Cheiron School

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X-ray Monochromator

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Outline

- → X-ray beamlines at SPring-8
- → Fundamental of x-ray monochromator
 - Bragg's law, Dynamical theory,...
 - Double crystal monochromator
 - Crystal cooling
- \rightarrow Other topics

X-ray beamlines at SPring-8

Example of beamline structure @SPring-8



SPring-8 x-ray beamlines with monochromator (1)

Bemline name		Optics
BL01B1	XAFS	CM (V) + <mark>DCM</mark> + FM (V)
BL02B1	Single Crystal Structure Analysis	CM (V) + <mark>DCM</mark> + FM (V)
BL02B2	Powder Diffraction	CM (V) + DCM
BL04B2	High Energy X-ray Diffraction	SBM (H)
BL05SS	Accelerator Beam Diagnosis	DCM
BL08W	High Energy Inelastic Scattering	SBM (V) & SBM (H)
BL08B2	Нуодо ВМ	CM (V) + <mark>DCM</mark> + FM (V)
BL09XU	Nuclear Resonant Scattering	DCM + HRM
BL10XU	High Pressure Research	DCM + Double mirror (V)
BL11XU	JAEA Quantum Dynamics	DCM + HRM
BL12XU	NSRRC ID	DCM + CM (V) + HRM + FM (V)
		& SBM (H)
BL12B2	NSRRC BM	CM (V) + <mark>DCM</mark> + FM (V)
BL13XU	Surface and Interface Structure	DCM + Tandem FM (H)

- DCM : Double-crystal monochromator
- SBM : Single-bounce monochromator
- HRM : High-resolution monochromator
- CM : Collimator mirror
- FM : Focusing mirror
- V : Vertical deflection
- H : Horizontal deflection

SPring-8 x-ray beamlines with monochromator (2)

Bemline name		Optics		
BL14B1	JAEA Materials Science	CM (V) + <mark>DCM</mark> + FM (V)		
BL14B2	Engineering Science Research II	DCM + FM (V)		
BL15XU	WEBRAM	Tandem DCM		
BL16XU	Industrial Consortium ID	DCM + FM (V)		
BL16B2	Industrial Consortium BM	DCM + FM (V)		
BL19LXU	RIKEN SR Physics (long undulator)	DCM + Tandem FM (V)	Medium-length beamline	
BL19B2	Engineering Science Research I	DCM + Tandem FM (V)	Medium-length beamline	
BL20XU	Medical and Imaging II	DCM	Medium-length beamline	
BL20B2	Medical and Imaging I	DCM	1st medium-length beamline	
BL22XU	JAEA Quantum Structural Sci.	Tandem DCM		
BL24XU	Hyogo ID	DCM & DCM & DCM		
BL26B1	RIKEN Structural Genomics I	DCM + FM (V)		
BL26B2	RIKEN Structural Genomics II	DCM + FM (V)		
BL29XU	RIKEN Coherent X-ray Optics	DCM + Tandem FM (V)	1st 1-km-long beamline	

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SPring-8 x-ray beamlines with monochromator (3)

Bemline name		Optics
BL32B2	Pharmaceutical Industry	DCM + FM (V)
BL35XU	High Resolution Inelastic Scattering	DCM + HRM + FM (V)
BL37XU	Trace element Analysis	DCM + Tandem FM (H) & SBM
BL38B1	Structural Biology III	DCM + FM (V)
BL38B2	Accelerator Beam Diagnosis	DCM
BL39XU	Magnetic Materials	DCM + FM (H)
BL40B2	Structural Biology II	DCM + FM (V)
BL41XU	Structural Biology I	DCM + K-B mirror
BL44XU	Macromolecular Assemblies	DCM + FM (H)
BL44B2	RIKEN Structural Biology II	DCM + FM (V)
BL45XU	RIKEN Structural Biology I	DCM + K-B mirror
BL46XU	R&D	DCM
BL47XU	HXPES-MCT	DCM + Tandem FM (V)

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- HRM : High-resolution monochromator
- CM : Collimator mirror
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Structural biology III, BL38B1



DCM + FM (V)

Surface and interface structure, BL13XU



DCM + tandem FM (H)

High resolution inelastic scattering, BL35XU



DCM + HRM + FM(V)

Medical imaging I, BL20B2



DCM

1 km beamline, BL29XU



DCM + tandem FM (V)

Monochromators @SPring-8

Beamlines with x-ray monochromator = 40 / 49 beamlines

Double-crystal monochromator:	43
Single-bounce monochromator:	5
High-resolution monochromator:	~4

X-ray monochomator is key component for x-ray beamline. Fundamental of X-ray monochromator

X-ray monochromator using perfect crystal

\rightarrow Principle of monochromator

Bragg reflection from perfect single crystal

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

d: Latiice *(d)*-spacing,

 θ : glancing angle,

 λ : X-ray wavelength



→ Crystal: silicon, diamond,...

