Advances in X-Ray/EUV Optics and Components III

Ali M. Khounsary
Christian Morawe
Shunji Goto
Editors

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## SESSION 1 MULTILAYERS

<table>
<thead>
<tr>
<th>Session</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
<tbody>
<tr>
<td>7077 02</td>
<td>Multilayer optics under CHESS A2 wiggler beam [7077-01]</td>
<td>A. Kazimirov, P. Revesz, Cornell Univ. (United States); R. Huang, Univ. of Chicago (United States)</td>
</tr>
<tr>
<td>7077 03</td>
<td>Ion beam sputtering of x-ray multilayer mirrors [7077-02]</td>
<td>P. Gawlitza, S. Braun, G. Dietrich, M. Menzel, S. Schädlich, A. Leson, Fraunhofer-Institut für Werkstoff- und Strahltechnik (Germany)</td>
</tr>
<tr>
<td>7077 04</td>
<td>Surface roughness analysis of multilayer x-ray optics [7077-03]</td>
<td>V. V. Martynov, Y. Y. Platonov, Rigaku Innovative Technologies, Inc. (United States)</td>
</tr>
<tr>
<td>7077 05</td>
<td>Single-layer and multilayer mirrors for current and next-generation light sources [7077-04]</td>
<td>M. Störmer, C. Horstmann, GKSS-Forschungszentrum Geesthacht GmbH (Germany); D. Häussler, E. Spiecker, Christian-Albrechts-Universität Kiel (Germany); F. Siewert, BESSY Berliner Elektronenspeicherring-Gesellschaft für Synchrotronstrahlung mbH (Germany); F. Scholze, Physikalisch-Technische Bundesanstalt (Germany); F. Hertlein, Incoatec GmbH (Germany); W. Jäger, Christian-Albrechts-Universität Kiel (Germany); R. Bormann, GKSS-Forschungszentrum Geesthacht GmbH (Germany)</td>
</tr>
<tr>
<td>7077 08</td>
<td>Fabrication and characterization of a new high density Sc/Si multilayer sliced grating [7077-07]</td>
<td>D. L. Voronov, R. Cambie, E. M. Gullikson, V. V. Yashchuk, H. A. Padmore, Lawrence Berkeley National Lab. (United States); Y. P. Pershin, A. G. Ponomarenko, V. V. Kondratenko, National Technical Univ. KhPI (Ukraine)</td>
</tr>
</tbody>
</table>

## SESSION 2 MIRRORS + METROLOGY

<table>
<thead>
<tr>
<th>Session</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
<tbody>
<tr>
<td>7077 09</td>
<td>Development of adaptive mirror for wavefront correction of hard x-ray nanobeam [7077-09]</td>
<td>T. Kimura, S. Handa, H. Mimura, Osaka Univ. (Japan); H. Yumoto, SPring-8/Japan Synchrotron Radiation Research Institute (Japan); D. Yamakawa, S. Matsuyama, Y. Sano, Osaka Univ. (Japan); K. Tamasaku, Osaka Univ. (Japan) and SPring-8/RIKEN (Japan); Y. Nishino, SPring-8/RIKEN (Japan); M. Yabashi, SPring-8/Japan Synchrotron Radiation Research Institute (Japan); T. Ishikawa, SPring-8/Japan Synchrotron Radiation Research Institute (Japan) and SPring-8/RIKEN (Japan); K. Yamauchi, Osaka Univ. (Japan) and Research Ctr. for Ultra-Precision Science &amp; Technology (Japan)</td>
</tr>
</tbody>
</table>
**SESSION 3  **  X-RAY LENSES  

| 7077 0B | Distance-dependent influences on angle metrology with autocollimators in deflectometry  
[7077-11]  
R. D. Geckeler, A. Just, Physikalisch-Technische Bundesanstalt (Germany)  
| 7077 0C | Development of surface gradient integrated profiler: precise coordinate determination of normal vector measured points by self-calibration method and new data analysis from normal vector to surface profile  
[7077-12]  
Y. Higashi, High Energy Accelerator Research Organization (Japan); T. Ueno, K. Enoki, J. Uchikoshi, Osaka Univ. (Japan); T. Kume, K. Enami, High Energy Accelerator Research Organization (Japan)  
| 7077 0D | Statistical analysis of the metrological properties of float glass  
[7077-13]  
B. W. Yates, A. M. Duffy, Canadian Light Source, Inc. (Canada)  
| 7077 0E | Opto-mechanical design considerations for the Linac Coherent Light Source x-ray mirror system  
[7077-14]  
T. J. McCarville, Lawrence Livermore National Lab. (United States); P. M. Stefan, Stanford Linear Accelerator Ctr. (United States); B. Woods, R. M. Bionta, R. Souflis, M. J. Pivovaroff, Lawrence Livermore National Lab. (United States)  

**SESSION 4  **  CRYSTALS + DIFFRACTION  

| 7077 0G | Numerical simulations of achromatic x-ray lenses  
[7077-17]  
M. Umbach, Forschungszentrum Karlsruhe (Germany); V. Nazmov, Univ. Karlsruhe (Germany); M. Simon, Forschungszentrum Karlsruhe (Germany); A. Last, Univ. Karlsruhe (Germany); V. Saile, Forschungszentrum Karlsruhe (Germany) and Univ. Karlsruhe (Germany)  
| 7077 0H | X-ray imaging with compound refractive lens and microfocus x-ray tube  
[7077-18]  
L. Pina, Czech Technical Univ. in Prague (Czech Republic); Y. Dudchik, Belarus State Univ. (Belarus); V. Jelinek, Reflex s.r.o. (Czech Republic); L. Sveda, Czech Technical Univ. in Prague (Czech Republic); J. Marsik, M. Horvath, O. Petr, Reflex s.r.o. (Czech Republic)  
| 7077 0J | Multi-plate crystal cavity with compound refractive lenses  
[7077-20]  
S.-Y. Chen, Y.-Y. Chang, National Tsing Hua Univ. (Taiwan); M.-T. Tang, Yu. Stetsko, National Synchrotron Radiation Research Ctr. (Taiwan); M. Yabashi, Spring-8/RIKEN (Japan); H.-H. Wu, Y.-R. Lee, National Tsing Hua Univ. (Taiwan); B.-Y. Shew, National Synchrotron Radiation Research Ctr. (Taiwan); S.-L. Chang, National Tsing Hua Univ. (Taiwan)  
| 7077 0K | Diffraction imaging with conventional sources  
[7077-21]  
W. Zhou, C. A. MacDonald, Univ. at Albany (United States)  
| 7077 0L | Bragg diffraction of a focused x-ray beam as a new depth sensitive diagnostic tool  
[7077-22]  
A. Kazimirov, Cornell Univ. (United States); V. G. Kohn, Russian Research Ctr. Kurchatov Institute (Russia); Z.-H. Cai, Advanced Photon Source (United States)  
| 7077 0M | Focused beam powder diffraction with polycapillary and curved crystal optics  
[7077-23]  
A. Bingölbalı, W. Zhou, D. N. Mahato, C. A. MacDonald, Univ. at Albany (United States)  

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Investigation of polycrystalline structure of CVD diamond using white-beam x-ray diffraction
[7077-24]
A. Souvorov, K. Kajiwara, H. Kimura, S. Goto, SPring-8/Japan Synchrotron Radiation Research Institute (Japan); T. Ishikawa, SPring-8/RIKEN (Japan)

Mosaic GaAs crystals for hard x-ray astronomy [7077-25]
C. Ferrari, L. Zanotti, A. Zappettini, IMEM Institute, CNR (Italy); S. Arumainathan, Univ. of Madras (India)

An application of the grazing-angle incidence hard x-ray optical nanoscope in ultra-high density digital data read-out device [7077-26]
H. P. Bezirganyan, Yerevan State Univ. (Armenia); S. E. Bezirganyan, Yerevan State Medical Univ. (Armenia); P. H. Bezirganyan, Jr., State Engineering Univ. of Armenia (Armenia); H. H. Bezirganyan, Jr., Yerevan State Univ. (Armenia)

A theoretical study of two-dimensional point focusing by two multilayer Laue lenses [7077-27]
H. Yan, Brookhaven National Lab. (United States) and Argonne National Lab. (United States); J. Maser, Argonne National Lab. (United States); H. C. Kang, Argonne National Lab. (United States) and Gwangju Institute of Science and Technology (Republic of Korea); A. Macrander, B. Stephenson, Argonne National Lab. (United States)

Fabrication of a 400-mm-long mirror for focusing x-ray free-electron lasers to sub-100 nm [7077-28]
H. Mimura, Osaka Univ. (Japan); S. Morita, Wakou/RIKEN (Japan); T. Kimura, D. Yamakawa, Osaka Univ. (Japan); W. Lin, Akita Prefectural Univ. (Japan); Y. Uehara, Wakou/RIKEN (Japan); H. Yumoto, SPring-8/Japan Synchrotron Radiation Research Institute (Japan); S. Matsuyma, Osaka Univ. (Japan); Y. Nishino, K. Tamasaku, SPring-8/RIKEN (Japan); H. Ohashi, SPring-8/Japan Synchrotron Radiation Research Institute (Japan); M. Yabashi, T. Ishikawa, SPring-8/Japan Synchrotron Radiation Research Institute (Japan) and SPring-8/RIKEN (Japan); H. Ohmori, Wakou/RIKEN (Japan); K. Yamauchi, Osaka Univ. (Japan)

Aberrations in curved x-ray multilayers [7077-30]
Ch. Morawe, J.-P. Guigay, European Synchrotron Radiation Facility (France); V. Mocella, European Synchrotron Radiation Facility (France) and Istituto per la Microelettronica e Microsistemi, CNR (Italy); C. Ferrero, European Synchrotron Radiation Facility (France); H. Mimura, S. Handa, K. Yamauchi, Osaka Univ. (Japan)

X-ray microfocusing by polycapillary optics [7077-31]
D. Hampai, INOA-CNR (Italy); INFN-LNF (Italy) and Univ. di Roma Tor Vergata (Italy); S. B. Dabagov, INFN-LNF (Italy) and P. N. Lebedev Physical Institute (Russian Federation); G. Cappuccio, INFN-LNF (Italy); A. Longoni, T. Frizzi, Politecnico di Milano (Italy); G. Cibin, Diamond Light Source Ltd. (United Kingdom); V. Guglielmetti, Univ. di Roma Tor Vergata (Italy); M. Sala, Univ. degli Studi di Milano (Italy); V. Sessa, Univ. di Roma Tor Vergata (Italy)

