θ_{hkl} : Bragg angle
 d_{hkl} : distance between the reflecting planes

$\vec{g}$ normal of reflecting planes,
 reciprocal lattice vector
 hkl indices of reflecting planes
 n 1, 2, ..., diffraction order

flat crystal, symmetrical reflection

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

Merrill, DuMond, Annals of Physics 14 (1961), Fig. 5

X-Ray Spectrometer with Flat and Bent Crystals

X-ray spectrometer
with a single flat crystal

Johann spectrometer
with a bent crystal

E. Förster et al., Journal of Quantitative Spectroscopy and Radiative Transfer 51(1994), 101

Application range of crystal reflection for Bragg angles 60° - 89°

X-ray imaging is best, if θ is maximal.

Toroidal Bent Crystals for Focusing of X-Ray from fs Laser Plasma

focal lengths: $f_h = (R_h/2) \cdot \sin \theta_B$

$f_v = R_v / (2 \cdot \sin \theta_B)$

Point to point focussing $R_v / R_h = \sin^2 \theta$

Fabrication and Test of Toroidally Bent Crystals

grinding and polishing of toroidal glass formers

control of surface quality and bending radius $\Delta R/R < 0.001$

X-ray topography → 'perfect' crystal block

oriented sawing (accuracy: 10' to 10''), grinding, polishing → 70 μm thick discs

optical contact with glass form sticking to crystal mounting with epoxy glue

optical and X-Ray imaging tests,

$$\text{Relation of the Curvature Radii: } R_v/R_h = \sin^2 \theta$$

Test of Bent Crystals for Monochromatic Imaging

Fabrication and Test of Toroidally Bent Crystals

grinding and polishing of toroidal glass formers

control of surface quality and bending radius $\Delta R/R < 0.001$

X-ray topography → 'perfect' crystal block

oriented sawing (accuracy: 10' to 10''), grinding, polishing → 70 μm thick discs

optical contact with glass form sticking to crystal mounting with epoxy glue

optical and X-Ray imaging tests,

determination of the reflectivity (X-Ray tube or synchrotron),

$$\text{Relation of the Curvature Radii: } R_v/R_h = \sin^2 \theta$$

Experimental Setup for Reflection Curve Measurement

Experimental set-up
for the rocking curve
measurement using a flat
and a cylindrically bent
von Hámos – crystal,
 $SC = a$.

Reflection Curves of a Cylindrically Bent Quartz 40.4

$$E_{\text{det}} = \iiint d\alpha d\phi d\lambda J_s(\alpha, \phi, \lambda) \times C\left(\sigma(\alpha, \phi) - \frac{\Delta\lambda}{\lambda} \tan \theta_0\right)$$

I. Uschmann, C. Malgrange, E. Förster, J. Appl. Cryst. 30 (1997), 1150

I. Uschmann et al., J. Appl. Cryst. **26** (1993), 405

Fabrication and Test of Toroidally Bent Crystals

grinding and polishing of toroidal glass formers

control of surface quality and bending radius $\Delta R/R < 0.001$

Relation of the Curvature Radii: $R_v/R_h = \sin^2 \theta$

cylindrically bent mica

$R = 100 \text{ mm}$

50 mm x 60 mm

toroidally bent GaAs 400

$R_h = 200 \text{ mm}$, $R_v = 189.4 \text{ mm}$

toroidally bent quartz 10.-1

$R_h = 500 \text{ mm}$, $R_v = 400 \text{ mm}$

High power laser: $E_L > 10^6 \text{J}$, $\tau_L < 1 \text{ns}$, $\lambda_L < 0,5 \mu\text{m}$

main aim: supression of Rayleigh-Taylor instabilities

X-Ray Monochromatic Camera Using Two Toroidal Crystals

Coma can be corrected in X-ray optical systems with the use of two mirrors. A single X-ray mirror strongly violates the Abbe sine condition, since β increases as α decreases, while the sine condition demands, that $\sin \alpha / \sin \beta$ remain constant (upper diag.). Approximate constancy of the sine ratio can be achieved through the use of two mirrors (lower diag.), so that β increases as α increases.

	one crystal	two crystals
spatial resolution	14 μm	1.6 μm
spectral window ($\lambda/\Delta\lambda$)	235	6069
relative luminosity	1	1/15

Setup of an Optical Pump X-Ray Probe Experiment

T. Feurer et al., Appl. Phys. B (2001) 72, 15

Non-thermal Melting: InSb X-ray signal ($7 \cdot 10^{16} \text{ W/cm}^2$)

electron-phonon-coupling time 10 ps

A. Rousse et al., Nature, 410, No. 6824 (2001) 65 - 68

Schematic of Four Cu $K\alpha$ Optics

- a. toroidally bent Ge crystal, 444 reflex, $\theta = 70^\circ$,
- b. two perpendicular elliptical Ni/C multilayer mirrors, $\theta \approx 3^\circ$,
- c. ellipsoidal lead-glass capillary,
- d. borosilicate poly-capillaries (59,000).