Requirements for X-ray monochromator

 \rightarrow Monochromatize x-rays of required photon energy

with required energy width

with required throughput (efficiency)

→ Fixed-exit beam using double-crystal monochromator

 \rightarrow Focusing using bending mechanism

. . .

 \rightarrow Cooling under heat-load from SR beam

Lattice planes of silicon

(400) : 1.3578 Å

(111) : 3.1356 Á

(311) : 1.6375 Å

(511) : 1.0452 Å

Top view



Side view



Energy range of SPring-8 standard monochromator

e.g. For SPring-8 standard monochromator



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

Dynamical theory - Two wave case -

Two wave equations

Following two equations are derived using Maxwell's equations for 3-dimensional periodic medium (= perfect single crystal).

$$\left(\mathbf{k}_{0}^{2}-k^{2}\right)E_{0}-\chi_{-h}PK_{0}^{2}E_{h}=0$$

$$\left(\mathbf{k}_{h}^{2}-k^{2}\right)E_{h}-\chi_{h}PK_{0}^{2}E_{0}=0$$

 $k_{\rm h} = k_0 + h$: Momentum conservation

- *h* : Reciprocal lattice vector
- E_0, E_h : Fourier components of electric field
- K_0 : Incident wave vector in vacuum
- k_0, k_h : Refracted and reflected wave vectors in crystal
- *k* : Mean wave number in crystal

 $\chi_0, \chi_h,...$: Fourier components of the polarizability (Negative values, 10⁻⁶~10⁻⁵)

P : Polarization factor (
$$\sigma$$
: *P*=1, π : *P*=cos2 $\theta_{\rm B}$)

Dispersion surface

Using two equations, we obtain following :



Two dispersion surfaces close to two Laue spheres (O, H) for each polarization, showing the gap near Bragg condition.

Boundary condition of wave vector

First, we must consider connections of waves from vacuum into crystal and from crystal to vacuum, to solve the equations.



Tangential component of wave vector must be continuous.

Incident wave in vacuum

- \rightarrow Refracted wave in crystal
- → Bragg reflection
- \rightarrow Reflected wave in the crystal
- \rightarrow Reflected wave in vacuum

Laue case and Bragg case





Laue case

Bragg case

Asymmetry ratio

$$\begin{cases} \gamma_0 = \hat{K}_0 \cdot n \\ \gamma_h = \hat{K}_h \cdot n \end{cases} \qquad b = \frac{\gamma_0}{\gamma_h} \end{cases}$$



Laue case: *b*>0

Symmetric Bragg case: *b*= 1

Bragg case: *b*< 0

Symmetric Bragg case: b= -1

Normalized parameter W for deviation from Bragg condition

Parameter W is related to the gap between two dispersion surfaces

and total reflection occurs at -1 < W < 1 for Bragg case.



 $\theta_{\rm BK}$: Kinematical Bragg angle $\Delta \theta$: Deviation angle from $\theta_{\rm BK}$

 K_0, K_h, h are in the same plane.

$$W = \left\{ \Delta \theta \sin 2\theta_{BK} + \frac{\chi_{0r}}{2} \left(1 - \frac{\gamma_h}{\gamma_0} \right) \right\} \sqrt{\frac{\gamma_0}{|\gamma_h|}} \frac{1}{|\chi_{hr}| \cdot |P|}$$

For symmetric Bragg case, sigma polarization:

$$W = \left\{ \Delta \theta \sin 2\theta_{BK} + \chi_{0r} \right\} \frac{1}{|\chi_{hr}|}$$