Polycapillary x-ray microbeams [7077-32]
A. Yu. Romanov, Institute for Roentgen Optics (Russia)
<table>
<thead>
<tr>
<th>Session</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
<tbody>
<tr>
<td>SESSION 6</td>
<td>Micro and imaging x-ray analysis by using polycapillary x-ray optics [7077-33]</td>
<td>K. Tsuji, K. Nakano, M. Yamaguchi, T. Yonehara, Osaka City Univ. (Japan)</td>
</tr>
<tr>
<td></td>
<td>X-RAY SOURCES</td>
<td>R. Schmitz, A. Bingölbalı, A. Hussain, C. A. MacDonald, SUNY/Univ. at Albany (United States)</td>
</tr>
<tr>
<td>SESSION 7</td>
<td>Development of polarized and monochromatic x-ray beams from tube sources [7077-37]</td>
<td>L. Poletto, F. Frassetto, P. Villoresi, INFM, Univ. degli Studi di Padova (Italy)</td>
</tr>
<tr>
<td></td>
<td>XUV OPTICS + APPLICATIONS</td>
<td>J. I. Larruquert, J. A. Mendez, Consejo Superior de Investigaciones Científicas (Spain)</td>
</tr>
<tr>
<td></td>
<td>Design of a beam separator for high-order harmonics below 10 nm [7077-42]</td>
<td>F. Frassetto, L. Poletto, INFM, Univ. degli Studi di Padova (Italy); J. I. Larruquert, J. A. Mendez, Consejo Superior de Investigaciones Científicas (Spain)</td>
</tr>
<tr>
<td></td>
<td>Efficiency measurements on gratings in the off-plane mount for a high-resolution grazing-incidence XUV monochromator [7077-43]</td>
<td>L. Poletto, F. Frassetto, P. Villoresi, INFM, Univ. degli Studi di Padova (Italy); J. I. Larruquert, J. A. Mendez, Consejo Superior de Investigaciones Científicas (Spain)</td>
</tr>
<tr>
<td></td>
<td>Design and characterization of the XUV monochromator for ultrashort pulses at the ARTEMIS facility [7077-44]</td>
<td>F. Frassetto, S. Bonora, P. Villoresi, L. Poletto, INFM, Univ. degli Studi di Padova (Italy); E. Springate, C. A. Froud, I. C. E. Turcu, A. J. Langley, D. S. Wolff, J. L. Collier, Rutherford Appleton Lab. (United Kingdom); S. S. Dhesi, Diamond Light Source Ltd., Rutherford Appleton Lab. (United Kingdom); A. Cavalleri, Univ. of Oxford (United Kingdom)</td>
</tr>
<tr>
<td></td>
<td>Innovative approaches to surface sensitive analysis techniques on the basis of plasma-based off-synchrotron XUV/EUV light sources [7077-45]</td>
<td>M. Banyay, L. Juschkin, RWTH Aachen Univ. (Germany)</td>
</tr>
<tr>
<td></td>
<td>Transmittance and optical constants of evaporated Pr, Eu, and Tm films in the 4-1600 eV spectral range [7077-46]</td>
<td>M. Fernández-Perea, M. Vidal-Dasilva, J. A. Aznárez, J. I. Larruquert, J. A. Méndez, Consejo Superior de Investigaciones Científicas (Spain); L. Poletto, D. Garoli, INFM, CNR (Italy) and Univ. degli Studi di Padova (Italy); A. M. Malvezzi, Univ. degli Studi di Pavia (Italy) and Consorzio Nazionale Interuniversitario per le Scienze Fisiche della Materia (Italy); A. Giglia, Lab. TASC-INFM, CNR (Italy); S. Nannarone, Lab. TASC-INFM, CNR (Italy) and Univ. degli Studi di Modena e Reggio Emilia (Italy)</td>
</tr>
<tr>
<td>SESSION 8</td>
<td>BEAMLINE OPTICS</td>
<td>R. Soufl, M. J. Pivovaroff, S. L. Baker, J. C. Robinson, Lawrence Livermore National Lab. (United States); E. M. Gullikson, Lawrence Berkeley National Lab. (United States); T. J. Mccarville, Lawrence Livermore National Lab. (United States); P. M. Stefan, Stanford Linear Accelerator Ctr. (United States); A. L. Aquila, Lawrence Berkeley National Lab. (United States); J. Ayers, M. A. McKernan, R. M. Bionta, Lawrence Livermore National Lab. (United States)</td>
</tr>
</tbody>
</table>
Present status of stability improvement of SPring-8 standard x-ray monochromators [7077-50]
H. Yamazaki, Japan Synchrotron Radiation Research Institute (Japan); Y. Shimizu, SPring-8 Service Co., Ltd. (Japan); N. Shimizu, M. Kawamoto, Japan Synchrotron Radiation Research Institute (Japan); Y. Kawano, RIKEN SPring-8 Ctr. (Japan); Y. Senba, H. Ohashi, S. Goto, Japan Synchrotron Radiation Research Institute (Japan)

Development of ultrahigh-resolution inelastic x-ray scattering optics [7077-51]
X.-R. Huang, Z. Zhong, Y. Q. Cai, S. Coburn, Brookhaven National Lab. (United States)

Crystal quality analysis and improvement using x-ray topography [7077-52]
J. A. Maj, K. Goetze, A. T. Macrander, Y. C. Zhong, X. R. Huang, Argonne National Lab. (United States); L. Maj, The Univ. of Chicago (United States)

Calibration of MCP transmissivity from 2-5.5keV [7077-54]
Z. Cao, H. Li, J. Dong, S. Wu, R. Yi, CAE Research Ctr. of Laser Fusion (China)

Ray traces of an arbitrarily deformed double-crystal Laue x-ray monochromator [7077-55]
J. P. Sutter, T. Connolley, M. Drakopoulos, T. P. Hill, D. W. Sharp, Diamond Light Source Ltd. (United Kingdom)

Diamond detectors for x-ray spectroscopy [7077-57]
P. Allegrini, M. Girolami, P. Calvani, G. Conte, S. Salvatori, E. Spiriti, Univ. Roma Tre, INFN, National Institute for Nuclear Physics (Italy); V. Ralchenko, Natural Science Ctr. of General Physics Institute (Russian Federation)

X-ray prism lenses with large apertures [7077-58]
M. Simon, Forschungszentrum Karlsruhe (Germany); E. Reznikova, V. Nazmov, A. Last, Univ. Karlsruhe (Germany); W. Jark, Sincrotrone Trieste S.c.p.A. (Italy)

EUV polarimetry with single multilayer optical element [7077-59]
S. Zuccon, INFN-LUXOR, Univ. degli Studi di Padova (Italy) and Lab. TASC-INFN (Italy); M.-G. Pelizzo, P. Nicolosi, INFN-LUXOR, Univ. degli Studi di Padova (Italy); A. Giglia, Lab. TASC-INFN (Italy) and Univ. degli Studi di Modena e Reggio Emilia (Italy); N. Mahne, Lab. TASC-INFN (Italy); S. Nannarone, Lab. TASC-INFN (Italy) and Univ. degli Studi di Modena e Reggio Emilia (Italy)

Graded multilayer mirrors for the carbon window Schwarzschild objective [7077-61]
I. A. Artyukov, P.N. Lebedev Physical Institute (Russia); Y. A. Bugayev, O. Y. Devizenko, National Technical Univ. Kharkov Polytechnical Institute (Ukraine); E. M. Guillikson, Lawrence Berkeley National Lab. (United States); V. V. Kondratenko, National Technical Univ. Kharkov Polytechnical Institute (Ukraine); Y. A. Uspenski, A. V. Vinogradov, P.N. Lebedev Physical Institute (Russia); D. L. Voronov, Lawrence Berkeley National Lab. (United States)

Development of an extreme ultraviolet spectroscope for exospheric dynamics (EXCEED) mission [7077-62]
K. Yoshioka, G. Murakami, M. Ueno, I. Yashikawa, The Univ. of Tokyo (Japan); A. Yamazaki, K. Uemizu, Japan Aerospace Exploration Agency (Japan)
Development of EUV multilayer mirrors for astronomical observation in IPOE [7077-63]
J. Zhu, X. Wang, J. Xu, R. Chen, Q. Huang, L. Bai, Z. Zhang, Z. Wang, L. Chen, Tongji Univ. (China)

Enhanced reflectivity and stability of high-temperature LPP collector mirrors [7077-64]
T. Feigl, S. Yulin, M. Perske, H. Pauer, M. Schürmann, N. Kaiser, Fraunhofer-Institut für Angewandte Optik und Feinmechanik (Germany); N. R. Böwering, O. V. Khodykin, I. V. Fomenkov, D. C. Brandt, Cymer, Inc. (United States)

The role of spatial coherence, diffraction, and refraction in the focusing of x-rays with prism arrays of the Clessidra type [7077-65]
W. Jark, M. Matteucci, R. H. Menk, Sincrotrone Trieste S. c. p. A. (Italy); L. Rigon, Univ. degli Studi di Trieste (Italy); L. De Caro, Istituto di Cristallografia, CNR (Italy)

Multilayers and crystal for a multi-bandpass monochromator [7077-66]
R. Feng, Canadian Light Source, Inc. (Canada); Y. Platonov, D. Broadway, Rigaku Innovative Technologies, Inc. (United States); G. Ice, Oak Ridge National Lab. (United States); A. Gerson, The Univ of South Australia (Australia); S. McIntyre, The Univ. of Western Ontario (Canada)

Author Index
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Session Chairs
1 Multi-layers
   Christian Morawe, European Synchrotron Radiation Facility (France)
2 Mirrors + Metrology
   Ali M. Khounsary, Argonne National Laboratory (United States)
3 X-Ray Lenses
   Werner H. Jark, Sincrotrone Trieste S.C.p.A. (Italy)
4 Crystals + Diffraction
   Shunji Goto, Japan Synchrotron Radiation Research Institute (Japan)
5 Focusing
   Ladislav Pina, Czech Technical University in Prague (Czech Republic)
6 X-Ray Sources
   Carolyn A. MacDonald, SUNY/University at Albany (United States)
7 XUV Optics + Applications
   Regina Soufl, Lawrence Livermore National Laboratory (United States)
8 Beamline Optics
   Alexander Yu. Kazimirov, Cornell University (United States)

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)

Hard x-ray focusing with curved reflective multilayers, Christian Morawe, European Synchrotron Radiation Facility (France)

Refractive x-ray lenses for hard x-ray microscopy, Christian G. Schroer, Technische Univ. Dresden (Germany)

Kinoform x-ray lens arrays, Werner H. Jark, Sincrotrone Trieste (Italy)

Monocapillary optics, Ladislav Pina, Czech Technical Univ. (Czech Republic) (presentation not available)

X-ray focusing with polycapillary optics, Carolyn A. MacDonald, SUNY, Univ. at Albany

Focusing of x-rays using crystal optics, Eckhart Förster, Friedrich-Schiller-Univ. Jena (Germany)

Multilayer Laue Lens for efficient nanometer focusing of hard x-rays, G. B. Stephenson, Argonne National Lab.