Characteristic Parameters of Cu K α Optics

CuK α optics	toroidal Ge	multilayer mirror system	ellipsoidal capillary	Poly- capillaries
size of focus (μm)	23	32	155	105
1D-convergence angle (deg)	1.5	0.45	0.2	3.5
solid angle (sr)	$2.3 \cdot 10^{-3}$	$8.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$	$1.1 \cdot 10^{-2}$
reflectivity or transmission	0.03	0.2	0.8	0.09
suppression of K β	0.017	$5 \cdot 10^{-4}$	1.4	1.7

red – best value

pink –second best value

M. Bargheer et al., Appl. Phys. B 80 (2005), 715

Ellipsoidal HOP Graphite Crystal Reflection Curve

Ellipsoidal HOP Graphite Crystal X-Ray Focus Test

Summary of X-Ray Crystal Optics Parameters

- Energy range: 500 eV – 40 keV (reflection case)
20 keV – 100 keV (transmission case)
- Spectral resolution: $\Delta E / E = 1,000 - 10,000$
- Used solid angle: $10^{-5} \text{ sr} - 10^{-3} \text{ sr}$
- Focal size: $1 \text{ }\mu\text{m} - 5 \text{ }\mu\text{m}$ @ large θ angles,
sub- μm for a two-crystal device
- Focal distance: 5 cm – 5 m
- Cost: 10,000 \$ for one crystal
- Availability: firms of precision optics and crystal manufacturer,
scientific institutes

... for a brighter future

U.S. Department
of Energy

UChicago ►
Argonne_{LLC}

A U.S. Department of Energy laboratory
managed by UChicago Argonne, LLC

Multilayer Laue Lens for Efficient Nanometer Focusing of Hard X-rays

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⁴Advanced Photonics Research Institute,
Gwangju Institute of Science and Technology

⁵National Synchrotron Light Source II,
Brookhaven National Laboratory

Workshop: Focus on X-ray Focusing, 2008 SPIE,
San Diego, CA, August 13, 2008

Outline

- Multilayer Laue Lens
 - Motivation and Approach
 - Status: 16 nm line focus, 31% efficiency, 19.5 keV
 - Future: Sub-nanometer focusing
- Overview of the Nanoprobe Beamline at Argonne

Ultimate Resolution of X-ray Focusing Optics

$$\text{Rayleigh criterion : Best Possible Resolution} = \frac{\lambda}{2 \text{ NA}}$$

Can x-rays be efficiently focused to the atomic scale?

--> Need larger Numerical Aperture (NA)

However, it is inherently difficult to produce large NA optics for hard x-rays

Currently, $\text{NA} \sim 10^{-3}$, resolution $\sim 500 \lambda$

- What type of optics will work (e.g. reflective, refractive, diffractive)?
- What is the fundamental limit using real materials ?
- Can we fabricate optics that reach the fundamental limit ?

High NA Hard X-ray Focusing Optics

■ **Refractive: Lenses**

Compound refractive lens

Compound Fresnel lens for high NA

■ **Reflective: Mirrors**

Figure by differential deposition,
multilayer coated for high NA

■ **Diffraction: Zone Plates**

High aspect ratio, tilted zones
for high NA

Lithographic zone plate

Transmission multilayer

Diffractive X-ray Optics with High NA Require High Aspect Ratio Nano-structures

- Small focal spot sizes require small zone widths.
- Zone plate structures for hard x-rays must be several microns thick to achieve high efficiency, which implies **high aspect ratios ($\gg 100$)**.
- It is difficult to produce such high aspect ratio structures using lithography.
- **Sectioning of multilayers allows very high aspect ratios to be produced.**

Sputtered-Sliced Fresnel Zone Plate

S. Tamura et al. (2002) & B. Kaulich et al. (1996)

- Deposition of zone plate structure on circular wire
- Imperfections of wire are amplified
- Later coating of the outermost zones gives worse layer position accuracy in most sensitive region
- Circular geometry gives 2-D focus in one optic, but can't tilt layers to get high efficiency
- Cu-Al materials are relatively difficult to section without damage
- Focal spot size $\sim 200 \text{ nm}$

