Mirrors and Multilayers

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outline

Mirrors
(1) role of mirrors
(2) refractive index and reflection of X-rays
(3) mirror materials and mirror coatings
(4) surface polishing and characterization
(5) curved mirrors
(6) transmission mirror
(7) heat load and cooling

Multilayers
(1) structure and X-ray reflectivity
(2) fabrication
(3) characterization
(4) graded multilayers
(5) applications
Mirrors

- Deflecting the beam
- Low pass filter
- Collimation
- Focusing
Mirror position on a beamline

- Light source
- Storage ring
- Optics hutch
- Experimental hutch
- Concrete shield
- Front-end
- Optics and transport channel
- Experimental hall
- Undulator
- Bending magnet & front-end
- Double-crystal monochromator
Refractive index

For X-rays, refractive index is slightly smaller than unity.

\[ n = 1 - \delta + i\beta \quad \text{for} \quad E \propto \exp(+i(\omega t - kr)) \]

\[ (n = 1 - \delta - i\beta \quad \text{for} \quad E \propto \exp(-i(\omega t - kr)) ) \]

\[ \delta = \frac{n a r_e \lambda^2}{2 \pi} f_1 \quad \beta = \frac{n a r_e \lambda^2}{2 \pi} f_2 \]

\[ f_1 = f_0 + f' \quad f_2 = f'' \]

\[ f_0 : \text{atomic scattering factor} \]

\[ f' \text{ and } f'' : \text{real and imaginary parts of anomalous dispersion term} \]

\[ f = \int \rho \exp(ikr) \]

For forward scattering, \( f_0 = Z \)

Refer to Chap. 3 of “soft X-rays and extreme ultraviolet radiation” by D. Attwood, Cambridge Univ. Press, 1999

<table>
<thead>
<tr>
<th>Material</th>
<th>( \delta \times 10^5 )</th>
<th>( \beta \times 10^7 )</th>
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</thead>
<tbody>
<tr>
<td>Si</td>
<td>0.488</td>
<td>0.744</td>
</tr>
<tr>
<td>Ge</td>
<td>0.908</td>
<td>2.33</td>
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<tr>
<td>Quartz</td>
<td>0.555</td>
<td>0.467</td>
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<tr>
<td>Pt</td>
<td>3.26</td>
<td>20.7</td>
</tr>
<tr>
<td>Au</td>
<td>2.96</td>
<td>19.5</td>
</tr>
</tbody>
</table>
**Experimental data**

Asymptotic behaviour for $E > E(K)$:

\[ f_1 \approx \text{const} \quad f_2 \approx \frac{1}{E^2} \]

\[ \delta \approx \frac{1}{E^2} \quad \beta \approx \frac{1}{E^4} \]

\[ \frac{\delta}{\beta} \approx E^2 \]

High energy applications!
Snell’s law

\[ \frac{n_1}{n_2} = \frac{\cos(\theta_2)}{\cos(\theta_1)} \]

if \( \frac{n_1}{n_2} \cos(\theta_1) = 1 \rightarrow \cos(\theta_2) = 1 \rightarrow \theta_2 = 0 \)

\[ \theta_1 = \frac{\theta_1}{2} \]

\[ n_1 = 1 \text{ in vacuum} \rightarrow \cos(\theta_1) = 1 - \frac{\theta_1^2}{2} = n_2 = 1 - \delta \]

\[ \theta_c = \sqrt{2\delta} \approx 1 \sim 20 \text{ mrad} \]

For 10 keV

<table>
<thead>
<tr>
<th>Material</th>
<th>( \delta \times 10^5 )</th>
<th>( \theta_c ) (degree)</th>
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<tr>
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<tr>
<td>Au</td>
<td>2.96</td>
<td>0.441</td>
</tr>
</tbody>
</table>

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

\[ R = \frac{(\theta - A)^2 + B^2}{(\theta + A)^2 + B^2} \]

\[ A = \sqrt{\frac{(a^2 + b^2)^{1/2} + a}{2}} \]

\[ B = \sqrt{\frac{(a^2 + b^2)^{1/2} - a}{2}} \]

\[ a = \theta^2 - \theta_c^2 = \theta^2 - 2\delta \quad b = 2 \beta \]

\[ q = 4\pi \frac{\sin(\theta)}{\lambda} \]

For \( q \gg q_c \)

\[ R \propto \frac{1}{q^4} \]


CXRO_ALS

“X-ray Interactions with Matter”
http://henke.lbl.gov/optical_constants/
Reflectivity curve

A total reflection mirror works as a low-pass energy filter. The cut-off energy can be controlled by selecting the material and the glancing angle.