Diffractive 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

Coherent Imaging group
at PSI & EPFL

Franz Pfeiffer
Oliver Bunk
Andreas Menzel
Xavier Donath
Pierre Thibault
Cameron Kewish
Martin Dierolf
Tobias Boehlen

Christian David, Joan Vila, Konstantins Jefimovs, Vitaliy Guzenko, Harun Solak, Sankha Sarkar,
Laboratory for Micro- and Nanotechnology,
Paul Scherrer Institut, CH
1. X-ray focusing – Why
   - Requires coherent illumination
   - Only x-ray transmission signal
   - Many detection schemes possible (elemental & chemical specificity, crystalline ordering, …)
   - Requires coherent illumination
   - Fast

X-ray Focusing – Why

Full-field microscope

Scanning microscope
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 mm
- SAXS gives access to length scales between 1 nm – 1 micron

~ $10^5$ SAXS patterns per hour
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
**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

---

**X-ray waveguides**

X-rays
- \( \lambda \sim 0.1 \text{ nm}, \ d \sim 50 \text{ nm} \)

![Waveguide Diagram](image)

Transmission \(<\!\!\!< 1\%\)

Y. P. Feng et al., Phys. Rev. Lett. 71, 537 (1993); 
S. Lagomarsino et al., Appl. Phys. Lett. 71, 2557 (1997); 
Resonant beam coupling waveguides

\[ \alpha_{c, n_1} < \alpha_i < \alpha_{c, n_2} \]

Field distribution in 1D resonant beam coupling waveguides


5 nm Cr/ 50 nm PMMA/ 20 nm Cr, \( \lambda = 0.1 \) nm

2D Waveguides


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

Farfield pattern of modes in 2D x-ray waveguides

**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
Spatial resolution limit in x-ray microscopy

\[ \delta_{\text{res}} \sim \Delta r = \frac{p}{2} \]

**e-beam lithography limits**

- **Isolated** lines (10 – 20 nm)
- **Dense** lines (10 – 20 nm)

**Possible solutions**

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

**The Swiss alternatives:**

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

Advantages in relation to e-beam lithography:

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

Both semitransparent window and pinhole

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
Δr_N ~ 260 nm
Zone-doubling for high resolution FZP (slides J. Vila)

A

\[ \delta_{\text{res}} \sim \Delta r = p/2 \]

B

\[ \delta_{\text{res}} \sim w = p/4 \]

Manufacturing advantages:

✓ No alignment required  ✓ One single EBL exposure


Low refractive index material

High refractive index material

Resolution 2x

FZP pattern
generation is
simple and
reproducible!

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

1) Substrate preparation and e-beam lithography

(0.25 duty cycle at the center region)

2) Cr mask dry etching

in a Cl\(_2\), CO\(_2\) plasma

3) Silicon RIE

in a CH\(_3\)F\(_2\)SF\(_6\):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 \( D = 100 \mu m, \Delta r = 20 \text{ nm} \)

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

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

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

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

20 \( \mu m \) diameter central stops made of 1.5 \( \mu m \) thick layer of Pt by FIB induced deposition.
STXM @ PolLux beamline of SLS (J. Raabe & G. Tzvetkov)

15 nm lines/spacings (30 nm period)
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

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

Some time ago a new principle was proposed for the registration of the complete information (amplitudes and phases) in a diffraction diagram, which does not—as does Holography—require the interference of the scattered waves with a single reference wave. The basis of the principle lies in the interference of neighboring scattered waves which result when the object function \( q(x,y) \) is multiplied by a generalized primary wave function \( p(x,y) \) in Fourier space (diffraction diagram) this is a convolution of the Fourier transforms of these functions. The above mentioned interferences necessary for the phase determination can be obtained by suitable choice of the shape of \( p(x,y) \). To distinguish it from holography this procedure is designated *ptychography* (DV=9; fold). The procedure is applicable to periodic and aperiodic structures. The relationships are simplest for plane lattices. In this paper the theory is extended to space lattices both with and without consideration of the dynamic theory. The resulting effects are demonstrated using a practical example.

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**Ptychographic phase retrieval**


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**Dynamische Theorie der Kristallstrukturanalyse durch Elektronenbeugung im inhomogenen Primärstrahlwellenfeld**

Some time ago a new principle was proposed for the registration of the complete information (amplitudes and phases) in a diffraction diagram, which does not—as does Holography—require the interference of the scattered waves with a single reference wave. The basis of the principle lies in the interference of neighboring scattered waves which result when the object function \( q(x,y) \) is multiplied by a generalized primary wave function \( p(x,y) \) in Fourier space (diffraction diagram) this is a convolution of the Fourier transforms of these functions. The above mentioned interferences necessary for the phase determination can be obtained by suitable choice of the shape of \( p(x,y) \). To distinguish it from holography this procedure is designated *ptychography* (DV=9; fold). The procedure is applicable to periodic and aperiodic structures. The relationships are simplest for plane lattices. In this paper the theory is extended to space lattices both with and without consideration of the dynamic theory. The resulting effects are demonstrated using a practical example.

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Ptychographic phase retrieval

- W. Hoppe,

- R. Hegerl, W. Hoppe, Ber.
  Bunsen-Ges. Phys. Chemie

LETTERS TO NATURE

Resolution beyond the ‘information limit’ in transmission electron microscopy

P. D. Nellist, S. G. McCellum & J. M. Rodenburg

Cavendish Laboratory, University of Cambridge, Madingley Road, Cambridge CB3 0HE, UK.

Test conventional resolution of transmission electron microscopes by orders of magnitude larger than the wavelength of the electron used. Alterations of the objective lens correct spatial information on length scales below a limit known as the point resolution. Methods to correct for lens aberrations now permit knowledge of the phase of the wave which makes up the image (this constitutes the ‘true problem’), beyond the point resolution, information can still be transferred by the microscope, but partial coherence of the illumination and diffraction effects as well as diffraction pattern contrast functions of the transmitted image information. Here we discuss some preliminary results.


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


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‘Super-Resolution’ Scanning X-Ray Transmission Microscopy

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

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

'Super-Resolution' Scanning X-Ray Transmission Microscopy

STXM image

10^4 coherent diffraction patterns in ~ 300 sec!

Simultaneous reconstruction of probe & specimen

probe

‘Super-Resolution’ X-Ray Microscopy

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

cytoskeleton
membrane bilayer
Summary

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

Swiss Light Source

Coherent Imaging group at PSI & EPFL

Franz Pfeiffer, Oliver Bunk, Andreas Menzel, Xavier Donath, Pierre Thibault, Cameron Kewish, Martin Dierolf, Tobias Boehlen

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

Achromatic optic (total reflection mirrors)

High efficiency $>90\%$
Large aperture $\approx 500\mu\text{m}$
Long working distance $10\text{mm} \sim 500\text{mm}$
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. Morawé 

75nm x 85nm focusing was achieved at APS by a optical figuring and a differential deposition. Short focal length Kirkpatrick-Baez mirrors for a hard x-ray nanoprobe 


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


Required accuracy for nano-focusing under D-limited condition

Kirkpatrick-Baez mirrors

Elliptical mirror × 2

Phase error = \frac{2d \sin \theta}{\lambda} \text{ wave}

Waves are in constructive interference state.

Diffraction-limited focusing

Beam profile

Designed profile (ellipse)

Error height:
- 2nm
- 4nm
- 6nm

Wave interference state.

Intensity (arb. unit)

Position (nm)

Error height:
- 2nm
- 4nm
- 6nm

Position (mm)
Fabrication and figure testing technologies of Osaka University

◎ Plasma CVM (chemical vaporization machining)
  → Rough figuring (Rapid figuring with sub-10nm (P-V) accuracy)

◎ EEM (elastic emission machining)
  → Final figuring and smoothing (Fine figuring and atomic smoothness)

◎ MSI (microstitching interferometry)
  → Figure tester with spatial resolution close to 1μm

◎ RADSI (relative-angle determinable stitching interferometry)
  → Figure tester for steeply curved ellipse of large NA mirror

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

Sub-100nm focusing mirrors have already been commercially available.
A chemical removal process utilizing reactive species generated in the atmospheric pressure plasma

- High density reactive species ⇒ High removal rate
- Chemical reaction ⇒ No damage on the surface
- Non contact ⇒ Insensitive against external disturbances

<table>
<thead>
<tr>
<th>Material</th>
<th>Removal rate (μm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fused silica</td>
<td>170</td>
</tr>
<tr>
<td>Silicon</td>
<td>94</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>36</td>
</tr>
<tr>
<td>Tungsten</td>
<td>32</td>
</tr>
<tr>
<td>Silicon carbide</td>
<td>6.4</td>
</tr>
<tr>
<td>Diamond</td>
<td>2.5</td>
</tr>
</tbody>
</table>
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

Planarization

Slicing

Figuring

Pipe electrode

Aspherical lens

Spherical type

Cylindrical type

Inner diameter blade type

Si, SiC

Si wafer

SOI wafer

Aspherical lens

SOI wafer

X-ray mirror

Aspherical lens

SOI wafer

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Figuring by Numerically Controlled Plasma CVM

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

Removal depth per scan [nm]

Inverse scanning speed of work table [min/mm]

Convolution

\[ h(x,y) \times f(x,y) \times g(x,y) \]
EEM (Elastic Emission Machining)

In EEM, chemical reaction between solid surfaces is utilized. The ultra-fine particle is supplied to the work surface by ultrapure water flow. Atom removal occurs selectively at the topmost site of the work surface. 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

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

The bump site is selectively removed.
95% of the EEM processed surface is constructed with only 3 atomic layers.