Amplitude ratio

From these equations, we obtain the ratio $r=E_h/E_0$ as a function of parameter *W*. For Bragg case, no absorption, and thick crystal:

$$\begin{cases} r = \frac{E_h}{E_0} = -\sqrt{\frac{\gamma_0}{|\gamma_h|}} \frac{|\chi_{hr}|}{|\chi_{-h}|} \frac{|P|}{P} (W + \sqrt{W^2 - 1}) & (W < -1) \\ r = \frac{E_h}{E_0} = -\sqrt{\frac{\gamma_0}{|\gamma_h|}} \frac{|\chi_{hr}|}{|\chi_{-h}|} \frac{|P|}{P} (W + i\sqrt{1 - W^2}) & (-1 \le W \le 1) & \leftarrow \text{Total reflection} \\ r = \frac{E_h}{E_0} = -\sqrt{\frac{\gamma_0}{|\gamma_h|}} \frac{|\chi_{hr}|}{|\chi_{-h}|} \frac{|P|}{P} (W - \sqrt{W^2 - 1}) & (W > 1) \end{cases}$$

Movement of tie point

(1) Lower angle (2) Near Bragg condition (3) Higher angle *W*< -1 -1< *W*< 1 *W*> 1 *K*₀ *K*₀ $K_{\rm h}$ $K_{\rm h}$ *K*₀ K_{h} Vacuum 1117 7777 7777 1/// 777 7777 Crystal **k**_h **k**_h **k**_h Η Η Η h h h **k**₀ k_0 **k**₀ Ο \mathbf{O} \mathbf{O}

Total reflection

Reflectivity

Finally, we obtain reflectivity

for Bragg case, no absorption, and thick crystal:

$$\begin{cases} R = \frac{|\gamma_h|}{\gamma_0} \left| \frac{E_h}{E_0} \right|^2 = \left(W + \sqrt{W^2 - 1} \right)^2 \quad (W < -1) \\ R = 1 \quad (-1 \le W \le 1) \quad \leftarrow \text{Total reflection} \\ R = \left(W - \sqrt{W^2 - 1} \right)^2 \quad (W > 1) \end{cases}$$
For symmetric to the symmetric term of te



For symmetric Bragg case, sigma polarization:

$$W = \left\{ \Delta \theta \sin 2\theta_{BK} + \chi_{0r} \right\} \frac{1}{|\chi_{hr}|}$$

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:

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

Intrinsic rocking curve for silicon

Based on the dynamical theory for perfect crystal

for thick crystal and absorption considered:



Features:

• Diffraction width (Darwin width) of $0.1 \sim 100 \mu rad$

Peak reflectivity of ~1 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.

Energy resolution



Double-crystal monochromator

Double crystal monochromator (DCM)

Fixed-exit operation of double-crystal monochromator is very crucial for end-users of beamlines.

To obtain fixed-exit beam independent of Bragg angle:

 → Use the same crystals and *d*-spacing for 1st and 2nd crystals
 → Keep parallel setting

Fixed-exit by DCM

Two arrangements for DCM

(1) θ_1 +translation+ θ_2 link

(2) θ + two translation link



θ_1 + translation + θ_2 computer link



<image>

PF BL-14C Kawata and Ando, NIM A246 (1986)

 $h=150 \text{ mm}, \ \theta_{\rm B}=2.6\sim 25^{\circ}$

SPring-8 BL15XU SPring-8 information Vol. 5, No.1 (2000) $h=100 \text{ mm}, \ \theta_{\rm B}=5.7\sim72^{\circ}$

Large offset, long-stroke translation Difficulty of parallelism between 1st and 2nd crystal

Boomerang link type



Kirkland, NIM-A291 (1990)

 $h=50 \text{ mm}, \theta_{\rm B}=5\sim85^{\circ}$

SR Ist CRYSTAL 2nd CRYSTAL

UVSOR BL 1A, BL 7A Hiraya et al., RSI 66 (1995)

 $\theta_{\rm B} = 18.5 \sim 71.5^{\circ}$

→ Difficulties for crystal cooling and multi-stage adjustment
 → Low rigidity
θ + two translation (KEK-PF)



Matsushita et al., NIM A246 (1986)



h=25 mm, $\theta_{\rm B}=5\sim70^{\circ}$

Two cams for two translation-stages Rotation center at 2nd crystal

θ + two translation (APS)





h=35 mm, $\theta_{\rm B}=5\sim30^{\circ}$

 θ -y-z computer link Rotation center at *h*/2-height

SPring-8 standard DCM





Sub-micron, sub-µrad control

 $3^{\circ} < \theta_{\rm B} < 27^{\circ}$ Offset *h*= 30 mm

θ-y-z computer-cam link

 θ -*y*: linked by computer control *y*-z: linked by cam

Cam shape for fixed-exit

$$\left(y^{2} - \frac{h^{2}}{4}\right)\left(z^{2} - \frac{h^{2}}{4}\right) = \frac{h^{4}}{16}$$



θ-*y* computer + *y*-*z* cam link



Crystal cooling

Heat load on 1st 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,..)