Influence of absorption edges sometimes appear in the reflectivity curve.

From Uruga & Nomura, Hoshako (放射光) 2006 vol.19 No.4 pp.248-257, Fig.3
Requisites for mirror materials

(1) Can be polished to a smooth and accurate surface figure.
(2) Large material available. (~1.4 m long mirror presently used)
(3) Mechanically hard, Radiation resistive
(4) Good thermal conductivity, low thermal dilation constant
(5) No (low) gas emission under X-ray irradiation
(6) Can be bent.
(7) Reasonable cost (material itself and fabrication)
Important parameters

• Surface figure
  flat, cylindrical, elliptic, parabolic, spherical, toroidal, ellipsoidal, paraboloidal,----
• Roughness
  root-mean-square roughness ~0.1 nm
• Slope error
  root-mean-square slope ~0.5 μrad
• Thermal property
  thermal conductivity
dilation constant

• Absorption edges : X-ray absorption
Materials for mirrors

- Silicon
- Zerodur
- Fused silica
- GlidCop (alumina dispersion strengthened copper alloys)
- Cu with electroless Ni layer
- Silicon Carbide (CVD)

Materials for mirror coating

- Desired critical energy with a given glancing angle
- Retain smooth surface
- No absorption edges desirable in the required energy range

Au, Pt, Rh, Ni, Pd, Ru

\[ \theta = 3.3 \text{ mrad} \]

\[ 100 \text{ nm coating} \]
Coating of mirrors
(Rh coating on silicon)

30 nm

50 nm

100 nm

300 nm
Fabrication of mirrors

- Grinding
- Diamond turning
- Lapping
- Polishing
Advanced Polishing Technique (Elastic Emission Machining)

In EEM, chemical reaction between solid surfaces is utilized.

Ultrafine powder particles having reactivity to the work surface are employed.

Ultra-fine particles are supplied to the work surface by ultrapure water flow.

Atom removal occurs selectively at the topmost site of the work surface.

K. Yamauchi, et al. 
Typical figuring properties using EEM

- Prefigured profile
- After 1st. figuring
- After 2nd. figuring
- Finished profile


(a) X-ray image reflected at figure corrected area
(15% of the total area on each side is not figure-corrected)

(b) X-ray image reflected at noncorrected area
Characterization of mirrors (surface roughness and figure)

Fizeau interferometer

Michelson interferometer
(Twyman-Green interferometer
(mirror: rotatable, point source)

Stitching interferometry

Atomic Force Microscopy
Stylus measurement
Characterization of mirrors (slope errors)

• Long Trace Profiler

“Results of x-ray mirror round robin metrology measurements at the APS, ESRF, and SPring-8 optical metrology laboratories”
At-wavelength phase-retrieval interferometry

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, California USA
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.

H. Yumoto et al.,
curved mirrors (focusing, collimating)

Cylindrical mirror

\[
\frac{1}{p} + \frac{1}{q} = \frac{2}{R\sin(\theta)}
\]

P=30 m, \( \theta = 0.3^\circ \), R=1,000 m, q = 2.87 m

R=100 m , q = 0.26 m

Elliptic mirror

\[
\frac{x^2}{a^2} + \frac{y^2}{b^2} = 1
\]

a = 15.5 m, b = 0.03 m, 60 cm long mirror at 30 m from the source,

\( \theta \sim 0.28^\circ - 0.37^\circ \), q = 1m
Bending mechanisms
bent-cylinder, toroidal mirror

\[ R_1 = \frac{2pq}{(p + q) \sin(\theta)} \]

\[ R_2 = \frac{R_1^2}{\sin^2 \theta} \]
bent conical mirror
– approximation to paraboloidal mirror –


double crystal monochromator
Kirkpatric-Baez (KB) optics for nanometer focusing


Spring-8/Osaka KB development

- Static and figured Si substrates
- Plasma Chemical Vapour Machining (PCVM)
- Elastic Emission Machining (EEM)
- Pt coating + thickness correction
- Focal spots below 30 nm
- Aiming less than 10 nm using multilayers