Multilayer Laue Lens

Deposit multilayer on flat substrate to produce one-dimensional focusing optic

(1) Deposit multilayer with depth-graded spacing to form zones of linear zone plate
(thinnest structures first)

(2) Make cross-sections to allow use in Laue geometry
(high aspect ratio structure)

(3) Assemble sections:
two opposite to collect full NA
(tilt to achieve high efficiency);
a second pair at right angles
to form point focus
(high efficiency allows two optics in series)

Depth-graded multilayers
on flat Si substrate

Multilayer Laue Lens

Theory for MLL

- MLL's operate in a different optical regime than standard zone plates
- Dynamical diffraction effects inside the volume of the structure are dominant
- Theory for focusing performance:
 - J. Maser et al. - Coupled wave theory (*Optics Commun.* **89**, 355 (1992); *Phys. Rev. Lett.* **96**, 127401 (2006))
 - C. Shroer - Parabolic wave equation (*Phys. Rev. B* **74**, 033405 (2006))
 - H. Yan et al. - Takagi-Taupin (*Phys. Rev. B* **76**, 115438 (2007))

Optimum Zone Geometry

- Flat geometry with all layers parallel is standard for lithographic zone plates
- Ideal “wedged” geometry where each layer makes the Bragg angle for its spacing becomes favorable for high NA focusing
- Ideal geometry can be approximated by tilting each half of MLL

Where is the Spatial Resolution Limit?

- Flat structure: loses efficiency below 10 nm
- Ideal “wedged” structure:
 - High diffraction efficiency
 - Resolution below 1 nm feasible
- Tilted MLL structure:
 - 6 nm resolution feasible

H. C. Kang et al.

Physical Review Letters **96**, 127401 (2006)

Multilayer Structure for MLL

WSi_2/Si , 1588 layers, $t_{\text{dep}} = 13.25 \mu\text{m}$

$\Delta r_{\text{max}} = 25 \text{ nm}$

$\Delta r_{\text{min}} = 5 \text{ nm}$

H. C. Kang et al., *Applied Physics Letters* **92**, 221114 (2008)

Measured and Calculated Performance

FWHM = 16 nm

Efficiency = 31% at 19.5 keV

Measurement of half-MLL structure agrees well with calculation

Assembly of complete MLL of this structure should give focus of 6 nm, which will be the smallest focus of photons yet achieved

H. C. Kang et al., *Applied Physics Letters* **92**, 221114 (2008)

Near-Atomic-Scale Focusing Possible with “Wedged” MLL

Calculations show that focusing to below 1 nm is possible using “wedged” layers

H. Yan et al., *Physical Review B* **76**, 115438 (2007)

Progress of Deposition of Wedged Multilayer Structures

- We have succeeded in depositing initial multilayer structures for a “wedged” MLL
- Examples:
 - 40% structure, outermost zone width 2.5 nm, 1588 layers, 6.6 μm tot.
 - Full structure, outermost zone width 3 nm, 6543 layers, 40 μm total
- Characterization is underway

R. Conley et al., *Review of Scientific Instruments* **79**, 053104 (2008)

Future Directions for MLL

- Theory
 - Determine optimum structure and performance as $f(\lambda)$
 - Determine fabrication accuracy required
- Experiment
 - Cross two linear optics to make point focus
 - Assemble complete “tilted” structure to achieve 6 nm focus
 - Develop techniques to deposit “wedged” layers to achieve near-atomic-scale focusing

Summary of Multilayer Laue Lens Properties

- Energy range: 5 - 100 keV
- Gain: ~1000X or more in each dimension
- Focal size: 20 nm or less
- Focal distance: 2 mm or less
- Cost, availability:
 - Currently a research effort at ANL, NSLS II, GIST
 - Potentially available in a few years
 - Potentially less than \$10K per optic

Nanoprobe Beamline at the Center for Nanoscale Materials

Advanced Photon Source, ANL

- Center for Nanoscale Materials
 - new nanoscience research center located adjacent to APS
- Nanoprobe Beamline
 - state-of-the-art hard x-ray microscopy beamline, built and operated in partnership between CNM and APS

Nanoprobe Beamline: Overall Specifications

- Hard X-ray microscopy at the highest achievable spatial resolution
 - Initial spatial resolution of 30 nm using lithographic zone plates
 - Energy range 3 - 30keV (nano-spectroscopy excitation of most elements)
 - Large penetration → sample environments/fields
- Planned capabilities
 - **Fluorescence:** atto-g elemental sensitivity, chemical state sensitivity
 - **Diffraction:** sensitivity to crystallographic phase, strain, orientation
 - **Tomography:** transmission absorption / phase contrast imaging
 - **Coherent x-rays:** disorder, imaging
 - **Magnetic contrast** using polarized x-rays
 - **Dynamic studies** at 100 ps time resolution

