X-ray monochromators

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1. Controlling X-ray beam properties and the roll of crystal monochromators
2. Bragg diffraction by crystals
3. Dynamical diffraction
4. Double crystal monochromator
5. Heat load and cooling
6. Higher harmonics rejection
7. High resolution monochromators
8. Phase retarder and polarization conversion
9. Curved crystals
10. Other issues and future problems
Synchrotron Radiation - Basic Properties

High flux and brightness

Flux = \( \frac{\text{# of photons in given } \Delta \lambda/\lambda}{\text{sec, mrad } \theta} \)

Brightness (Brilliance) = \( \frac{\text{# of photons in given } \Delta \lambda/\lambda}{\text{sec, mrad } \theta, \text{ mrad } \phi, \text{ mm}^2} \)

(a measure of concentration of the radiation)

Pulsed time structure

Broad spectral range

Polarized (linear, elliptical, circular)

Small source size

Partial coherence

High stability

HIGH VACUUM ENVIRONMENT

Winick: Presentation at JASS02 Seminar
http://conference.kek.jp/JASS02/PDF_PPT/3_2_winick.ppt
Designing your experiment
- X-ray optical consideration -

**EXAFS**
(Extended X-ray Absorption Fine Structure)

Protein crystallography

\[ \lambda : 0.7 \, \text{Å} \sim 3 \, \text{Å} \quad (E : 18 \, \text{keV} \sim 4 \, \text{keV}) \]
\[ \Delta \lambda / \lambda \ (\Delta E / E) : 10^{-3} \sim 10^{-4} \]
Angular divergence : 2 \sim 0.2 \, \text{mrad}
Beam spot size : 5 \sim 200 \, \mu\text{m}
Designing your experiment
- X-ray optical consideration -

Small angle scattering

- Graph showing scattering pattern

PF 15A (bending magnet)

E: 6keV - 20 keV

\( \Delta E/E: 10^{-3} \)

Angular convergence:
  - 0.1 - 0.2 mrad (V)
  - 1 - 2 mrad (H)

Focal spot size:
  - 0.3 mm (V) - 1.0 mm (H)

Powder diffraction

- Graph showing diffraction pattern

Spring-8 BL02B (bending magnet)

E: 10 keV – 35 keV

\( \Delta E/E: \sim 10^{-4} \)

Angular divergence:
  - 0.5 – 1 mrad

Beam size:
  - V: 0.1 ~ 0.5 mm
  - H: 1 ~ 3 mm
Controlling the X-ray beam properties by X-ray crystal monochromators

- Energy, energy resolution
- Spatial spread
- Time structure
- Angular divergence
- Intensity
- Polarization
Example of beamline structure @SPring-8

- Light source
- Optics hutches
- Storage ring
- Experimental hutch
- Concrete shield
- Front-end
- Optics and transport channel
- Experimental hall
- Double-crystal monochromator
- Undulator
- Bending magnet & front-end
Lattice planes of silicon

Top view

Side view

$a_0 = 5.43095 \text{ Å}$

d-spacing

(400) : 1.3578 Å
(111) : 3.1356 Å
(311) : 1.6375 Å
(511) : 1.0452 Å

$a = 5.43095 \text{ Å}$
Crystal monochromators

Bragg’s law of diffraction

\[ 2d(\text{Å})\sin(\theta) = n\lambda(\text{Å}) = n\frac{12.4}{E(\text{keV})} \]

- \(d\): Lattice \((d)\)-spacing,
- \(\theta\): glancing angle,
- \(\lambda\): X-ray wavelength
- \(10\text{ keV} : 1.24\ \text{Å},\)
- \(1\ \text{Å} : 12.4\text{ keV}\)

Energy (wavelength) resolution

\[ \frac{\Delta E}{E} = \frac{\Delta \lambda}{\lambda} = \Delta \theta \cot \theta \]

Higher harmonics

- \(E_1 = 10\text{ keV} \ (n = 1)\)
- \(E_2 = 20\text{ keV} \ (n = 2)\)
- \(E_3 = 30\text{ keV} \ (n = 3)\)