Transmition mirror: a high-pass energy filter

Silicon nitride transmission X-ray mirrors

Cornaby and D. H. Bilderback

Heat load on and cooling of a mirror

Heat load: 86 W, 0.13W/mm²
Glancing angle: 1.5°  $T_{\text{water}}=25^\circ\text{C}$
Multilayers
Recursive calculation of Fresnel coefficients and propagation

- Parratt formalism (widely used in x-ray optics) [8]

**Principle**

- Start at semi-infinite substrate surface (no reflection from back side)
- Recursive construction of amplitudes and phases from layer to layer

\[ r_{n-1,n} = a_n^{-4} \left[ \frac{r_{n,n+1} + f_{n-1,n}}{r_{n,n+1} f_{n-1,n} + 1} \right] \]

\[ r_{n,n+1} = a_n^2 \frac{E_n^R}{E_n} \]

\[ a_n = e^{-\frac{i \pi}{2} t_n \sqrt{n_n^2 - n_0^2 \cos^2 \theta}} \]

\[ R = |r_{0,1}|^2 \]
Numerical calculations

Main features
- Bragg peaks and fringes due to interference
- Positions depend on $E$ and $\Lambda$
- Intensities depend on $\Delta \rho$, $N$, $\sigma$ ...

Corrected Bragg equation

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

For $\theta >> \theta_C \Rightarrow m \cdot \lambda \approx 2 \cdot \Lambda \cdot \sin \theta$
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!
W/C multilayer $\Gamma = \frac{t_w}{\Lambda}$
Effect of interlayer diffusion

$C/W \ \Lambda=6.9 \ \text{nm} \ \Gamma=0.4$
### X-ray multilayer fabrication

<table>
<thead>
<tr>
<th>Deposition techniques</th>
<th>Vacuum</th>
<th>Particle energy</th>
<th>Deposition rate</th>
<th>Deposition area</th>
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</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
- Vacuum and purity important for EUV and soft X-rays
- High particle energy favors very thin and uniform layers
Sputtering techniques

DC (conductors)

RF (insulators)
Large area coatings (uniform, gradient)

- Relative motion source - substrate
- Masking techniques

\[ t(\vec{r}) = \int \varphi(\vec{r}, \vec{r}') \frac{d\vec{r}'}{\nu(\vec{r}')}. \]
X-ray multilayer fabrication

ESRF magnetron sputter deposition system

- Loading bay
- Escape area
- Load lock
- Deposition zone
- Mobile and modular assembly
X-ray multilayer fabrication

Sputtering techniques (Ion beam sputtering at Tohoku Univ.)

**Target**

**Shutter**

**Substrate**

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**TEM**

Mo/Si  
\( D = 6.9 \text{ nm} \)  
\( \lambda = 13.5 \text{ nm} \)  
\( \phi = 5^\circ \)

**TEM**

Cr/Sc  
\( D = 1.77 \text{ nm} \)  
\( \lambda = 3.1 \text{ nm} \)  
\( \phi = 27.5^\circ \)
X-ray multilayer characterization

X-ray reflectivity

Simulation of x-ray reflectivity

Vertical density profile

Reflectivity

$\Theta$

$10^{-6}$

$10^{-5}$

$10^{-4}$

$10^{-3}$

$10^{-2}$

$10^{-1}$

$10^{0}$

$0^\circ$ $1^\circ$ $2^\circ$ $3^\circ$ $4^\circ$ $5^\circ$ $6^\circ$

$[\text{Ir/Al}_2\text{O}_3]_{10}$

Mass density [g/cm$^3$]

Sample depth [nm]

$\rho(\text{Ir})$

$\rho(\text{Al}_2\text{O}_3)$

$\rho(\text{Si})$
X-ray scattering

- Access to atomic structure
- Phase transitions
- Coherence lengths
- Superlattice formation


Transmission electron microscopy (TEM)

- Fabrication errors
- Roughness evolution
- Crystallinity
- Interface diffusion

Complementary to x-ray measurements!
Surface metrology

- Atomic Force Microscopy (AFM)
- Interferometry
- Long Trace Profiler (LTP)
- From nm to m scale
- No depth information!