- \rightarrow with temperature rise ΔT
- $\rightarrow \alpha \Delta T = \Delta d$ (*d*-spacing change)

 α : thermal expansion coefficient

or $\rightarrow \varDelta \ \theta$ (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
- \rightarrow Melting or limit of thermal strain \rightarrow Broken !



Solution for crystal cooling

We must consider:

Thermal expansion of crystal: α , Thermal conductivity in crystal: κ , Heat transfer to coolant and crystal holder.

Solutions:

(S-1) κl α → Larger
(S-2) Large contact area between crystal and coolant/holder
→ larger
(S-3) Irradiation area → Larger, and power density → smaller

Figure of merit

	Silicon 300 K	Silicon 80 K	Diamond 300 K
<i>⊮</i> (W/m/K)	150	1000	2000
<i>α</i> ′ (1/K)	2.5x10 ⁻⁶	-5x10 ⁻⁷	1x10 ⁻⁶
κ / α x10 ⁶	60	2000	2000

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

For SPring-8 case

Bending magnet beamline

Power and density : ~ 100 W, ~1 W/mm² @40 m Method:

 \rightarrow Direct cooling with fin crystal

← S-2

Undulator beamline

(Linear undulator, N= 140, $\lambda u= 32$ mm) Power and density : ~500 W , ~500 W/mm² @40 m Methods:

- \rightarrow Direct cooling of silicon pin-post crystal \leftarrow S-2
 - + Rotated inclined geometry (\rightarrow 10 W/mm²) \leftarrow S-3
- \rightarrow or Cryogenic cooling using LN₂ circulation \leftarrow S-1
- \rightarrow or Indirect cooling of IIa diamond crystal \leftarrow S-1

Direct cooling with fin crystal









Fins with Inserted metal

Applied to bending magnet beamline

Performance of fin crystal (1)



Mechanical deformation removed

Direct cooling with fin crystal

Improvement of fin-cooling crystal

Reduce radiation damage of rubber O-ring





Au-Si eutectic bonding

Direct cooling with fin crystal

Reduce radiation damage of rubber O-ring







Performance of fin crystal (2)

X-ray image for Si 311 refl.



$$\theta_{\rm B}$$
= 5°, E = 43.4 keV
 $\theta_{\rm B}$ = 10°, E = 21.8 keV

$$\theta_{\rm B}$$
= 20°, E = 11.1 keV

Slit: $3 \text{ mm}^{\vee} \times 40 \text{ mm}^{H}$

Current status

- Practical use both (311) and (111) crystals
- $\cdot 2 \sim 4$ sec. twist due to fabrication process must be reduced.
- -No heat strain for (111) crystal at 12 keV photon
- -Radiation damage of O-ring is improved by side-inlet.
- Durability test is under way.

Performance of fin crystal (3)



Si 111 refl. Ring current= 100 mA

Direct cooling of silicon pin-post crystal + Rotated inclined geometry



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.



Pin-post structure



Fine pins are fabricated to increase cooling efficiency.

Limitation of sandblast

Water flow in the pin-post crystal



DCM with pin-post crystal



DCM view from upstream

Inside DCM: Stages + pin-post crystal

Performance of pin-post crystal



Rocking curve widths agree well.

Cryogenic cooling

<image>

Applied to undulator beamline



LN₂ circulator with He refrigerator



Performance of pin-post cooling and cryogenic cooling

Heat load test (June 2000) up to 500 W, 500 W/mm²



Ila diamond indirect cooling

Merit:

- Good thermal properties \rightarrow Capability of indirect cooling
- Higher resolution (\leftarrow > Less throughput (30 \sim 40% 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 mm[□])
- Alignment: using CCD camera, PIN photodiode, thermocouple

Ila diamond crystal

Nearly perfect crystal using High Pressure High Temperature (HPHT) synthesis is available in <001> growth direction.

To maximize the photon flux, 111 refl. in Bragg case is used. Effective size of Diamond (111) is, however, small !