\(E_1, E_2, E_3, \ldots\)
Energy range of SPring-8 standard monochromator

e.g. For SPring-8 standard monochromator

\[ 2d(\text{Å}) \sin(\theta) = n\lambda(\text{Å}) = n \frac{12.4}{E(\text{keV})} \]

→ Reflection
- Si 111: \( d = 3.1356 \text{ Å} \)
- Si 311: \( d = 1.6375 \text{ Å} \)
- Si 511: \( d = 1.0452 \text{ Å} \)

→ Bragg angle
- \( 3 \sim 27^\circ \)

→ Energy range
- \( 4.4 \sim 110 \text{ keV} \)

Photon energy (wavelength) can be selected by crystal, net planes, and Bragg angle.
Preparation of crystal monochromator

No or less imperfections

dislocations
stacking faults
point defects (non-uniformly distributed, striations, aggregations)

Courtesy of Sharan Instruments Co. Ltd.
X-ray diffraction by a single crystal

**Kinematical diffraction**
(imperfect crystal, small crystal)

Single scattering
X-rays are scattered only once in the crystal.

**Dynamical diffraction**
(nearly perfect crystal)

Multiple scattering
stationary wave field

Silicon
Germanium
Quartz
Diamond
gallium arsenide
X-Ray Dynamical Diffraction

• P. Ewald (1912, 1917):
  dipoles in the crystal which are excited by the incident X-ray wave and radiate X-rays.
• C. Darwin (1914): multiple reflection by lattice planes.
• M. von Laue (1931):
  continuous medium consisting of periodic dielectric constant.

• Experimental proof: in 1960’s and 1970’s, big perfect crystals (silicon, germanium, etc) became available
• Since late 1970’s, perfect crystals have been used as monochromators on synchrotron beamlines.

Textbooks and reviews
  B. W. Batterman and H. Cole, Rev. Mod. Phys. 36, 681 - 717 (1964)
    Dynamical Diffraction of X Rays by Perfect Crystals

  A. Authier, Dynamical Theory of X-Ray Diffraction, International Union of Crystallography


  M. von Laue: Roentgenstrahlen Interferenzen, 1941
Laue case and Bragg case
Reflectivity

reflectivity for Bragg case, no absorption, and thick crystal:

\[ F_{0r} = \sum_{j} f \exp(-ikr_j) \]

\[ R = \left| \frac{\gamma_h}{\gamma_0} \right|^2 \left( W + \sqrt{W^2 - 1} \right)^2 \quad (W < -1) \]

\[ R = 1 \quad (-1 \leq W \leq 1) \quad \Leftarrow \text{Total reflection} \]

\[ R = \left( W - \sqrt{W^2 - 1} \right)^2 \quad (W > 1) \]

For symmetric Bragg case, sigma polarization:

\[ \chi_{hr} = \frac{e^2 \lambda^2}{\pi mc^2 V} F_{hr} e^{-M} \]

\[ \chi_{0r} = \frac{e^2 \lambda^2}{\pi mc^2 V} F_{0r} \]

Darwin width \( \rightarrow \Delta W = 2 \)

\[ \omega = \frac{2|\chi_{hr}|}{\sin 2\theta_{BK}} \propto |F_h| \]

Shift of Bragg angle due to refraction:

\[ \Delta \theta_{\text{refraction}} = -\frac{\chi_{0r}}{\sin 2\theta_{BK}} \]
## Silicon single crystal, $E = 10$ keV