STM image of EEM processed surface

Distribution of atom classified for every atomic layer

1st layer (0.034%)
2nd layer (1.4%)
3rd layer (16.0%)
4th layer (47.9%)
5th layer (30.7%)
6th layer (4.0%)
Others (0.13%)

Typical figuring properties using EEM
Sub-30nm focusing

Designed configuration

From undulator

Slit (100 × 100 μm²)

950m

WD=25mm

Focus

M_A mirror

M_B mirror

30 nm

25 nm

< Wave-optically expected beam profile >
Focusing performance

![Graph showing focusing performance with 30nm and 25nm resolution](image-url)
Beam waist structures

Focal point

100μm

Measured profile

Calculated profile using measured shape

Intensity (arb. unit)

Relative position (nm)

-500 -250 0 250 500
Tunability of beam size and photon flux

Photon flux:

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

Installed in a new hutch of BL29

From Mono-chrometer

$M_A$ mirror

100mm

$M_B$ mirror

100mm

Focus

50 m

253 mm

150 mm

50.103 m

Slit size

10\(\mu\)m (Experimental)

50\(\mu\)m (Experimental)

100\(\mu\)m (Experimental)

10\(\mu\)m (Calculated)

50\(\mu\)m (Calculated)

100\(\mu\)m (Calculated)

Slit size

S. Matsuyama et al.
Demonstration of zooming performance

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

Se image
Probe X-ray: 15keV

SXFM 500nm/pix

Magnified Se image 35nm/pix

<100nm

4µm

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“Hard-X-ray sub-10nm focusing
By KB mirrors”
To realize Sub-10nm focusing K-B mirrors

- Slit (10μm)
- From undulator
- Focal point
- X-ray energy: 20keV
- Focal length: 150mm
- Acceptance width: 1.1mm
- Incidence angle: 11.1mrad

MIS with RADS and EEM can prepare the 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

Mirror surface
(Phase error originates in errors of surface profile and ML thickness)

Phase error compensation

A strategy to fabricate KB mirrors for 10 nm hard X-rays focusing,
K. Yamauchi, X-Ray Optics: A Roadmap for the Next 10 Years
A one-day (August 1, 2005) Workshop at SPIE's Optics & Photonics 2005 San Diego
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.

New knife-edge method

Conventional knife-edge method → New method

X-ray mirror → Focal point → X-ray detector

Spherically diffracted X-ray

Details of the new knife-edge method

Scanner is made of Pt with the thickness of 2.5 μm.

Scanner is acting as a phase object

Spherically diffracted X-ray

Modified wavefront

Beam waist

Phase is shifted with λ/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. (50μm upstream from the focal point)

On-line compensation of wavefront

In-situ phase compensation

Sub-10nm

Piezo-electric phase compensator

Focusing mirror with phase error
Phase error = $2kd \sin \theta$

- $d$: Shape error
- $\theta$: Glancing angle
- $k = \frac{2\pi}{\lambda}$: Wave number

An example
- Glancing angle of 10nm-level focusing mirror: $7 \text{ [mrad]}$
- Glancing angle of active mirror: $1.0 \text{ [mrad]}$

★ 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

Objective shape
- Elementary beam theory
- Controlled shape using optical interferometer

Feedback system

Optical interferometer

Measurement control

PC for Optical interferometer

Mirror profile

PC for active mirror control

Voltage supply

Active mirror
Optical configuration of 10nm-level focusing with AM

Compensator (AM: Active mirror)

Optical interferometer (ZYGO GPI)

Focusing mirror

Zooming tube + CCD

Glancing angle: 1 mrad

(Graded multilayer (Pt/C) coated, theoretical spot size: 12 nm)
Achieved beam size at Spring-8

FWHM: 15 nm

Focus spot size (nm)

Achieved beam size at Spring-8/Osaka Univ

Intensity vs. Position (μm)

Focus spot size vs. Year

15 nm
400mm-long mirror for XFEL


WD: 350mm

Measured profile
Ideal profile

75nm
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 0.1\(\lambda\)-level accuracy.

6. 10nm-level X-ray beams will be realized in the near future.
Acknowledgement

Co-workers


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**
\[ NA = n \cdot \sin \epsilon \]

**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
Geometrical approximation

Total reflection mirrors

\[ \sin \theta = \frac{b}{\sqrt{p \cdot q}} \]

\[ \sin \theta_c = \sqrt{2 \cdot \delta} \]
Geometrical approximation

Multilayer mirrors

Strong lateral + weak normal thickness gradient

\[ 2\varepsilon \]

\[ \sin \theta = \frac{b}{\sqrt{p \cdot q}} \]

\[ \Lambda = \frac{\lambda}{2\sqrt{n^2 - \cos^2 \theta}} \]
Basic considerations

Ellipse geometry

\[ 2\varepsilon = \phi_p (\theta_2) - \phi_q (\theta_1) \]

\[ \sin \phi_q = \frac{p \cdot \sin 2\theta}{2 \cdot c} \]

\[ NA = n \cdot \sin \varepsilon \]

Correct only for flat aperture!
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

\[ \sin \varepsilon = \frac{1}{4 \cdot c} \left( p_2 \cdot \sin 2\theta_2 - p_1 \cdot \sin 2\theta_1 \right) \]

\[ \sin 2\theta \approx 2 \cdot \sin \theta \approx \frac{\lambda}{\Lambda}, \; p \approx 2 \cdot c \]

\[ \Rightarrow \sin \varepsilon \approx \frac{\lambda}{2 \cdot \Lambda} (1/\Lambda_2 - 1/\Lambda_1) \]

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

\[ D_{FWHM} \approx 5 \text{ nm} \]

No explicit energy dependence!
Theoretical models

Geometrical phase shift due to height/slope errors

\[ \varphi_h = \frac{4\pi \cdot \sigma \cdot \sin \theta}{\lambda} \]

\( \sigma \): 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


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

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

**Osaka model**


E = 24550 eV
P = 150 m
Q = 0.077 m
δ = 2.7e-6
ρ = 5776 nm
s = 0.253 nm
**Theoretical models**

Chromaticity

\[ \delta \sim \frac{1}{E^2} \quad \Rightarrow \quad \left| \frac{df}{dE} \right| = 2 \cdot \frac{\Delta f}{E} \]

\[ D_{tot}^2 = D_{diff}^2 + D_{chrom}^2 \leq 2 \cdot D_{diff}^2 \]

<table>
<thead>
<tr>
<th></th>
<th>FZP</th>
<th>CRL</th>
<th>RML</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \frac{dE}{E} )</td>
<td>( \frac{D_{tot}^2}{1.76 \cdot f \cdot \lambda} )</td>
<td>( \frac{D_{tot}^2}{3.52 \cdot f \cdot \lambda} )</td>
<td>( \frac{D_{tot}^2}{3.52 \cdot \Delta f \cdot \lambda} )</td>
</tr>
</tbody>
</table>

**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!

Ch. Morawe – WS SPIE 13.08.08
Theoretical models

Alternative approach

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

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

Theoretical models

Results of numerical optimization


Focal spot blurring < 1 nm!

Single deep reflection

Multiple shallow reflections

10 layer pairs

Focal plane

X

Reflection position on mirror (mm)

Position of ray on focal plane (nm)

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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)

**Supervisors:**
- ESRF: Dr. Ch. Morawe (+33) (0)4 76 88 25 88
- Göttingen: Prof. Dr. T. Salditt (+49) (0)551 39 9427

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 Δρ, 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 ($\beta_{\text{spacer}}$)
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 ($\beta_{\text{abs}}$)
4. Make sure that both materials can form stable and sharp interfaces (lower $d$-spacing limit)

Computational search algorithms


Period number $N$:

Peak versus integrated reflectivity:

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

High and low resolution MLs

Optimize $N$ according to needs!
Multilayer properties

Filling factor $\Gamma = \frac{t_{abs}}{\Lambda}$

- Harmonics suppression
- Reflectivity enhancement

Optimum $\Gamma$ for large $N$


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

Best $\Gamma$ drops with growing $N$!
Multilayer properties

Non-periodic stacks:
- Ni/B₄C structure
- R(θ) = const over 20% bandwidth

Theoretical design

Experimental result

E = 8048 eV

Normalized Reflectivity

Relative Bandwidth dE/E [%]

ESRF Multilayer properties

Ch. Morawe – WS SPIE 13.08.08
Multilayer properties

Integrated reflectivity

\[ \Delta E/E \]

\[ R(\text{peak}) = 100\% \]

Single Crystals

Traditional ML's

High-resolution ML's

Depth-graded ML's

(Mirrors/Filters)

\[ E = 8 \text{ keV} \]

(forbidden area)
**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”**
Intensity profiles: (Kirkpatrick-Baez multilayer optics on ESRF BM05)

## Technological options

<table>
<thead>
<tr>
<th>Deposition techniques</th>
<th>Vacuum</th>
<th>Particle energy</th>
<th>Deposition rate</th>
<th>Deposition area</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal evaporation</td>
<td>HV (UHV)</td>
<td>Low</td>
<td>Low</td>
<td>Small</td>
</tr>
<tr>
<td>E-beam evaporation</td>
<td>UHV</td>
<td>Low</td>
<td>Low</td>
<td>Small</td>
</tr>
<tr>
<td>Magnetron sputtering</td>
<td>HV (+Gas)</td>
<td>High</td>
<td>High</td>
<td>Large</td>
</tr>
<tr>
<td>DECR sputtering</td>
<td>HV (+Gas)</td>
<td>High</td>
<td>Low</td>
<td>Medium</td>
</tr>
<tr>
<td>Ion beam sputtering</td>
<td>UHV (+Gas)</td>
<td>Very high</td>
<td>High</td>
<td>Medium</td>
</tr>
<tr>
<td>Pulsed laser deposition</td>
<td>HV</td>
<td>Very high</td>
<td>High</td>
<td>Medium</td>
</tr>
</tbody>
</table>

- 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

\[ t(\vec{r}) = \int \varphi(\vec{r}, \vec{r}') \frac{d\vec{r}'}{v(\vec{r}')} \]
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!
Example: $[\text{W/B}_4\text{C}]_{25}$ ML @ 24550 eV

$R_{\text{peak}} = 84\%$

$\Delta E/E = 8\%$
ESRF focusing experiment

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

ESRF BLs since 1998:
- 23 focusing devices
- 37 ML coatings

**ID19:** low $\beta$ @ 150 m, $E = 15...24$ keV
86*83 nm$^2$: $2 \times 10^{11}$ ph/s @ 80 mA

**ID22:** high $\beta$ @ 60 m, slit source, $E = 17$ keV
76*84 nm$^2$: $10^9$ ph/s @ 200 mA
150*100 nm$^2$: $10^{12}$ ph/s @ 200 mA

$3 \times 10^5$ ph/s/mA/nm$^2$

P. Cloetens, O. Hignette, ESRF
Summary

ML - KB “Data Sheet”

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

\[ \mu = \frac{4\pi\beta}{\lambda} \]

2 main contributions:

- photo absorption \( \tau \sim Z^3/E^3 \)
- Compton scattering \( \mu_c \)

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)