X-ray Fluorescence Spectroscopy in the Hard X-ray Nanoprobe

K excitation

L excitation

Fluorescence Spectroscopy

Fluorescence Mapping

© Argonne, ORNL

Nanoprobe Beamline Schematic

Scanning Probe Mode

Full-Field Transmission Mode

Nanoprobe Scanning Design

- Laser interferometers monitor positions of optics and sample to Angstrom precision
- Nanoscale scanning of focusing optic using piezo stages
- Feedback used to lock beam to desired position on sample at each point in scan

Vertical Flexure stage

Horizontal Flexure stage

Nanoprobe Beamline Construction Complete

- Zone plate performance at 30 nm verified
- Beamline commissioning underway
- First user program experiments started this year

Smallest visible structures are ~30nm

Recent Example: Nanodiffraction from Strained Silicon

~50 nm resolution
scanning diffraction
maps of device features
of strained silicon on
insulator (SOI)

Courtesy: Conal Murray*, Sean Polvino⁺, Andrew Ying⁺, Ozgur Kalenci⁺, I.C. Noyan⁺

* IBM, ⁺Columbia University

Summary

■ MLL

- Achieved 16 nm FWHM line focus, 31% efficiency at 19.5 keV
- Sub-nanometer focusing predicted

■ Nanoprobe

- Currently: 30 nm resolution zone plates
- Plan: 10 nm resolution MLL

Diffraction Focusing By Zone Plates

3D X-ray Imaging for Science and Industry

Dr. Michael Feser
Vice President / General Manager
nano-Imaging
Xradia Inc.

SPIE X-ray
Focusing Workshop

High-resolution Optics: Comparison

	KB Mirror	Refractive Lens	Zone Plates (+Laue optics)
Demonstrated Resolution (nm)	<30	<50	<15 for 0.5 keV 22 for 8 keV
Flux Density Gain	>500,000	10000	>500,000
Imaging Optic	No	Yes	Yes
Chromatic Aberration	No	$1/\lambda^2$	$1/\lambda$
Theoretical resolution limit (nm)	<10	~ 2 ? (Schroer et al.)	$\sim 1^{(1)}$?
Minimum focal length for 100 um aperture (mm)	~ 30	100 for 10 keV	0.5 for 0.5 keV 20 for 10 keV

⁽¹⁾ Kang et al., PRL96(2006)

Diffractive Focusing By Zone Plates

Outline

- ❑ History and working principle
- ❑ Fabrication, limitations and future developments
- ❑ Applications
 - X-ray imaging / microscopy
 - X-ray nano-probing: Diffraction, spectroscopy
- ❑ Summary and outlook

Diffraction From a Grating

- ❑ Recall diffraction from slits separated by d
- ❑ diffraction *maxima* for positive interference of light waves occurs when
 $d \sin \theta = n\lambda$
 n = diffraction order
 θ = diffraction angle
- ❑ Constant grating pitch acts like a prism for one diffraction order (deflects light)
- ❑ Deflection is a linear function of wavelength λ

Diffraction from Circular Grating

- By varying the grating pitch radially in a circular grating, positive interference on-axis at a focal point is obtained

- Excellent Reference: M. Young, JOSA 62(8), pp. 972-976

Diffractive Lenses: Fresnel Zone Plates

- ❑ Focal length has a strong wavelength dependence:

$$f = \frac{OD \Delta R_N}{\lambda}$$

- ❑ Necessitates use of monochromatic beam, with bandwidth $E/\Delta E >$ number of zones
- ❑ Resolution limited by outermost zone width ΔR :

$$\text{Res} = 1.22 \Delta R$$

Diffraction Orders of Zone Plates

- ❑ Diffractive elements have more than one diffraction order
- ❑ Directly transmitted beam: 0th order
Higher diffraction orders with decreasing intensity
(even orders forbidden for 1:1 mark to space ratio)
- ❑ With use of apertures and stops one diffraction order can be isolated and ZP acts like a thin lens (disadvantage of zone plates)

Central stop and order sorting aperture (OSA) to isolate first order focus in a nano-probing application