<table>
<thead>
<tr>
<th>hkl</th>
<th>Bragg angle (degree)</th>
<th>$\omega$ (arc sec)</th>
<th>$\omega$ ((\mu)rad)</th>
</tr>
</thead>
<tbody>
<tr>
<td>111</td>
<td>11.403</td>
<td>5.476</td>
<td>26.55</td>
</tr>
<tr>
<td>220</td>
<td>18.836</td>
<td>3.984</td>
<td>19.32</td>
</tr>
<tr>
<td>311</td>
<td>22.246</td>
<td>2.273</td>
<td>11.02</td>
</tr>
<tr>
<td>400</td>
<td>27.167</td>
<td>2.495</td>
<td>12.10</td>
</tr>
<tr>
<td>422</td>
<td>34.001</td>
<td>1.886</td>
<td>9.142</td>
</tr>
<tr>
<td>333</td>
<td>36.379</td>
<td>1.228</td>
<td>5.952</td>
</tr>
<tr>
<td>440</td>
<td>40.22</td>
<td>1.543</td>
<td>7.479</td>
</tr>
</tbody>
</table>

$\chi_0h$ on the web!!

http://sergey.gmca.aps.anl.gov/x0h.html
For thick absorbing crystal in the Bragg-case (reflection geometry), the reflectivity is given by

\[ R = L - \sqrt{L^2 - (1 + 4\kappa^2)} \]

\[ L - \sqrt{(W^2 - 1 - g^2)^2 + 4(gW - \kappa)^2 + W^2 + g^2} \]

\[ \kappa = \frac{\kappa h i}{\kappa h r} \]

\[ g = \frac{\kappa 0 i}{\kappa h r} \]

For thin absorbing crystal of the Laue-case (transmission geometry), the reflectivity is given by

\[ R_T = \frac{\exp(-\mu t / \gamma)}{(1 + W^2)} \left[ \sin^2 (A \sqrt{1 + W^2}) + \sinh^2 \left( \frac{\kappa A}{\sqrt{1 + W^2}} \right) \right] \]

\[ A = \pi k \left| \kappa_h r \right| t / \gamma \]
Intrinsic rocking curve for silicon

Based on the dynamical theory for perfect crystal for thick crystal and absorption considered:

\[ \omega = \frac{2|\chi_{hr}|}{\sin 2\theta_{BK}} \]

\[ \chi_{hr} = \frac{e^{2\chi_2^2}}{\pi mc^2 V} F_{hr} e^{-M} \]

\[ \Delta \theta_{refraction} = -\frac{\chi_{0r}}{\sin 2\theta_{BK}} \]

\[ \chi_{0r} = \frac{e^{2\chi_2^2}}{\pi mc^2 V} F_{0r} \]

Features:
- Diffraction width (Darwin width) of 0.1\textasciitilde100 µrad
- Peak reflectivity of \textasciitilde1 for low absorption case
Angular divergence of sources and diffraction width

Divergence of undulator radiation is the same order as diffraction width of low order reflection.

\[ \sigma_r \approx \frac{1}{\gamma} \approx 60 \mu\text{rad} \]

\[ \sigma_r \approx \frac{1}{\gamma \sqrt{N}} \approx 5 \mu\text{rad} \]
Energy resolution \[ \Delta E/E = 10^{-5} \sim 10^{-3} \]

\[ \frac{\Delta E}{E} = \cot \theta_B \sqrt{\omega^2 + \Delta \theta^2} \]

\( \omega \): intrinsic angular width of diffraction

\( \Delta \theta \): angular divergence of X-ray beam

(1) \( \omega \)

For Si 111, \( \omega = 2.66 \times 10^{-5} \) rad at 10 keV

\[ \frac{\Delta E}{E} = \omega \cot \theta = 2.66 \times 10^{-5} \times \cot(11.4^\circ) = 1.32 \times 10^{-4} \]

(2) \( \Delta \theta \)

source-to-slit distance = 30 m

slit width = 1 mm

\( \Delta \theta = 3.3 \times 10^{-5} \)

Si 111, 10 keV: \( \theta = 11.4^\circ \)

\[ \frac{\Delta E}{E} = \Delta \theta \cot \theta = 1.6 \times 10^{-4} \]
Energy resolution

\[ \frac{\Delta E}{E} = \cot \theta_B \sqrt{\Omega^2 + \omega^2} \]

