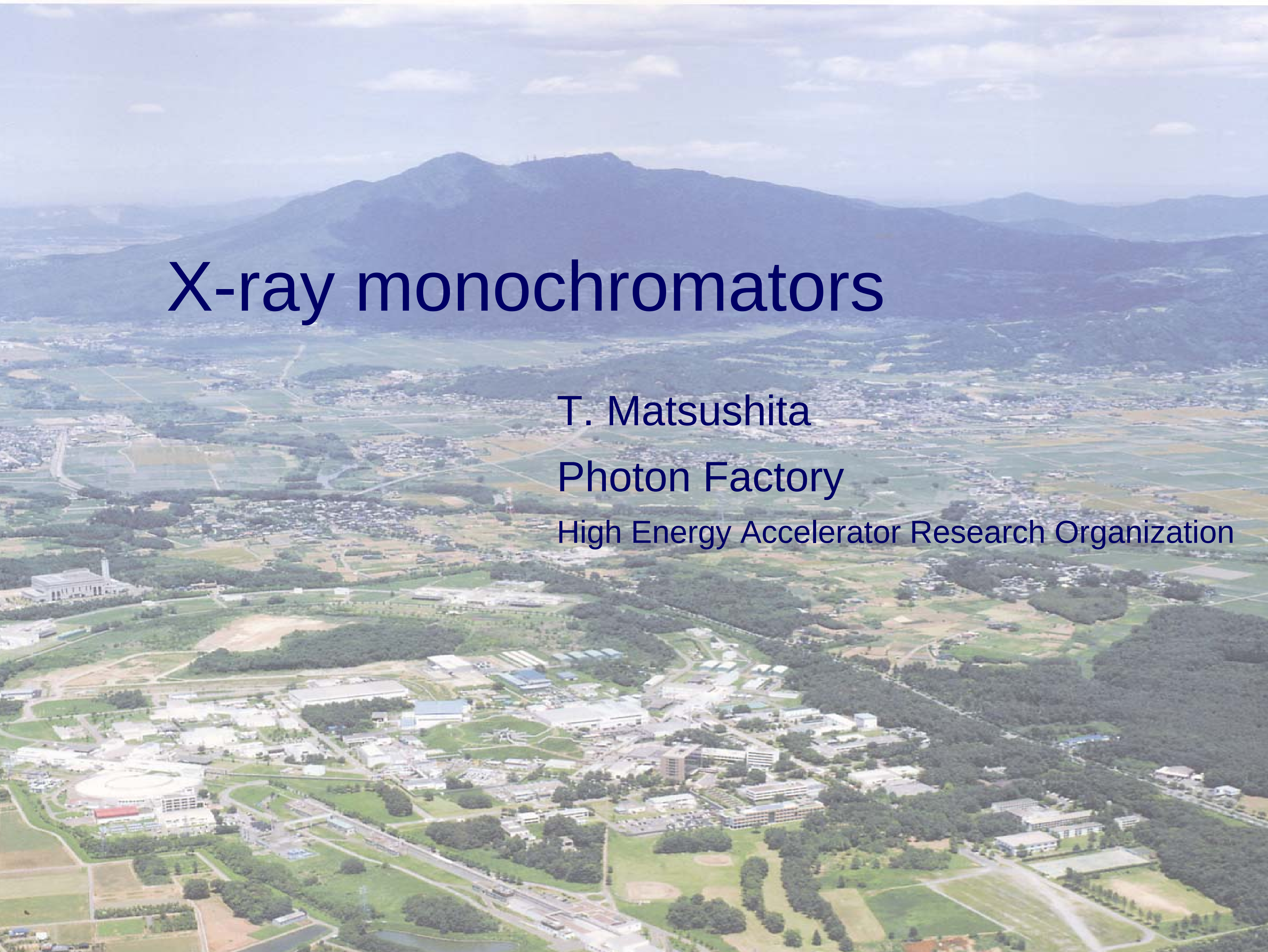


X-ray monochromators

T. Matsushita
Photon Factory
High Energy Accelerator Research Organization

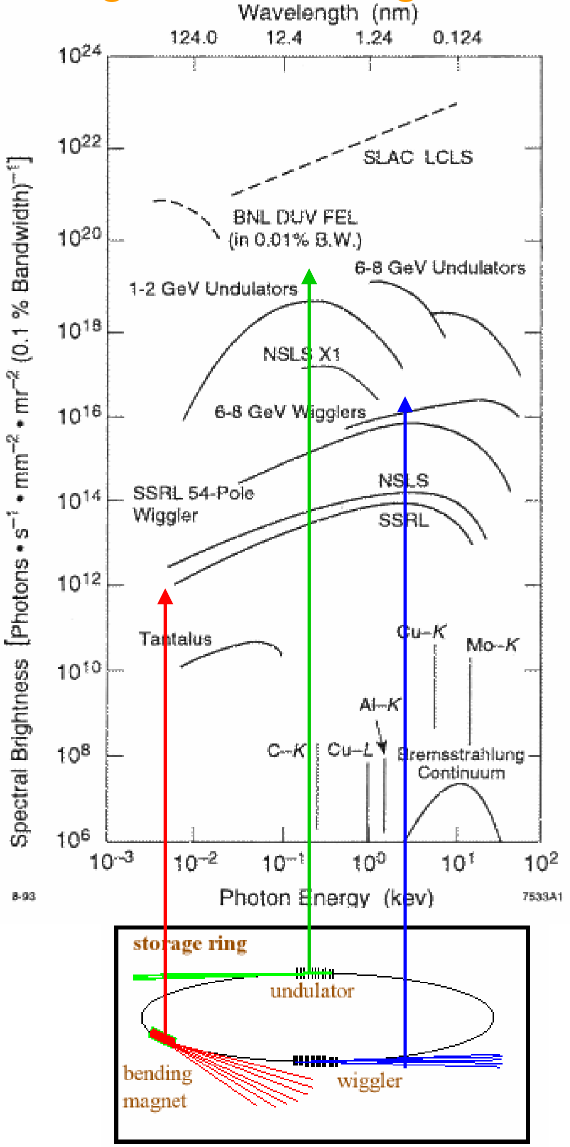
outline

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

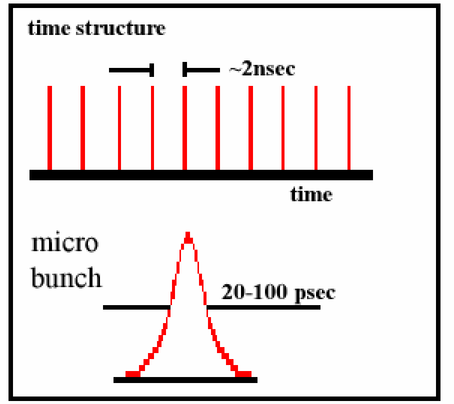
Winick: Presentation at JASS02 Seminar
http://conference.kek.jp/JASS02/PDF_PPT/3_2_winick.ppt

Synchrotron Radiation - Basic Properties

High flux and brightness



Pulsed time structure



- Broad spectral range
- Polarized (linear, elliptical, circular)
- Small source size
- Partial coherence
- High stability
- HIGH VACUUM ENVIRONMENT

Flux = $\dfrac{\text{\# of photons in given } \Delta\lambda/\lambda}{\text{sec, mrad } \theta}$

Brightness (Brilliance)