$R = 50 \, \mu m - 1000 \, \mu m$

$d = 10 \, \mu m - 30 \, \mu m$

$2R_0 = 450 \, \mu m - 1000 \, \mu m$

variable number of lenses: $N = 10 ... 300$

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

\[ NA = \frac{D_{eff}}{2L_1} \approx 10^{-4} - 10^{-3} \]
Full-Field Imaging

Ni-mesh (2000mesh)

For comparison:
spherical lens

(simulation)

imaging parameters:

\[ E = 12\text{keV} \]
\[ N = 91 \text{ (Be)} \]
\[ f = 495 \text{ mm} \]
\[ m = 10x \]

spatial resolution:

\( \sim 100 \text{ nm} \)
Full-Field Imaging: Spatial Resolution

- Expected resolution: 84 nm
- Deconvolve resolution of film
- Resolution of x-ray optical setup: 105 nm ± 30 nm

(SPIE 2002)
Hard X-Ray Microbeam

Focus size and shape determined by:
- source size
- magnification \( L_2/L_1 \)
- diffraction limit
- aberrations

\[ L_2 = \frac{L_1 f}{L_1 - f} \]

Flux in focus determined by:
- brilliance
- source size
- focusing cross-section of lens

on sample
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 $R = 50\mu m$: Microbeam

vertical focus

200nm

ESRF ID10

$E = 8$ keV
$N = 31$
$L_1 = 60.8$ m
$f = 156$ mm
gain $\sim 10^5$
flux: $3 \cdot 10^{11}$ph/s

mono: Si 111

expected focus size: 170 nm

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

focus source size and stability limited!
Rotationally Parabolic Refractive X-Ray Lenses

State-of-art (Be, Al):

\[ R = 50 \, \mu m \] [tested for focusing < 200 nm (source size limited)]
\[ R = 200 \, \mu m \] [tested for full field imaging with ~100 nm resolution]
\[ R = 300 \, \mu m, 500\mu m, \text{and} 1000\mu m \] [not tested, yet]

Lenses with \( R = 1500 \, \mu m \) 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

injection molded PE

APL 88, 164102 (2006)

Collab.: N. Stribeck, Univ. Hamburg
SAXS-Tomography

101 projections with
70 steps each
80µm step size

$I\tilde{q}(r)$

101 projections with
70 steps each
80µm step size

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SAXS-Tomography

Reconstruction:

- Attenuation
- Diffraction

\[ q_r = 0 \]

Integral scattering along rotation axis
SAXS-Tomography

Sample is fibre textured:

scattering cross section

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

interpretation:
Stribeck, et al.
Effective Aperture and Diffraction Limit

$D_{\text{eff}}$ limited by:

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

→ low Z lens material

Numerical aperture:

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

Diffraction limit:

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

large $f$: aperture dominated by attenuation

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

$\rightarrow$ reduce $\mu/\delta$ (low $Z$ lens material)

$\rightarrow$ $NA = D_{\text{eff}}/2f \propto 1/\sqrt{f}$: reduce focal size to minimum
Nanofocusing Lenses (NFLs)

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

Strong lens curvature:
$R = 1\mu m - 5\mu m$

$N = 35 - 140$

APL 82, 1485 (2003)
Crossed Nanofocusing Lenses

Setup at ID13 of ESRF
Focusing with NFLs

Si lens: E = 21keV, L₁ = 47m

source:
ID13 low-β invac. undulator

source size: 150 x 60µm²

vertical focus: 55nm

horizontal focus: 47nm

demagnification:
~ 2400 x 4400

flux: 1.7 ·10⁸ph/s

APL 87, 124103 (2005)
2D Scanning Mode: X-Ray Fluorescence

*Arabidopsis thaliana*

Fluorescence map

pollen

~ 100 nm resolution

$E = 15.25$ keV
High-Resolution Fluorescence Microtomography

Arabidopsis thaliana

tip of trichome (freeze dried)

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

trichome (leaf hair)
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 x 200 nm\(^2\)
flux: > 10\(^9\) ph/s

facet-rods

Map of diffuse scattering around Si(004)-reflection
Limits of Focusing with NFLs

Diffraction limit:

\[ d = \sqrt{\frac{2\delta}{\sqrt{\sum_{i} a_i}}}, \]

where \( d \) is the diffraction limit, \( \delta \) is the angular divergence, and \( a_i \) are the amplitudes of the harmonics.

Limits of Focusing with NFLs:

- \( N = 100 \)
- \( I \geq 0.084 \)
- \( R = 0.5 - 50\mu m \)

NA limited by

\[ \sqrt{2\delta} \]

adapt aperture to converging beam (AFLs):

focus < 5 nm

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

[PR 94, 054802 (2005)]
Nanoprobe: Coherent Nanodiffraction

\[ E = 15.25 \text{ keV} \]
\[ \lambda = 0.813 \text{ Å} \]

Beam size:
\[ < 150 \times 150 \text{ nm}^2 \] (amplitude)
Wave Front in Diffraction Limited Focus

divergence angle: numerical aperture

\[ d_t = \frac{2\sqrt{2\ln2}}{\pi} \frac{\lambda}{2NA} \approx 0.75 \frac{\lambda}{2NA} \]

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:
- tilted side wall
- deviation from parabola

Roughness:
- ~300 nm
- ~10 mrad
- ~1 µm
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 Si$_3$N$_4$-Membrane

size < 100 nm
Diffraction Pattern of Gold Nanoparticle

sample-detector distance: 1250 mm (in air)
detector: FReLoN 4K
50µm pixel size
exposure time: 10 x 60 s
intensity on sample: 3300 ph/s/nm²
integral dose in beam: $10^{11}$ ph
compared to $10^{12}$ ph/pulse at XFEL

> 2 month in flat coherent beam

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Reconstruction

reconstruction by HIO
shrink-wrap
phase left free to evolve
200 reconstructions
(9 converged to wrong solutions)

averaged solution:

resolutions:
left-handed right-handed

PRL (2008), accepted
Conclusion

Refractive x-ray lenses:

- high resolution imaging optics
- small numerical apertures (~ 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
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

www.elettra.trieste.it/organisation/experiments/laboratories/multilayer_technology/index.html

Read on CLESSIDRA kinoform lenses on pages 331-351
What happens here?

µXRF
multilayer

µXFA@ELETTRA
BL 10.1L

my hair 60 µm

Cu wire 100 µm

my hair 60 µm

Cu 30 µm

my hair 70 µm

3.7 keV

11.8 keV

transparent hairs can deviate (i.e. refract) x-rays
Refraction and reflection: x-rays

- Snell’s law: \( n \sin \alpha = \text{const} \)

- Beam deviation at one interface: \( \Delta = \phi_o - \phi_1 \)

- with \( \cos \Delta = 1, \sin \Delta = \Delta \): \( \cos \phi_1 = \cos \phi_o + \Delta \sin \phi_o \) then \( \Delta = \delta / \tan \phi_o \)
Refraction in transmitted x-rays

\[ n > 1.0: \text{convex} \]
\[ \text{lens focuses} \]

\[ n < 1.0: \text{concave} \]
\[ \text{lens focuses} \]

\[ \Delta = 2\delta / \tan \phi_o \]

We just heard it!
Refraction in transmitted x-rays

Back to hair and Cu wire
Refraction in transmitted x-rays

Equation for parabola
\[ 2R(x-x_o) = y^2 \]

focal length
\[ f = \frac{R}{2\delta} \]

Parabolic material increase
\[ \Delta = \frac{2\delta}{\tan \phi_o} \]

Gaussian transmission function
\[ T = \exp\left(-\frac{y^2}{2\delta f L}\right) \]

with \( L \) = attenuation length
Refraction in transmitted x-rays

\[ \Delta = 2\delta / \tan \phi_0 \]

\[ T = \exp\left(-\frac{y^2}{(2\delta fL)}\right) \]

Optimum aperture

\[ A = 2\sqrt{2f\delta L} \]

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)!

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 $2\pi$!

$D = m\lambda/\delta$

- $m$ = integer multiple
- $\lambda$ = wavelength
- $\delta$ = refractive index
Reduced material!!

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

These are kinoform lenses produced by lithography

D = \( m \lambda / \delta \approx 40 \, \mu m \)
For Si@12 keV

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!!

\[ D = \frac{m\lambda}{\delta} \approx 40 \, \mu m \]

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 100μm-deep silicon planar parabolic lens with minimized absorption. The lens aperture $A = 150\mu m$, the number of unit lenses $n = 5$, the maximum phase variation number $M = 2$, the focal length $F = 80\mu m$ at the design wavelength $\lambda_0 = 0.071\, nm$ ($\epsilon_0 = 17.48\, keV$).
Problem: small outer zones


Transmission: integrated ≤30%
i.e. ≤45% of expectation
Drops off when segment height < 2 µm

Nevertheless in single lens
smallest focus ≈320 nm fwhm
(Stein et al, JVST B26, 122 (2008))

private communication Evans-Lutterodt:
more recently <<320 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: CLESSI DRA

Jark et al, JSR 13, 239 (2006)

As before
$D \approx 40 \mu m$
for
SU8@8 keV
Si@12 keV
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 clessidra shape

Adjust tilt, yaw, roll for sharp shadow

SYRMEP beamline, 12 keV photon energy, CCD camera 9 μm pixel
Rapidly alignable x-ray optics

Radiography

SYRMEP beamline, 12 keV photon energy, CCD camera 9 µm pixel

You are done.