\( \Omega \): divergence of source

\( \omega \): diffraction width

Using slit, collimator mirror,.. we can reduce \( \Omega_{\text{eff}} \),

\[ \frac{\Delta E}{E} = 10^{-5} \sim 10^{-3} \]
SPring-8 standard DCM

$3^\circ < \theta_B < 27^\circ$

Offset $h = 30$ mm

Adjustment stages for undulator beamline DCM

Sub-micron, sub-$\mu$rad control
Goniometer and angle accuracy

Energy scan (Si 111 d = 3.119478 Å)

\[ E_0: 10.000 \text{ keV} \quad \theta = 11.46246^\circ \]
\[ E_1: 10.001 \text{ keV} \quad \theta = 11.46130^\circ \]
\[ \Delta \theta = 0.001162^\circ = 4.18 \text{ arc s} = 20.3 \mu\text{rad} \]

0.2 arc s / step (1 \(\mu\)rad / step)

Courtesy of Kohzu Seiki Co. Ltd.
Double crystal monochromator

- Constant beam direction
- Beam height changes slightly when E is changed.
- Two crystals should be parallel to each other within sub-arc-seconds.
- Tail intensity falls off rapidly.

\[ h = 2g \cos(\theta) \]
Alignment of a double crystal monochromator

(1) Parallelity in the scattering plane: sub-\(\mu\)rad

(2) Parallelity normal to the scattering plane: \(~10^{-3} - 10^{-4}\) rad

\[
\Delta \theta = -\frac{\phi^2}{2} \tan \theta
\]

\(\psi: 10^{-3} - 10^{-4}\)

\[
\delta = 2\psi \sin \theta
\]
Courtesy of Kohzu Seiki Co. Ltd.
Stability and disturbances

Variation at the sample

- Energy:
  - 0.1 eV for XAFS,
  - sub-meV for high resolution inelastic scattering
- Intensity: less than 1%.
- Beam position: typically several tens ~ 1 μm

Source instability

- Source size change, source position change, charge distribution change

Room temperature variation
Heat load by the radiation itself
Vibration
  - floor, cooling water, vacuum pump
A number of different schemes have been developed to realize a fixed-exit beam.
$\theta$ + two translation (KEK-PF)

PF BL-4C..
Matsushita et al., NIM A246 (1986)

$h = 25\text{ mm}, \theta_B = 5\sim70^\circ$

Two cams for two translation-stages
Rotation center at 2$^{\text{nd}}$ crystal
Heat load on the mochromator 1st crystal:

→ For SPring-8 bending magnet source
  100 W & 1 W/mm²

→ For SPring-8 standard undulator source
  ~500 W & ~ 500 W/mm²

cf. Hot plate : ~ 0.02 W/mm²
CPU : ~ 0.3 W/mm²
Crystal cooling

Why crystal cooling?
Qin (Heat load by SR) = Qout (Cooling + Radiation,..)

→ with temperature rise $\Delta T$

→ $\alpha \Delta T = \Delta d$ (d-spacing change)

$\alpha$: thermal expansion coefficient

or $\Delta \theta$ (bump of lattice due to heat load)

**Miss-matching between 1st and 2nd crystals occurs:**

→ Thermal drift, Loss of intensity, Broadening of beam, loss of brightness

→ Melting or limit of thermal strain $\rightarrow$ Broken!

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[Diagram of heat transfer and cooling with labels: Qin, $T_1$, Crystal, $T_0$, Coolant/holder, Radiation & convection, Heat conduction in crystal, Heat transfer to coolant/holder.]
Solution for crystal cooling

We must consider:

- Thermal expansion of crystal: $\alpha$,
- Thermal conductivity in crystal: $\kappa$,
- Heat transfer to coolant and crystal holder.