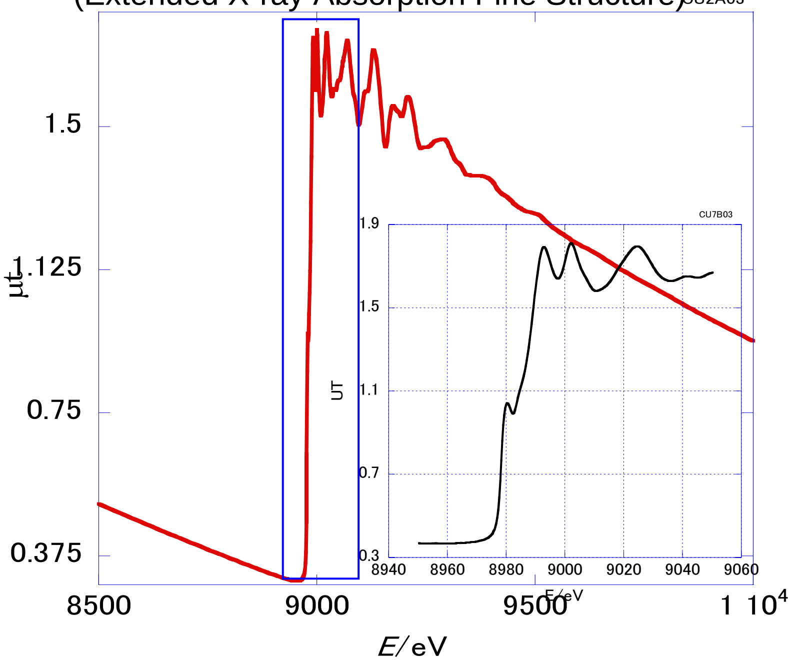
$$= \frac{\text{\# of photons in given } \Delta\lambda/\lambda}{\text{sec, mrad } \theta\text{, mrad } \varphi\text{, mm}^2}$$

(a measure of concentration of the radiation)

Designing your experiment
- X-ray optical consideration -

EXAFS

(Extended X-ray Absorption Fine Structure)

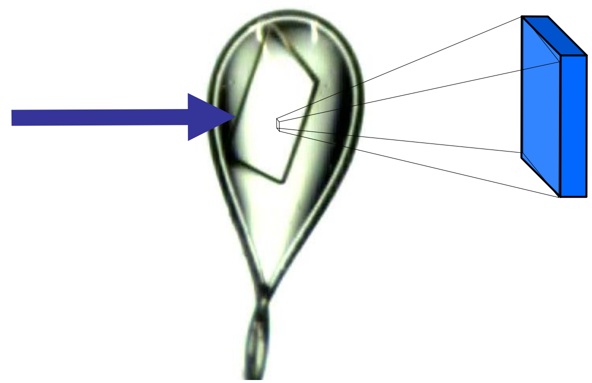


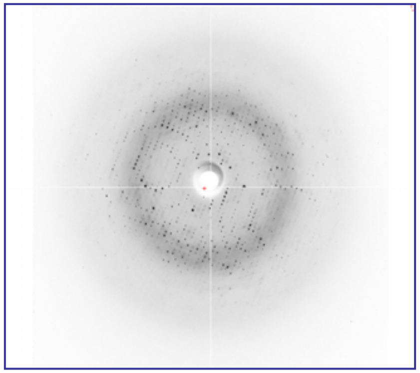
E: ~4 keV - ~20 keV (~10 keV - ~50 keV)

ΔE/E: ~10-4

Beam size: 1 mm x 10 mm – 1 μm²

Protein crystallography





λ : 0.7 Å ~ 3 Å (E : 18 keV ~4 keV)

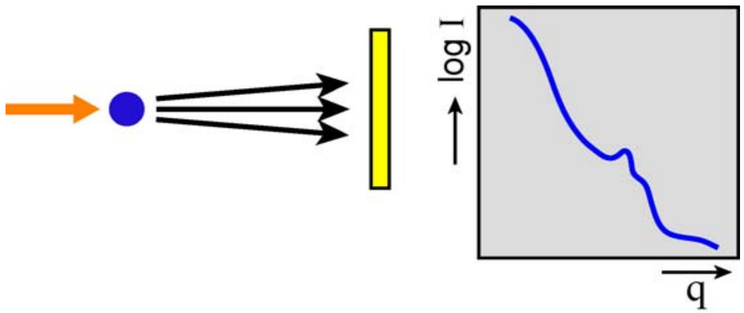
Δλ/λ (ΔE/E) : 10^{-3} ~10^{-4}

Angular divergence : 2 ~ 0.2 mrad

Beam spot size : 5 ~ 200 μm

Designing your experiment
- X-ray optical consideration -

Small angle scattering



PF 15A (bending magnet)