Zone Plates as Thin Lenses

- ❑ Works just like a thin lens, except:
 - 1st order efficiency
 - ❑ Opaque zones ~10%
 - ❑ Phase reversing zones ~40%
 - ❑ Blazed zones 100%
 - ❑ (less for real materials)
 - Need to deal with unwanted orders
 - Highly chromatic : $f \sim 1/\lambda$
 - No spherical aberrations

Zone Plate Efficiency for Real Materials

Scanning Electron Micrographs of Zone Plates

- ❑ Resolution limited to approximately the zone width ΔR
- ❑ Focusing efficiencies up to 30% for high x-ray energies

Zone Plates: Early History

- ❑ Rayleigh 1871 – unpublished
 - ❑ Soret 1875 – first publication
 - ❑ Rayleigh 1888 – phase zone plates
 - ❑ Wood 1898 – experiments with zone plates using light
-
- ❑ Some simple cameras use zone plates instead of pinholes!

First Images With a Fresnel Zone Plate

□ R. W. Wood (1898): zone plate figure drawn with a pen and a compass!
Photographically reduced

PLATE 2. ZONE-PLATE, FROM A DRAWING.

X-ray zone plate history

- ❑ Albert Baez (UNESCO, SAO/Cambridge)
 - “The possibility constructing a single Fresnel zone plate for x-rays should be explored” - J. Opt. Soc. Am. 42, 756 (1952) - paper on x-ray holography.
 - Demonstrated free-standing metal zone plate for X rays: “auguring well for resolution at 100 Å” - Nature 186, 958 (1960).
- ❑ Gunter Schmahl and Dietbert Rudolph (Göttingen)
 - Proposed holographic fabrication method - Optik 29, 577 (1969)
 - First TXM demonstration using synchrotron radiation - Niemann et al., Opt. Comm. 12, 160 (1974)
- ❑ Janos Kirz (Stony Brook)
 - Phase enhancement of efficiency - J. Opt. Soc. Am. 64, 301 (1974)
 - First STXM demonstration using synchrotron radiation - Rarback et al., 1983 XRM conference proceedings; Kenney et al., J. Microscopy 138, 321 (1985). Zone plates: D. Kern et al., IBM.
- ❑ E-beam zone plates:
 - Proposed by D. Sayre, IBM tech report RC 3974 (1972).
 - First demonstrated by Nat Ceglio, MIT, in E. Ash, Scanned Image Microscopy (Academic Press, 1980); J. Vac. Sci. Tech. B 1, 1285 (1983)

Zone Plate Patterning Techniques

- ❑ E-beam writing
 - (Xradia, CXRO, Agere/SB, Göttingen, Trieste, Kings, etc)
 - current method of choice for high resolution patterning
 - Direct write into resist, very high resolution (<15nm demonstrated)
- ❑ Optical, Holographic Patterning (Göttingen)
 - First high resolution zone plates for x-ray imaging at synchrotron
 - Limited by diffraction of light used (wavelength)
 - New efforts with EUV radiation at synchrotrons
- ❑ Sputtered, sliced (Göttingen, Japan)
 - Engineering challenges have proven hard to overcome
- ❑ Imprint litho (U. Texas/SB)
 - Master for imprint has to be fabricated using E-beam techniques
 - Imprint mainly motivated by mass-production aspect

Zone Plates By Electron Beam Lithography

- ❑ Produces the finest possible arbitrary 2-D structure (other than what nature can be persuaded to make by itself)
- ❑ Top end machines (such as JEOL JBX-9300FS, Vistec VB300) offer ~2 nm spot size at ~1 nA and 100 kV, 500 μm field, ~1 nm positioning with 5-10 nm absolute placement on a rectangular grid. DoE nanocenters have such systems

A. Stein at the NJNC JBX-9300FS

Zone plate efficiency and thickness

- ❑ For binary zones, 1:1 mark:space ratio.
- ❑ See Kirz, *J. Opt. Soc. Am.* **64**, 301 (1974)

Gold Zone Plate Efficiency

2 aligned ZPs

Quantitative Efficiency Measurements of Zone Plates Using Laboratory Sources

- Quantitative measurements have been restricted to synchrotron
- Balanced filter method to obtain monochromaticity with laboratory sources
- Final test and process development tool

Differential Filter

Poly-chromatic source output

Quantitative Efficiency Measurements of Zone Plates Using Laboratory Sources

Monochromatic projection x-ray image of 70nm zone width, 160um diameter ZP

$$Efficiency = (T - M_Z A_F) / (M_B A_Z) = 8.7\%$$

- ❑ Quantitative measurements that agree with synchrotron measurements
- ❑ Objective measurement of zone plate efficiency
- ❑ Efficiency of 70nm zone plate 700nm zone height measured at 73% of theoretical
- ❑ Agreement with synchrotron measurements