Solutions:

(S-1) $\kappa / \alpha \rightarrow$ Larger
(S-2) Large contact area between crystal and coolant/holder $\rightarrow$ larger
(S-3) Irradiation area $\rightarrow$ Larger, and power density $\rightarrow$ smaller
## Figure of merit

<table>
<thead>
<tr>
<th></th>
<th>Silicon 300 K</th>
<th>Silicon 80 K</th>
<th>Diamond 300 K</th>
<th>Copper 300 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\kappa$ (W/m/K)</td>
<td>150</td>
<td>1000</td>
<td>2000</td>
<td>401</td>
</tr>
<tr>
<td>$\alpha$ (1/K)</td>
<td>2.5x10^{-6}</td>
<td>-5x10^{-7}</td>
<td>1x10^{-6}</td>
<td>16.5x10^{-6}</td>
</tr>
<tr>
<td>$\kappa / \alpha \times 10^6$</td>
<td>60</td>
<td>2000</td>
<td>2000</td>
<td>24</td>
</tr>
</tbody>
</table>

**Figure of merit of cooling:**
Good for silicon (80 k) and diamond (300 K)

Liquid $N_2$ : 77.36° K (-195.79° K)
Direct cooling with fin crystal

Improvement of fin-cooling crystal
Reduce radiation damage of rubber O-ring

Au-Si eutectic bonding
Direct cooling of silicon pin-post crystal + Rotated inclined geometry

Inclination angle $\beta = 80^\circ$

Grazing angle down to $1^\circ$ using $\phi$-rotation

Irradiation area enlarged to x50, power density reduced to 1/50

Applied to undulator beamline
Structure of pin-post crystal

Top plate with pin-post is bonded to base plate (manifold) using Au-Si eutectic bonding.
A rocking curve is obtained by rocking (rotating) the second crystal of the double crystal arrangement and recording the diffracted intensity.
Cryogenic cooling

Indirect side cooling

Applied to undulator beamline

LN$_2$ circulator with He refrigerator
Silicon, Liquid Nitrogen Cooling
465W, $\theta=6.9^\circ$, 25 W/mm$^2$

Spring-8 undulator beamline

Temperature distribution

Deformation distribution

prepared for SIR2000,T. Mochizuki et al. Cryogenic cooling monochromators for the SPring-8 undulator beamlines.

Coutecy of Dr. T. Mochizuki, Spring-8
Performance of pin-post cooling and cryogenic cooling

Heat load test (June 2000) up to 500 W, 500 W/mm²
IIa diamond indirect cooling

Merit:
- Good thermal properties $\rightarrow$ Capability of indirect cooling
- Higher resolution $\leftrightarrow$ Less throughput (30\textasciitilde40\% of Si)

Issues:
- Perfection of crystal $\rightarrow$ HPHT IIa diamond (Sumitomo)...
  Successive upgrade is crucial!
- Holding of crystal $\rightarrow$ X-ray topograph, Zygo
- Optimization of thermal contact $\rightarrow$ New process with In insert
- Small crystal ($<10 \text{ mm}^2$)
- Alignment: using CCD camera, PIN photodiode, thermocouple
Topograph and rocking curve

Rotation angle (arcsec)

Intensity (a.u.)

Measured
Calculated
Characterization of diamond monochromator

Improvement of crystal growth and surface finish is needed.

\( \varepsilon = 3 \) nm rad, \( I_b = 100 \) mA
Front-end slit aperture = 0.7 (v) x 1.0 (h) mm²

Low energy: 50~60% of Si DCM
High energy: Infinite size effect?
Higher harmonics rejection - total reflection mirror -

$$2d(Å) \sin(\theta) = n\lambda(Å) = n \frac{12.4}{E(keV)}$$

Film thickness: 50 nm
Surface roughness: 1 nm
Higher harmonics rejection
- Detuning of DCM -

\[ \Delta \theta = 12 \, \mu \text{rad} \]
\[ \Rightarrow 70\% \, \text{of peak intensity for fundamental (111 refl. @ 10 keV)} \]
\[ \Rightarrow 0.3\% \, \text{of peak for 3rd harmonics (333 refl. @30 keV)} \]

\[ \Delta \theta_2 = 10 \, \mu \text{rad} \Rightarrow \text{Angle change of exit beam} = 2\Delta \theta_2 = 20 \, \mu \text{rad} \]
\[ \text{Beam position change of 0.2 mm @10 m from DCM.} \]