A. Rommeveaux, ESRF Optics Group
Aperiodic design:

Goal: ML with particular reflectivity profile

• \( R = R(\theta) \) for \( E = \text{const} \)
• \( R = R(E) \) for \( \theta = \text{const} \)

Find vertical composition profile

Method:

• Derive analytical expression for reflectivity
• Do inversion to obtain 1st estimate for layer sequence
• Apply fit algorithm to optimize the structure

Example:

- Ni/B₄C structure
- R(θ) = const over 20% bandwidth

Energy resolution of multilayers

\[ E = 8048 \text{ eV} \]

- Non-periodic ML \([\text{Ni/B}_4\text{C}]_{43}\)
  - \(dE/E = 20\%\)

- Periodic ML \([\text{Ru/B}_4\text{C}]_{0}\)
  - \(dE/E = 2.0\%\)

- Periodic ML \([\text{Ni/B}_4\text{C}]_{401}\)
  - \(dE/E = 0.25\%\)
Reflecting X-ray optics – Overview

Integrated reflectivity vs. $\Delta E/E$

- ESRF MLs
- Traditional ML’s
- Depth-graded ML’s
- (Mirrors/Filter)

forbidden area

$R(\text{peak}) = 100\%$

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

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Ch. Morawe – SPring-8 11.09.07
Laterally graded multilayers

Surface curvature and beam divergence define lateral gradient $d\Lambda / dx$

<table>
<thead>
<tr>
<th>Shape</th>
<th>Parabolic</th>
<th>Elliptic</th>
<th>Flat</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Angle $\theta$</strong></td>
<td>$\sin \theta = \sqrt{\frac{p}{2f}}$</td>
<td>$\sin \theta = \frac{b}{\sqrt{pq}}$</td>
<td>$\sin \theta = \frac{s}{f}$</td>
</tr>
<tr>
<td><strong>Geometry</strong></td>
<td><img src="image1" alt="Parabolic" /></td>
<td><img src="image2" alt="Elliptic" /></td>
<td><img src="image3" alt="Flat" /></td>
</tr>
</tbody>
</table>

$d$-spacing including refraction correction (modified Bragg equation)

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

Ch. Morawe – SPring-8 11.09.07
Laterally graded multilayers

- Divergent beam, focusing applications with curved substrates
- Incidence angle variation → laterally graded multilayers
- Additional depth gradient negligible (< $10^{-5}$)

Elliptic case

Focus

Source

├── Ideal
├── Model
└── Exp

Active area

B₄C

W

$\Lambda$

$q$
laterally graded multilayers

• Divergent beam, focusing applications with curved substrates
• Angle of incidence variation → laterally graded multilayers

Thickness control by
• Relative motion of source - substrate (in magnetron sputtering)
• Moving shutter (for a large gradient)
Synchrotron optics: Multilayer high flux monochromators

- Two bounce optics
- 100x larger bandwidth compared with Si(111)
- Harmonics suppression due to refraction and filling factor
- Radiation and heat load issues!
ESRF focusing experiment

- Full undulator spectrum
- Vertical line focus
- Dynamical bender
- Raw data 45 nm FWHM @ 100 μm aperture

Schwarzschild objective for laboratory microscope

Mo/Si multilayer coated
$\lambda = 13.5 \text{ nm}$

Transmission image of polymer microgrids (50×)

Yamamoto and Yanagihara group, Tohoku Univ.
Multilayer phase plate

Polarization characteristics of a transmitting Sc/Cr phase shifter.

M. Suzuki and Hirono, Hoshako (放射光), Nov. 2006, Vo. 19 No6, pp. 444-453

Reflectance of a [Sc(2.2 nm)/Cr(2.2 nm)]\textsubscript{200} multilayer, which works as a reflecting polarizer.
Application
Simultaneous measurement of X-ray reflectivity curve
Laterally d-graded multilayer on an elliptic surface

T. Matsushita et al., 2008
Other issues

- Brewster’s angle,
- Other advanced surface polishing techniques
- Influence of imperfections
  - lower reflectivity,
  - broader multilayer reflectivity curve,
  - diffuse scattering
- Radiation damages
- Standing waves
On-going developments

• Mirror and multilayers for handling much more coherent X-ray beam
  
  slope error < 0.1 μrad, roughness < 0.05 nm
  fabrication and characterization

• Nanometer focusing optics
• Multilayers with shorter period
• Stable mount, tables and support
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Dr. T. Uruga, Spring-8

CXRO_ALS web site: “X-ray Interactions with Matter” http://henke.lbl.gov/optical_constants