S. Chen, et al., *Journal of x-ray science and technology*, in press

M_B Bkgd intensity (counts/pixel)	M_Z Pedestal intensity (counts/pixel)	A_Z ZP area (pixels)	T focused intensity (counts)	A_F focus area (pixels)
9.40 ± 0.1	7.00 ± 0.009	157000 ± 7100	157000 ± 400	4100 ± 300

The Proximity Effect in E-Beam Lithography

Electrons in thick resist:
sidescatter

Thin resist, higher voltage:
reduce sidescatter blurring

Write two lines next to each other:
proximity effect

- ❑ Scattering limits the depth of structures that can be produced
- ❑ High (100keV) voltage e-beam writing preferred
- ❑ Dense gratings (such as zone plates) suffer proximity effect leading to collapse of tall structures
- ❑ Direct write to produce zone plate limited to small zone height

Avoiding the Proximity Effect

- Proximity effect can be reduced by splitting the process of producing a zone plate into two steps

Heroic efforts at Lawrence Berkeley Lab

- ❑ Chao *et al.*, *Nature* **435**, 1210 (2005):
15.1 nm half-pitch multilayer slice
imaged with a 15 nm outermost zone
width zone plate.
 - Efficiency ~3%.
 - Focal length if used at 290 eV
edge: 100 μm .
- ❑ Other results: 9.2% at 20 nm: Peuker,
Appl. Phys. Lett. **78**, 2208 (2001)

Tri-Level Processing Scheme

- Write high resolution pattern in top layer.
- Use highly directional reactive ion etching to transfer to a hard mask, and then into a secondary mask. Tennant et al., JVST 19, 1304 (1981); Schneider et al., JVST B 13, 2809 (1995); Spector et al., JVST B 15, 2872 (1997).

1. E-beam expose, develop

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3. Etch plating mold; strip hard mask

Tri-Level Processing Scheme

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1. E-beam expose, develop

2. Etch hard mask

3. Etch plating mold; strip hard mask

4. Metal plating

Zone Plates: Stony Brook

- Spector *et al.* [*J. Vac. Sci. Tech. B* **15**, 2872 (1997)], Stein *et al.* [*J. Vac. Sci. Tech. B* **21**, 214 (2003)], Lu *et al.* [*J. Vac. Sci. Tech. B* **24**, 2881 (2006)].
- Support from NSF and from BNL, collaboration with Don Tennant (Lucent/NJNC; now Cornell)

30 nm wide, 130 nm tall Ni, 160 μm diameter

18 nm wide, 60 nm tall Ni, 80 μm diameter

Hard x-ray zone plates from Xradia Inc.

Gold zone plates, Xradia, Inc.: 70 nm outermost zones

Recent Fabrication Highlights at Xradia

Cross section view of control structure

Single ZP:
24nm zones
133µm dia
300nm height

- ❑ 32nm gold zone plates, 450nm thick fabricated for CNM nanoprobe project (Xradia under contract), AR=14
- ❑ 24nm available now (330nm thick, AR=14), procedure developed to align and bond two ZPs to double AR and thickness (660nm thick, AR=28)

High-resolution, High-efficiency Zone Plates

ZP Alignment

Single ZP:
24nm zones
133µm dia
300nm height

- ❑ High-resolution zone plates usually low efficiency
- ❑ Alignment to increase zone height increases efficiency
- ❑ 24nm zone width zone plates with combined 600nm height in use at ANL ID-26 nanoprobe.

Disposable zone plates?

- ❑ Nanoimprint lithography: many cheap copies from one master.
- ❑ These slides: step-and-flash imprint lithography (SFIL) as pioneered by Wilson, Srinivasan *et al.*, UT Austin

Step 1: template approaches liquid transfer layer

Template

Release layer

Transfer layer

Etch layer

31

Disposable zone plates?

- ❑ Nanoimprint lithography: many cheap copies from one master.
- ❑ These slides: step-and-flash imprint lithography (SFIL) as pioneered by Wilson, Srinivasan *et al.*, UT Austin

Step 2: compress liquid transfer layer, and UV flash to harden

Template

Release layer

Transfer layer

Etch layer

32

Disposable zone plates?

- ❑ Nanoimprint lithography: many cheap copies from one master.
- ❑ These slides: step-and-flash imprint lithography (SFIL) as pioneered by Wilson, Srinivasan *et al.*, UT Austin

Step 3: remove template

Template

Release layer

Transfer layer

Etch layer

33

Disposable zone plates?