Now you can go to refractive focus position.
Rapidly alignable x-ray optics

10 m downstream from lens with $h=12.83 \, \mu m$

Gain in focus is 6x
Size is 110 \( \mu \)m (≈ hair)
Lens efficiency = 25%

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

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

SYRMEP beamline, 19.5 keV photon energy, CCD camera 9 \( \mu \)m pixel
Rapidly alignable x-ray optics

with better beam coherence and high resolution CCD

BM05(MOTB)@ESRF, 8 keV photon energy, CCD 0.65 µm pixel
Rapidly alignable x-ray optics

Gain=25 for 20 rows

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

\[ f = \frac{h^2}{m\lambda} \]

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

- \( h = 12.45 \mu\text{m} \) for crossed hairs

- \( m = 1 \) for material independence

- In resists (pmma or SU8) \( b = 36.7 \mu\text{m} \)

Needs lithography (we have one of few beamlines for deep x-ray lithography (DXRL) at ELETTRA)
Spatially coherently illuminated area:
\[ A = 0.44\lambda q/s \]

8 keV or \( \lambda = 0.155 \text{ nm} \)

- SYRMEP (\( q=23 \text{ m}, s=90 \mu\text{m} \)): \( A = 17 \mu\text{m} \)
- BM05 (\( q=53 \text{ m}, s=85 \mu\text{m} \)): \( A = 42 \mu\text{m} \)
- ID22 (\( q=40 \text{ m}, s=30 \mu\text{m} \)): \( A = 91 \mu\text{m} \)
- \( q=100 \text{ m}, s=23 \mu\text{m} \): \( A = 300 \mu\text{m} \)
Quality control: transmission

filling: 50%

pmma
h=25.7 µm
m=2, N=29

L_{exp} = 1.612 mm at 8.5 keV

T variation in last row
0.25 ---- 1.0

damping: CCD cross talk

expected average transmission
Quality control: refraction efficiency

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

Relative efficiency
>50%
out to border

Rounding in connected prism tips

average transmission from before
Optimising the wavelength

pmma, \( m=2 \)
\( h=25.7 \, \mu m \)

illuminating 1.0 mm centered: 40 rows

Lens with curved prisms

Peak width 4.0 \( \mu m \)

“Coherent” illumination

8.0 keV detuned

MOTB@ESRF (BM05-beamline)
CCD with 0.645 \( \mu m \) equivalent pixel
Optimising the wavelength

Illuminating 1.0 mm centered: 40 rows

Lens with curved prisms

Distance scan: full aperture 1.5 mm

Peak width 4.0 µm

“Coherent” illumination

8.0 keV detuned

MOTB@ESRF (BM05-beamline)

CCD with 0.645 µm equivalent pixel

PMMA, m=2
h=25.7 µm
Optimising the wavelength

illuminating 1.0 mm centered: 40 rows

pmma, m=2
h=25.7 µm

Distance scan: full aperture 1.5 mm

Lens with curved prisms

2.15 m
narrowest peaks

2.43 m

Peak width 4.0 µm

8.0 keV detuned

“Coherent” illumination

MOTB@ESRF (BM05-beamline)
CCD with 0.645 µm equivalent pixel

Refractive focus

simulation
Optimising the wavelength

pmma, m=2
h=25.7 µm

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 µm)
→ reduced spatial coherence
   (from 42 µm to 21 µm<h)

Peak width 7.3 µm

“Coherent” illumination

8.0 keV detuned

MOTB@ESRF (BM05-beamline)
CCD with 0.645 µm equivalent pixel
Optimising the wavelength

pmma, m=2
h=25.7 μm

illuminating 1.0 mm centered: 40 rows

Lens with curved prisms

“Coherent” illumination

7.9 keV better tuned

MOTB@ESRF (BM05-beamline)
CCD with 0.645 μm equivalent pixel

Peak width 6.6 μm
Optimising the wavelength

pmma, $m=2$
$h=25.7 \, \mu m$

illuminating 1.0 mm centered: 40 rows

Lens with curved prisms

“Coherent” illumination

Peak width 5.7 \, \mu m

7.7 keV best tune

MOTB@ESRF (BM05-beamline)
CCD with 0.645 \, \mu m equivalent pixel
Optimising the wavelength

pmma, \( m=2 \)
\( h=25.7 \, \mu m \)

illuminating 1.0 mm centered: 40 rows

Lens with perfect prisms

7.9 keV best tune

“Coherent” illumination

Peak width 6.5 \( \mu m \)

MOTB@ESRF (BM05-beamline)
CCD with 0.645 \( \mu m \) equivalent pixel
Optimising the wavelength

pmma, m=2
h=25.7 µm

illuminating 1.0 mm centered: 40 rows

Focus size is 12.5 µm = expected demagnified source image
But also h/2

Lens with perfect prisms

reduced spatial coherence (from 21 µm to 13 µm<h)

7.9 keV best tune

MOTB@ESRF (BM05-beamline)
CCD with 0.645 µm equivalent pixel
Refraction efficiency

vertical focusing: illuminating 65 µm (3 rows) at 270 µm off-axis

Lens with perfect prisms

Peak width 8 µm

270 µm

65 µm

“Coherent” illumination

7.9 keV best tune

MOTB@ESRF (BM05-beamline)

CCD with 0.645 µm equivalent pixel
Refraction efficiency

vertical focusing: illuminating 25 µm (1 row) at 270 µm off-axis

“Coherent” illumination

Lens with perfect prisms

270 µm

25 µm

Diffraction at border between two rows

270 µm

Refraction in single row with 10 prisms

MOTB@ESRF (BM05-beamline)

CCD with 0.645 µm equivalent pixel

7.9 keV best tune

7.9 keV best tune

µXRF multilayer
Refraction efficiency

vertical focusing: illuminating 25 µm (1 row) at 270 µm off-axis

Near field or Fresnel diffraction: a 25 µm slit focuses!

Peak width 14 µm

Peak width 12 µm

“Coherent” illumination

7.9 keV best tune

MOTB@ESRF (BM05-beamline)
CCD with 0.645 µm equivalent pixel
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)

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 $h=12.83\ \mu m$

Aperture is $140*h$
Etching depth is only $25*h$

1.8 mm
Conservative outlook

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

Stack $M = 2$ or Cederstroem prism array

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

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

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

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

image could be 110 nm
More ambitious outlook

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

Stack $M = 2$ or Cederstroem prism array

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

Spatial resolution limit $r = 0.88 \times \lambda \times f / A$  
$r = 53 \ nm$!

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

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

image could be 27 nm
Fresnel lens outlook


- Arrived already at A=0.3 mm, f=0.1 m
  - Image size \(<<320\) nm fwhm
- Stein et al, JVST B26, 122 (2008), priv. comm. Evans-Lutterodt
- Only center segment is >6 \(\mu\)m!
- In Si better shape fidelity at smaller dimensions than in photoresist!
- In turn etch depth limitation for RIE at 0.1 mm?

Relative efficiency \(\approx 25\%\)
- For segments \(\approx 1.7\) \(\mu\)m
- And <10\% for \(\approx 0.7\) \(\mu\)m
Follow adiabatically shrinking beam size

In 1166 lenses

Lens thickness: 37.8 µm ... 0.100 µm
Lens aperture: 42.2 µm ... 0.224 µm
Outer segment: 4.5 µm ... 0.024 µm

focus r=2.21 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
Outline

Structure of Polycapillaries

- Physics of Reflection, bending limits
- Divergence
- Alignment and Characterization
  - Source Angle
  - Transmission vs Energy
  - Spot Size

- Gain and Liouville's Theorem
- Defect Analysis
- Applications
  - MicroXRF
  - XRD
  - Astronomy
  - Therapy
Polycapillary Optics

- COLLIMATING
- 50 mm x 50 mm output
- 250 mm input focal length
- 12 µm channel

- FOCUSING
- 0.5 mm

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

\[
\begin{align*}
\nabla \cdot \vec{E} &= 0 \\
\nabla \cdot \vec{B} &= 0 \\
\n\nabla \times \vec{E} &= -\frac{\partial \vec{B}}{\partial t} \\
\n\nabla \times \vec{B} &= \mu_0 \varepsilon \frac{\partial \vec{E}}{\partial t}
\end{align*}
\]

\[
\Rightarrow \frac{\partial^2 \vec{E}}{\partial t^2} = \frac{1}{\mu_0 \varepsilon} \nabla^2 \vec{E}
\]

\[
v = \frac{1}{\sqrt{\mu_0 \varepsilon}} = \frac{1}{\sqrt{\mu_0 \varepsilon_0 \varepsilon}} = \frac{c}{n}
\]

\[
\Rightarrow n, \text{index of refraction} = \sqrt{\frac{\varepsilon}{\varepsilon_0}}
\]

So, given an incident plane wave of the form:

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

we expect a response

\[
\vec{E} = \vec{E}_0 \varepsilon \vec{E} + \vec{P}
\]

hence

\[
\vec{E} = \vec{E}_0 \varepsilon \vec{E} + \vec{P} \Rightarrow \varepsilon = \varepsilon_0 + \frac{\vec{P}}{\vec{E}}
\]

\[
\text{needed to model the response, } \vec{P} \text{ of the material to the EM wave}
\]
Free Electrons in one dimension:

\[ F = m\ddot{x} = qE = qE_o \cos \omega t \]

\[ x = x_o \cos \omega t \]

\[ \Rightarrow -m\omega^2 x_o = qE_o \quad \Rightarrow x_o = -\frac{qE_o}{m\omega^2} \]

\[ P_o = Nqx_o = -\frac{Nq^2}{m\omega^2} E_o \]

\[ \therefore \epsilon = \epsilon_o + \frac{-2}{m\omega^2} = \epsilon_o \left(1 - \frac{m\epsilon_o}{\omega^2}\right) \]

\[ \epsilon = \epsilon_o \left(1 - \frac{\omega_p^2}{\omega^2}\right) \]

\[ P = x \]

number of electrons per volume

current on an electron

displacement of the electron due to the electric field
Index of Refraction

\[ n = \sqrt{\frac{\varepsilon}{\varepsilon_0}} = \sqrt{1 - \frac{\omega_p^2}{\omega^2}} \approx 1 - \frac{1}{2} \frac{\omega_p^2}{\omega^2} = 1 - \delta \]

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

Consequences for normal incidence mirror:

Total External Reflection

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

\[ n_1 \sin (90 - \theta_1) = n_2 \sin (90 - \theta_2) \]

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

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

\[ \theta_c = \frac{\omega_p}{\omega} \approx 3 \times 10^{-3} \quad R = 1 \]
Damping

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

\[ x = x_0 \cos(\omega t + \phi) \]

\[ n = 1 - \delta - i\beta \quad R < 1 \]

Reflectivity vs Angle (mRad)

Reflectivity vs Angle (mRad)

Ener g

- 20 eV
- 40 eV
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

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

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

$f = 50 \text{ mm}, \quad r = 5 \text{ mm} \implies \phi = 11.5^\circ \quad L = 100 \text{ mm} \implies R \approx 1 \text{ m}$
Cutoff Energy dependence on bend radius and channel size

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

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

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

Source

Detector

Focusing Lens

58 mm input focal length

119 mm output focal length
Beam Filtering

Normalized Intensity vs Photon Energy (keV)

- No Optic
- With Stage II Optic + 8 µm Ni filter
Characterization of Polycapillary Optics

Capture Angle, \( \omega \)
\( 3^\circ \)

\( \Omega \) 2 millisteradian

\( F_{\text{in}} = 62 \text{ mm} \)

\( F_{\text{out}} = 47 \text{ mm} \)

Capture Angle,
\( \omega = 3^\circ \)
\( \Omega = 2 \text{ milli-radian} \)

Input focal length
Transmission = 9.9%

\( \epsilon = \frac{\Delta y}{Z} \)

\( F_{\text{in}} = 62 \text{ mm} \)

\( F_{\text{out}} = 47 \text{ mm} \)
Focusing Lens Output Spot Measurement

Knife Edge Measurement

Output spot 0.039 mm

Counts

Spot Size (mm)