Crystal handling is crucial: → Mounting without strain → Alignment

Crystal mounting



Topograph and rocking curve





Diamond monochromator (SPring-8 BL39XU)



→ Bragg angles: $4.5 \sim 45^{\circ}$

(Photon-energy compatible with silicon)

 $\rightarrow \theta$ -y-z computer link

Diamond monochromator (SPring-8 BL39XU)



Characterization of diamond monochromator

Photon flux





 ε = 3 nm. rad, $I_{\rm b}$ = 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 ? Improvement of crystal growth and surface finish is needed.

Efficiency with monochromator - Photon flux estimation -

Effective band width (Integrated intensity)

Simple understanding



For double crystal monochromator



When you need flux \rightarrow Si 111 refl. When you need resolution \rightarrow Si 311, Si 511 refl,... Photon flux (throughput) after monochromator can be estimated by effective band width.

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Photon flux (ph/s) =
Photon flux from light source
(ph/s/0.1%bw)
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x 1000

x Effective band width of monochromator

Photon flux at bending magnet beamline



Example of photon flux estimation at bending magnet beamline BL02B1. (Photon flux density at 50 m from the source)
Photon flux at undulator beamline



We can obtain photon flux of $10^{13} \sim 10^{14}$ ph/s/100 mA/mm² using standard undulator sources and Si 111 reflections at SPring-8 beamlines.

Other topics

Higher harmonics rejection

For higher harmonics rejection

 \rightarrow Total reflection mirror

 \rightarrow Detuning of DCM

Total reflection mirror

U Substrate material

Si for white radiation SiO_2 for monochromatic beam

Coating material

Pt, Rh, Ni,...



Depending on energy, reflectivity, absorption edges,..

Glancing angle

 $2 \sim 10 \text{ mrad}$ (For SPring-8 X-ray beamline)

Depending on energy, reflectivity, absorption edges,..

□ Mirror length

400 mm \sim 1 m (For SPring-8 X-ray beamline) Depending on the beam size and glancing angle e.g. 100 µrad × 50 m/5 mrad= 1 m

Example of mirror reflectivity



Surface roughness: 1 nm

Detuning of DCM



e.g. $\Delta \theta$ = 12 µrad

 \rightarrow 70% of peak intensity for fundamental (111 refl. @ 10 keV)

 \rightarrow 0.3% of peak for 3rd harmonics (333 refl. @30 keV)

e.g. $\Delta \theta_2 = 10 \mu rad \rightarrow Angle change of exit beam = 2\Delta \theta_2 = 20 \mu rad Beam position change of 0.2 mm @10 m from DCM. We should understand the beam position change by DCM detuning!$

Adjustable inclined geometry



We change the lattice plane by tilting the 1st and 2nd crystals simultaneously, and we can select 111 refl., 311 refl., 511 refl,...

Sagittal focusing



$$r = \frac{2pq}{p+q}\sin q$$

- *r*: radius of 2nd crystal
- θ : Bragg angle
- *p*: source ~ crystal distance
- *q*: crystal ~ focal point distance

0



e.g. Sagittal focusing images



Si 311 refl. 40 keV Source~Crystal= 36.5 m

Crystal~focal point= 16.5 m

Applied for bending magnet beamline

Inclination stage and elevation stage for bending magnet beamlines



(b) Ideal reflection axis



- \rightarrow For higher energy resolution using collimator mirror
- \rightarrow For higher photon flux using focusing mirrors
- \rightarrow For higher harmonics rejection

Other issues (1)

Compton scattering shielding

Cooling for 2nd crystal, stages, and others

Vibration control

Rigidity of stages \rightarrow less number of stages Coolant circulation, flexible tube,...

Radiation damages

Degradation of rubber O-ring Charge-up and damage to the control system

Other issues (2)

Surface contamination

Vacuum quality → UHV monochromator Recovering using ozone cleaning

Heat transfer analysis, simulation, fundamental data acquisition

Thermal conductivity measurement as material, surface roughness, pressure, temperature,...

Issues in the future

→ For more stable operation of monochromator Vibration control (→ sub-microns, sub-µrad)

Prevention of degradation due to radiation damage

 \rightarrow For better fabrication of crystal,

lower strain under high-heat-load condition

- \rightarrow For higher power and power density
- \rightarrow For wider energy range (\rightarrow lower, \rightarrow higher)
- \rightarrow For higher energy resolution
- → For 4GLS monochromator

Thank you for your attention