\text{We should recognize the beam position change by DCM detuning!}
High resolution monochromator

double crystal arrangements and the DuMond diagram
Asymmetric diffraction for high resolution monochromator
Extremely High-Resolution X-Ray Monochromators

Fig. 1. Schematic top view of the experimental setup (a), and that of the intensity interferometer (b). The interferometer consists of a high-resolution monochromator using four separated crystals, a precision four-jaw slit, two semitransparent avalanche photodiodes (APDs), and coincidence circuits.

Alfred Q. R. Baron, a* Yoshikazu Tanaka, b Daisuke Ishikawa, b Daigo Miwa, b Makina Yabashia and Tetsuya Ishikawaa, b J. Synchrotron Rad. (2001). 8, 1127-1130
\[ \frac{\Delta E}{E} = \sqrt{\omega^2 + \Delta \theta^2} \cot \theta \]

When \( \theta = 89.97^\circ \)

\( \cot \theta \sim 5.2 \times 10^{-4} \)

If \( \sqrt{\omega^2 + \Delta \theta^2} \sim 10^{-3} - 10^{-4} \)

\[ \Delta E/E \sim 10^{-7} - 10^{-8} \]
Power Load Effects on Backscattering Monochromator

~130 mW Incident Beam From Si (111) mono

Flat Crystal Analyzer

Detector

Vertical Translation

Backscattering Mono At the (11 11 11)

Grazing Incidence

Normal Incidence

Energy Offset (meV)
phase retarder, polarization conversion - transmission through a thin crystal -

Concept of dispersion surfaces should be utilized to understand.

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

Principle of sagittal focusing

\[ r = \frac{2pq}{p+q} \sin \theta \]

- \( r \): radius of 2nd crystal
- \( \theta \): Bragg angle
- \( p \): source \~ crystal distance
- \( q \): crystal \~ focal point distance

Bending mechanism for SPring-8 sagittal focusing

 Applied for bending magnet beamline

\[ \text{Si 311 refl.} \]

\[ 40 \text{ keV} \]

\[ \text{Source} \sim \text{Crystal}= 36.5 \text{ m} \]

\[ \text{Crystal} \sim \text{focal point} = 16.5 \text{ m} \]
Dispersive X-ray Absorption Spectroscopy
T. Matsushita & R. P. Phizackerley:

$R = 100\text{~to~}300\text{ cm}$
$E_{H}-E_{L} = \approx 1\text{ keV}$

$n = 1000$
$n = 100$
$n = 10$
$n = 1$

t = 100\text{ ps} \times n$

Ni K-edge

稻田、丹羽、野村: 放射光、20, 242 (2007)
Multiwavelength Dispersive X-Ray Reflectometry

\[ Q = 4\pi \sin(\alpha)/\lambda \]
\[ \sim E \sin(\alpha) \]
\[ Q: 0.08 \sim 0.8 \ \text{Å}^{-1} \]
\[ Q_{\text{max}}/Q_{\text{min}} = 10 \]
\[ E_{\text{max}}/E_{\text{min}} = 10 \]

Other issues

- Phase space analysis
  position-angle (position-momentum) space
- Extended Phase space
  position-angle-wavelength space
  position-angle-energy space
- Ray tracing
- Feedback control of the DCM
- Compton scattering (heating of the 2nd crystal)
- Stress due to crystal mounting
- Surface finish and residual stress layer
- Wide band-pass monochromators
- Quick-scan monochromators

- Dispersion surface
Future subjects

• Smaller slope error, smaller roughness, smaller residual stress
• optics for handling more coherent X-rays
• More higher resolution
• Wide bandpass crystal monochromator

• optics for handling more bright (intense) beam
• optics for handling extremely short pulses
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