- ❑ Nanoimprint lithography: many cheap copies from one master.
- ❑ These slides: step-and-flash imprint lithography (SFIL) as pioneered by Wilson, Srinivasan *et al.*, UT Austin

Step 4: etch transfer layer to break through to etch layer

34

Disposable zone plates?

- ❑ Nanoimprint lithography: many cheap copies from one master.
- ❑ These slides: step-and-flash imprint lithography (SFIL) as pioneered by Wilson, Srinivasan *et al.*, UT Austin

Step 5: etch through the etch layer

35

SFIL zone plates: basic demonstration

50 nm zones replicated in transfer layer from quartz wafer. Stein *et al.*, *JVST B* **21**, 214 (2003)

Sputter-sliced or “jelly roll” zone plates

- ❑ It's easy to make thin layers! Successive layer deposition on a rotating wire. First proposed by Schmahl and Rudolph in 1980 (Ash, *Scanned Image Microscopy*).
- ❑ Many efforts, including Göttingen, Livermore, SPring-8, ESRF...
- ❑ Challenges: circularity, error accumulation....

Multilayer Laue lenses

- ❑ Forget top-down circles, and go sideways! Start by depositing thinnest zones first on a flat substrate, and work your way up to thicker zones. Cross two 1D lenses for 2D focusing.
- ❑ For thick optics, you want to tilt to be on the Bragg condition anyway [Maser, PhD thesis; Maser and Schmahl, *Opt. Comm.* **89**, 355 (1992)]

NSLS II: stated goal is 1 nm resolution using MLLs (or kinoform refractive lenses)

J. Maser et al., *SPIE* **5539**, 185 (2004); plus tests by Liu et al., *J. Appl. Phys.* **98**, 113519 (2005); Kang et al., *Appl. Phys. Lett.* **86**, 151109 (2005); Kang et al., *Phys. Rev. Lett.* **96**, 127401 (2006).

MLLs are not without challenges

Must stay on-Bragg for
good efficiency

High resolution: lots of layers!

- Transverse resolution $\Delta_t = 1.22 \Delta_{rN}$, where Δ_{rN} = outermost zone width
- Diameter $d = 1.22 \lambda f / \Delta_t$, # zones $N = 1.22^2 \lambda f / \Delta_t^2$

Challenge at high resolution: depth of focus

- Depth of focus is $1.22\lambda/\text{N.A.}$, or $4.88(dr_N)^2/\lambda$ where dr_N is the outermost zone width

How clean is the focus?

- $a = (\text{central stop diameter}) / (\text{zone plate diameter})$

Circular

$a=0$

$a=0.5$

$a=0.9$

Crossed
cylindrical

You really want all zones!

X-ray optics: best resolution

X-ray optics: best resolution

- ❑ Microscope objective
- ❑ Condenser/monochromator
- ❑ Microprobe forming lens
- ❑ Beam splitter
- ❑ Works for any wave (including neutrons and atoms)

ZP based x-ray microscope

Full-field and scanning

❑ TXM

- ❑ Incoherent illumination; works well with a bending magnet, with fast imaging
- ❑ More pixels (e.g., 2048^2)
- ❑ Optic efficiency specimen dose
- ❑ Moderate spectral resolution if zone plate condenser used - but most new TXMs use grating/crystal and reflective condenser!

TXM: transmission x-ray microscope

STXM

- Coherent illumination; works best with an undulator
- Less dose to sample ($\sim 10\%$ efficient ZP)
- Better suited to conventional grating monochromator [high $E/(\Delta E)$]
- Microprobes: fluorescence etc.

STXM: scanning transmission x-ray microscope

X-Ray Advantage: High-resolution imaging of buried Structures

Computed Tomography

(1) Sample imaged at various angles to acquire tomographic projections.

Sinogram

Line Image

Sample rotation angle

(2) 3D reconstruction by backprojection results in 3D image of the sample.

Imaging Length Scale Coverage

Loss of contrast and throughput leads to a crossover at $\sim 1\mu\text{m}$ resolution

X-ray Imaging with High-resolution (nanoXCT) Optics and High Contrast

□ Key components:

- High efficiency, reflective ellipsoidal capillary condenser
- High-resolution objective zone plate
- Zernike phase contrast phase plate
- High-efficiency, high resolution x-ray detector
- Precision tomography stages

nm-scale: Tubular Fuel Cell (SOFC)

Courtesy of Dr. W. Chiu (U. Connecticut), Adaptive Materials Inc.