Distance from Lens Output (mm)

Knife Edge Displacement

Spot Size (mm)

Distance from Lens Output (mm)
Focusing Optic Output

Source spot

Focal spot size = 0.8 mm

Image plate

Global Divergence =

slope = \(2\beta = 0.55^\circ\)
FOCAL SPOT SIZE

Minimum due to Local Divergence $2\alpha$

Minimum size:

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

$c = 5 \, \mu m \quad 2\alpha = 0.28^\circ$

$$\begin{cases} f = 50 \, mm & \Rightarrow d = 250 \, \mu m \\ f = 5 \, mm & \Rightarrow d = 30 \, \mu m \end{cases}$$
Double Gaussian Peaks of Width 0.28°
Global divergence

Rocking Curve focusing optic

Rocking Curve Width = 0.83° = 2α + 2β = 0.28° + 0.55°
To make the optic look good: compare to flux through a pinhole of diameter < spot size compared to source at focal point:

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

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

\[d = 200 \, \mu m \quad r_{\text{Lens}} = 5 \, mm \quad f = 50 \, mm \quad L = 100 \, mm \Rightarrow \quad \text{Gain} = 8000\]
Liouville’s Theorem

Angle area product cannot decrease

\[ A_f \Omega_f \geq A_o \Omega_o \]
Optics Defects

Cut through

profile from image at focal point
Gaussian fit

intensity

location of the image plate in mm
Optic defects

Bending:

\[ \lambda_b = 10 \text{ cm} \]

parameter: bending radius, \( R \)

Waviness:

\[ \lambda_r < \lambda < \lambda_b \]

parameter:

Modeled by random angle shifts of \( \delta \theta \) after each bounce
**Optic defects**

\[ \lambda_r = 1 \ \mu m \]

**Roughness:**

- **correlation length:**
- **rms height:**

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

10 keV

- data
- 0
- 0.5 nm
- 1 nm

70 keV

- data
- 0
- 1 nm
- 2 nm
Simulation Analysis: Single Fiber

Simulation with $w=0.15 \text{ mrad}$ and $R=120 \text{ m}$.

Experiment
Simulation
Transmission

<table>
<thead>
<tr>
<th>Energy, keV</th>
<th>30</th>
<th>50</th>
<th>70</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source position (mm)</td>
<td>-2</td>
<td>0</td>
<td>2</td>
</tr>
</tbody>
</table>

Normalized transmission

-2 0 2

20keV
40keV
59keV
80keV

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Simulation Analysis: Lens made from fibers

![Simulation Analysis: Lens made from fibers](image)

- **Transmission (%)**
- **Source optic distance (mm)**

**Experiment**
- **Ideal optic**
- **Non-ideal optic**

**Simulation Analysis:** Lens made from fibers

- **Energy (keV)**
- **Transmission**
- **Source optic distance (mm)**

- **Experiment**
- **Ideal Optic**
- **Non-ideal Optic**

- **w = 0.15 mrad**
- **w = 0.15, rh = 0.5 nm**
Optics Defect: Channel Blockage

- 39 mm input focal length
- 7.8 mm output focal length
- 30 µm glass in length of lens

Transmission (%)

Energy (KeV)

Detector

Focusing Lens

Source

39 mm input focal length
7.8 mm output focal length
30 µm glass in length of lens
Application to Radiation Damage

- Data, UNEXPOSED
  - R = 110 m, \( \Delta \theta = 0.285 \text{ mrad} \), 0.8 nm

- Data, EXPOSED to 1.8 MJ/cm² white beam
  - R = 90 m, \( \Delta \theta = 0.450 \text{ mrad} \), 0.8 nm

Transmission vs. Photon energy graph.
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 Kα, FWHM, 17.4keV): 10um 18um 30um 45um 100um 180um

Gain (Compared to pinhole aperture 100 mm from source): 100x - 10000x
Spatial Resolution of MXRF with 39 $\mu$m spot

Micrograph of Cu grid

254 $\mu$m 49 $\mu$m

Cu-Grid Scan Along Y-Axis

Counts

Displacement (mm)

FWHM

M

49 $\mu$m

Applications: XRF
Elemental Mapping

MXRF maps of a quartz phenocryst with small volcanic glass inclusions

Courteously of Ning Gao, XOS
Applications: Protein Crystallography

Lysozyme pattern taken in 20 seconds with 2.8 kW rotating anode, comparable to 30-35 min. without optic.

Linear R factor without optic 6.4%
with optic on same sample: 6.9%
Crystall size: Less than 200 µm

Oscillation angle: 1.5 deg (44 frames)

Time / frame: 60 min

PIN diode intensity: $3 \times 10^{-4}$

Resolution: 2.0 Å

R-factor: 5.2%
Applications: Protein Crystallography

Reciprocal lattice

Vector, $\mathbf{G}$

Origin of Reciprocal lattice

Detector

Lysozyme Data

Simulation

5 min

1 mA

2.1°
Applications: Powder Diffraction

Ni filter
24 ìm

source

optic

aperture
Ö=3.8mm

beam stop

sample

beam stop

f_{in}

x

L
Applications: Powder Diffraction

Optic Results | Relative diffracted beam intensity | Measurements:
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>i sample at focus, plate at 66-75 mm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Peak width</td>
<td>Average Peak error</td>
</tr>
<tr>
<td>one</td>
<td>1</td>
<td>0.7</td>
</tr>
<tr>
<td>f 47</td>
<td>5</td>
<td>1.1</td>
</tr>
<tr>
<td>f 119</td>
<td>113</td>
<td>1.9</td>
</tr>
</tbody>
</table>

Ni sample at focus, plate at 66-75 mm
Applications: $\mu$-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

- Measured using image plate
- Measured using knife edge

- Applications: Orthovoltage Therapy
Applications: Synchrotron Focusing and Astronomy

Table 1. Results for monolithic focusing optic.

<table>
<thead>
<tr>
<th>X-Ray Energy (eV)</th>
<th>Spot size (mm)</th>
<th>transmission (%)</th>
<th>measured Gain 350 µm pinhole</th>
<th>calculated Gain 350 µm pinhole</th>
<th>calculated Gain 90 µm pinhole</th>
<th>calculated Gain 10 µm pinhole</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>0.09</td>
<td>36</td>
<td>78</td>
<td>81</td>
<td>645</td>
<td>911</td>
</tr>
<tr>
<td>8</td>
<td>0.08</td>
<td>49</td>
<td>96</td>
<td>110</td>
<td>933</td>
<td>1359</td>
</tr>
<tr>
<td>10</td>
<td>0.09</td>
<td>39</td>
<td>83</td>
<td>87</td>
<td>624</td>
<td>842</td>
</tr>
<tr>
<td>12</td>
<td>0.09</td>
<td>39</td>
<td>74</td>
<td>87</td>
<td>654</td>
<td>903</td>
</tr>
<tr>
<td>white</td>
<td>0.17</td>
<td>42</td>
<td>11</td>
<td>89</td>
<td>243</td>
<td>266</td>
</tr>
</tbody>
</table>

Applications: Synchrotron Focusing and Astronomy
References


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FOCUSING OF X-RAYS USING CRYSTAL OPTICS

Eckhart Förster

Friedrich-Schiller-University Jena,
Institute for Optics and Quantum Electronics,
X-Ray Optics Group
07743 Jena, Germany

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
- \( d_{hkl} \): distance between the reflecting planes
- \( \theta_{hkl} \): Bragg angle
- \( \vec{g} \): normal of reflecting planes, reciprocal lattice vector
- \( hkl \): indices of reflecting planes
- \( n \): 1, 2, ..., diffraction order
flat crystal, symmetrical reflection

\[ n \lambda = 2d \sin \theta \]

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

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

X-ray imaging is best, if $\theta$ is maximal.

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

focal lengths: \( f_h = \frac{R_h}{2} \cdot \sin \theta_B \)

\( f_v = \frac{R_v}{2 \cdot \sin \theta_B} \)

Point to point focussing \( \frac{R_v}{R_h} = \sin^2 \theta \)

vertical bending radius

horizontal bending radius

Rowland circle

laser produced X-ray source

toroidally bent crystal

sample crystal
Fabrication and Test of Toroidally Bent Crystals

grinding and polishing of toroidal glass formers

crystal mounting
tangential error < 1"

epoxy glue

crystal

glass former

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

X-ray topography $\rightarrow$ 'perfect' crystal block

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

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

optical and X-Ray imaging tests,

Relation of the Curvature Radii: $R_v/R_h = \sin^2 \theta$
Test of Bent Crystals for Monochromatic Imaging

tilt $\alpha$ between the normal vector of the surface and the lattice planes.

alternative sources

100 $\mu$m grid as object

visible light source X-ray tube

toroidally bent crystal

$R_v$ - horizontal bending radius

$R_h$ - vertical bending radius

shadowgraphy of a needle in front of the film

$X$-ray image

image plane

visible light image
Fabrication and Test of Toroidally Bent Crystals

1.1 Crystal mounting

1.2 X-ray topography → 'perfect' crystal block

2.1 Tangential error < 1"

2.2 Oriented sawing (accuracy: 10′ to 10″), grinding, polishing → 70 μm thick discs

3. Optical contact with glass form sticking to crystal mounting with epoxy glue

4. Optical and X-ray imaging tests,

5. Determination of the reflectivity (X-ray tube or synchrotron),

Relation of the Curvature Radii: \( \frac{R_v}{R_h} = \sin^2 \theta \)
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

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

Dependency of the Reflection Power of the Curvature Radius

Fabrication and Test of Toroidally Bent Crystals

grinding and polishing of toroidal glass formers

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

crystal mounting tangential error < 1”

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),

use in X-Ray spectroscopy, diffractometry and X-Ray imaging

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

cylindrically bent mica
$R = 100$ mm
$50$ mm x $60$ mm

toroidally bent GaAs $400$
$R_h = 200$ mm, $R_v = 189.4$ mm

toroidally bent quartz $10.-1$
$R_h = 500$ mm, $R_v = 400$ mm
High power laser: $E_L > 10^6 J$, $\tau_L < 1 \text{ns}$, $\lambda_L < 0.5 \mu m$

main aim: suppression of Rayleigh-Taylor instabilities

- ablation front
- interface compressor/
  D, T fuel
- laser pusher
  few % Argon
  or Krypton
- e.g. chlorine

$I_x(\lambda, x, y, z, t, \ldots)$

- X-ray spectrograph
- X-ray imaging device

streak or
framing camera
X-Ray Monochromatic Camera Using Two Toroidal Crystals

- Aperture Ly8
- C
- SOpm
- 4.11614.6
- IIn.(lst1.3p)
- He13
- Intensity ratio
- $h\nu = 3.68$ keV
- a hot core plasma with tiny Ar
- $h\nu = 3.93$ keV
- Electron Temperature (key)

CRE model
Ten Channel Imaging System

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 $\beta$ increases as $\alpha$ decreases, while the sine condition demands, that $\sin \alpha / \sin \beta$ remain constant (upper diagr.). Approximate constancy of the sine ratio can be achieved through the use of two mirrors (lower diagr.), so that $\beta$ increases as $\alpha$ increases.