2D transmission x-ray images

Xradia nanoXCT-8-50-Z
8keV x-ray energy (stand alone)
sub-50nm resolution
Zernike phase contrast

Tubular Fuel Cell (SOFC)

2D transmission x-ray projection
images (0-90 degree rotation)

Reconstructed 3D volume

SOFC Pore Structure Imaging – cont'd

□ nanaoXCT Experiment details

- Xradia (Concord, CA)
- 8 keV copper source
- 181 projections at 300 sec per projection
- 22.6 μm field of view
- 50 nm resolution
- 3-D tomographic reconstruction

Putting It All Together

M1 Metal Layer IC Sample

W-plugs

Magnified Image
Minim. Feature size: 120 nm
Resolution: < 60 nm

Raw Image Data of Cu-Interconnect

- ❑ Cr 5.4keV x-ray energy
- ❑ Cu interconnect sample with 5 layers of trenches and interconnecting vias
- ❑ 6 hr data collection time
- ❑ Bright structures are Cu

A solid black horizontal line representing a scale bar.

1 μm

CT Reconstruction – Planar Slices

Slice: 0

- Cu Layers, and W Layer clearly resolved

Extracted Layers of Pentium 4 chip (120nm node)

Cu (8 keV) Laboratory x-ray source
Zernike Phase contrast Imaging

1 μ m

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SPIE X-ray Focusing Workshop

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Technologically Relevant Application: TSV - Through-Silicon Vias (10 μ m Diameter)

10 μ m vias tilted at 45 degree.

Tile of 3x3 images 66x66 μ m each

**All vias have missing electroplating
(key hole) in center**

50 μ m

Large field of view mode
2 minute exposure per tile

Detailed X-ray Tomography of Single Via

nanoXCT allows direct visualization of the key hole without physical cross-sectioning

Scanning Microscope Geometry and Detectors

**Coherent X-ray
Beam**

Sample

**Transmission
Detector
“Structure”**

Zone Plate

OSA

Laser Encoder

Closed loop

**Fluorescence / Diffraction
Detector
“Phase / Chemistry”**

- ❑ 30nm x-ray probe probes local elemental composition (fluorescence) and crystallinity (diffraction)
- ❑ Vibration and drift stability between zone plate and sample are critical for performance
 - laser encoding and stiff stages needed!
- ❑ Platform also suited for other coherence based experiments such as diffraction imaging, zone plate holographic methods

Sub-nm Resolution Laser Doppler Encoder

Application Example Fluorescence: Marine Biology

Trace Metals in Plankton and Global Carbon Balance

- Standard approach: bulk analysis
- X-ray microscopy: separate and study individual organism

Visible light

- B. Twining, S. Baines *et al.*, Marine Sciences Research Center, SUNY Stony Brook
- A. Osanna, C. Vaa, B. Winn, S. Wirick, C. Jacobsen, Dept. of Physics & Astronomy, SUNY Stony Brook

Composite:
Cl, Fe, Cu

Application Example Diffraction: - Quantum dot stressors on Si Nanomembranes

*Ge-Si Lattice Lattice Mismatch
Distorts Si Membrane*

Prediction: F. Liu, M. Huang, P. Rugheimer, D. E. Savage, and M. G. Lagally, Phys. Rev. Lett. (2002)

Microdiffraction at
APS Sector 2
 $h\nu = 11.2 \text{ keV}$
200 nm spot

University of Wisconsin MRSEC IRG1

P. G. Evans, D. S. Tinberg, M. M. Roberts, M. G. Lagally,
Y. Xiao, B. Lai, and Z. Cai, *APL* **87**, 073112 (2005).

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Summary / Outlook

Acknowledgments

NSRRC

Gung-Chian Yin, Y. Whu, K. Liang

SSRL

P. Pianetta, S. Brennan, K. Lüning

APS

**Q. Shen, Y. Chu, W-K Lee,
J. Maser, S. Vogt, B. Lai**

BSRF

Ziyu Wu, Peiping Zhu

UCSF, NASA Ames

E. Almeida

Cornell

M. van der Meulen

IBM

D. Valett

AMD

E. Zschech, A. Vairagar

Xradia, Inc. team

NSRRC (Taiwan)

**SSRL BL-6-2
(Stanford, USA)**

**APS ID-32, ID-26
Argonne, USA**

Acknowledgments

- ❑ Chris Jacobsen (Stony Brook), Janos Kirz (LBL) for supplying parts of the material for this presentation
- ❑ Customers, collaborators and colleagues for data and information

Thank you for your attention!