James Underwood, American Scientist 66 (1978), 476 - 486
Scheme of monochromatic imaging using a single-bent-crystal optic and two-bent-crystal optics. The compact design of the two-bent-crystal system allows an installation of optic and detector onto a single port of the experimental chamber.

<table>
<thead>
<tr>
<th></th>
<th>one crystal</th>
<th>two crystals</th>
</tr>
</thead>
<tbody>
<tr>
<td>spatial resolution</td>
<td>14 µm</td>
<td>1.6 µm</td>
</tr>
<tr>
<td>spectral window ($\lambda/\Delta \lambda$)</td>
<td>235</td>
<td>6069</td>
</tr>
<tr>
<td>relative luminosity</td>
<td>1</td>
<td>1/15</td>
</tr>
</tbody>
</table>

Study of Ultrafast Processes in Crystalline Matter

Setup of an Optical Pump X-Ray Probe Experiment

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


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).

M. Bargheer et al., Appl. Phys. B 80 (2005), 715
### Characteristic Parameters of Cu Kα Optics

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

*red – best value
pink – second best value*

M. Bargheer et al., Appl. Phys. B 80 (2005), 715
Ellipsoidal HOP Graphite Crystal Reflection Curve

- Quartz 10-1-1
- Graphite 002

- Thin foil < 10 µm
- Thick foil 100 µm

FWHM ~ 0.11°
Ellipsoidal HOP Graphite Crystal X-Ray Focus Test

1. Amptek spectrometer
2. X-ray film

X-ray detector

FWHM: 490 µm

I. Uschmann et al., Appl. Optics 44, (2005), 5069
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: $10^{-5}$ sr – $10^{-3}$ sr

- Focal size: 1 µm – 5 µm @ large $\theta$ 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
Multilayer Laue Lens for Efficient Nanometer Focusing of Hard X-rays

G.B. Stephenson¹,², H.C. Kang²,⁴, H. Yan¹,⁵, R.P. Winarski¹, M.V. Holt¹, J. Maser¹,³, C. Liu³, R. Conley³,⁵, S. Vogt³, and A.T. Macrander³

¹Center for Nanoscale Materials,
²Materials Science Division, and
³X-ray Science Division, Argonne National Laboratory

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

⁵National Synchrotron Light Source II, Brookhaven National Laboratory

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**

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

--> Need larger Numerical Aperture (NA)

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?

---

**Rayleigh criterion** : Best Possible Resolution = \[
\frac{\lambda}{2 \text{ NA}}
\]

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

Currently, NA ~ 10^{-3}, resolution ~ 500 \lambda
**High NA Hard X-ray Focusing Optics**

- **Refractive: Lenses**
  - Compound refractive lens

- **Reflective: Mirrors**
  - Figure by differential deposition, multilayer coated for high NA
  - Lithographic zone plate
  - Transmission multilayer

- **Diffractive: Zone Plates**
  - High aspect ratio, tilted zones for high NA
  - 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 (>>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

- 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 ~ 200 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)

Multilayer Laue Lens on flat Si substrate
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:
- C. Shroer - Parabolic wave equation (Phys. Rev. B 74, 033405 (2006))
**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.
**Multilayer Structure for MLL**

WSi$_2$/Si, 1588 layers, $t_{\text{dep}} = 13.25$ μm

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

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

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

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

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

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

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

- 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

Advanced Photon Source, ANL
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
**Nanoprobe Beamline Schematic**

**Scanning Probe Mode**

- Sample
- Area detector: transmission
- Energy dispersive detector: X-ray fluorescence

**Area detector:** microdiffraction

- Crystal monochromator
- Double mirror system
- Beam defining Aperture (closed)
- Two collinear undulators

- Hard X-ray zone plate (focusing)
- Condenser system
- Crystal Monochromator
- Phase ring

**Full-Field Transmission Mode**

- Sample
- Area detector: transmission
- Energy dispersive detector: X-ray fluorescence

- Beam defining Aperture (open)
- Hard X-ray zone plate (imaging)
- Rotation for tomography
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
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)

SOI features stressors

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
Diffractive Focusing By Zone Plates

3D X-ray Imaging for Science and Industry

Dr. Michael Feser
Vice President / General Manager
nano-Imaging
Xradia Inc.
### High-resolution Optics: Comparison

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

(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
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
  - $\theta =$ diffraction angle

- Constant grating pitch acts like a prism for one diffraction order (deflects light)
- Deflection is a linear function of wavelength $\lambda$
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
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 \( \Delta R \):

\[ \text{Res} = 1.22 \Delta R \]
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
Scanning Electron Micrographs of Zone Plates

- Grating bar width: 100nm (Resolution)
- Grating height: 1600nm (Efficiency)

- Resolution limited to approximately the zone width $\Delta R$
- 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
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)

- Janos Kirz (Stony Brook)

- E-beam zone plates:
  - Proposed by D. Sayre, IBM tech report RC 3974 (1972).
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 manly 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.
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

Poly-chromatic source output

W-L_α (8.4 keV)
W-L_β (9.6 keV)

Differential Filter

W-L_α (8.4 keV)
Quasi-monochromatic

8/13/2008
SPIE X-ray Focusing Workshop
Quantitative Efficiency Measurements of Zone Plates Using Laboratory Sources

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

Efficiency = \( \frac{T - M_Z A_F}{M_B A_Z} = 8.7\% \)

- 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


| \( M_B \) | \( M_Z \) | \( A_Z \) | \( T \) | \( A_F \) |
| Bkgd intensity (counts/pixel) | Pedestal intensity (counts/pixel) | ZP area (pixels) | focused intensity (counts) | 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

- 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.

If lines are farther apart, proximity effect is reduced.

Process every other line, then go back and do the ones you missed (Chao et al., CXRO)
Heroic efforts at Lawrence Berkeley Lab

  - Efficiency ~3%.
  - Focal length if used at 290 eV edge: 100 μm.

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

![Diagram of the tri-level processing scheme](image-url)
Tri-Level Processing Scheme

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1. E-beam expose, develop
   - E-beam resist
   - Hard mask
   - Plating mold
   - Plating base
   - Window
   - Si frame

2. Etch hard mask
Tri-Level Processing Scheme

- Write high resolution pattern in top layer.
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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

   - Si frame
   - E-beam resist
   - Hard mask
   - Plating mold
   - Plating base
   - Window

2. Etch hard mask

3. Etch plating mold; strip hard mask

4. Metal plating
Zone Plates: Stony Brook

- 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

- 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)

Cross section view of control structure

Single ZP:
24nm zones
133um dia
300nm height
Two zone plates are aligned and **permanently** bonded together face-to-face. (Patented process)

Two zone plates act effectively as one diffractive element if aligned precisely laterally and in very close proximity (within depth of focus of lens)

- 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.

Y. Feng, et al., *Journal of vacuum science and technology B*, 25 (6), 2008
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
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
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
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

Diagram:

- Template
- Release layer
- Transfer layer
- Etch layer
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
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.

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

MLLs are not without challenges

Must stay on-Bragg for good efficiency
High resolution: lots of layers!

- Transverse resolution $\Delta t = 1.22 \Delta r_N$, where $\Delta r_N =$ outermost zone width
- Diameter $d = 1.22 \frac{\lambda f}{\Delta t}$, # zones $N = 1.22^2 \frac{\lambda f}{\Delta t^2}$
Challenge at high resolution:

Depth of focus

Depth of focus is $1.22 \lambda / \text{N.A.}$, or $4.88 (d r_N)^2 / \lambda$ where $d r_N$ is the outermost zone width.
How clean is the focus?

- $a = \frac{\text{central stop diameter}}{\text{zone plate diameter}}$

You really want all zones!
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!

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

---

![TXM: transmission x-ray microscope](image-url1)

![STXM: scanning transmission x-ray microscope](image-url2)
X-Ray Advantage: 
High-resolution imaging of buried Structures

Dielectric
Copper Lines and Vias
Computed Tomography

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

Sample rotation angle

Sinogram

Line Image

(2) 3D reconstruction by backprojection results in 3D image of the sample.
Loss of contrast and throughput leads to a crossover at ~1um resolution.
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
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
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

Source: Prof Wilson Chiu, University of Connecticut
Putting It All Together

nXCT-Imaged SOFC Anode

Gas Transport, Reformation & Electrochemistry

3D Reconstruction

User Models

Optimum Structure

Parameters

\( \varepsilon, \tau, <r>, <r^2>, D_{ij}, D_{ij}^k, \mu, k \)

\( \eta_{\text{ohm}}, \eta_{\text{conc}}, \rho \)

\([i_0, \beta, \eta_{\text{act}}, L_{TPB}]\)

Ionic & Electronic Charge Transfer

Source: Prof Wilson Chiu, University of Connecticut
M1 Metal Layer IC Sample

Magnified Image

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

W-plugs
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

1μm
CT Reconstruction – Planar Slices

- 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 μm

M1
M2
M3
M4
M5
M6
M7

8/13/2008 SPIE X-ray Focusing Workshop
Technologically Relevant Application:

TSV - Through-Silicon Vias (10um Diameter)

10 μm vias tilted at 45 degree.
Tile of 3x3 images 66x66 μm each

All vias have missing electroplating (key hole) in center

50 μm
Large field of view mode
2 minute exposure per tile
nanoXCT allows direct visualization of the key hole without physical cross-sectioning.
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

Zone Plate
Stage
Sample Stage
Zone Plate
Sample
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
Application Example Diffraction: -
Quantum dot stressors on Si Nanomembranes

30 nm Si Ge hut QD

Ge-Si Lattice Lattice Mismatch
Distorts Si Membrane


Microdiffraction at
APS Sector 2
hv=11.2 keV
200 nm spot

membrane Si (004) reflection with Ge huts
without Ge

University of Wisconsin MRSEC IRG1
<table>
<thead>
<tr>
<th>NSRRC (Taiwan)</th>
<th>SSRL BL-6-2 (Stanford, USA)</th>
<th>APS ID-32, ID-26 Argonne, USA</th>
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<td>NSRRC</td>
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Thank you for your attention!