E: 6keV- 20 keV

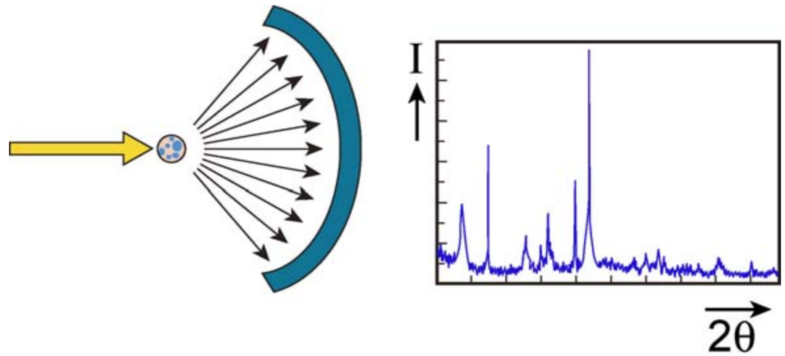
ΔE/E: 10^{-3}

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



Spring-8 BL02B (bending magnet)

E: 10 keV – 35 keV

ΔE/E: ~10^{-4}

Angular divergence:

0.5 – 1 mrad

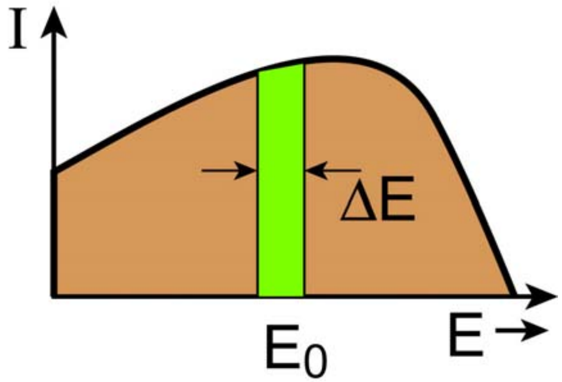
Beam size:

V: 0.1 ~ 0.5 mm

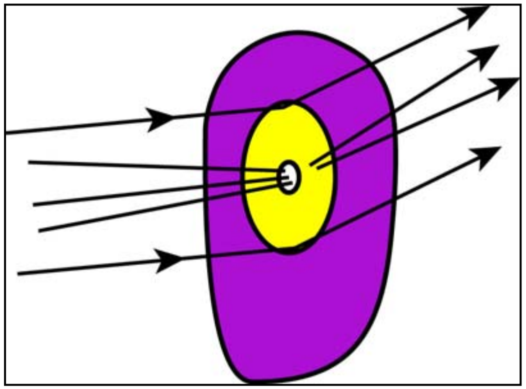
H: 1 ~ 3 mm

Controlling the X-ray beam properties by X-ray crystal monochromators

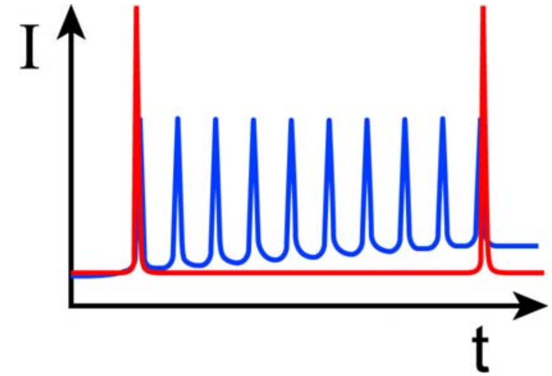
Energy, energy resolution



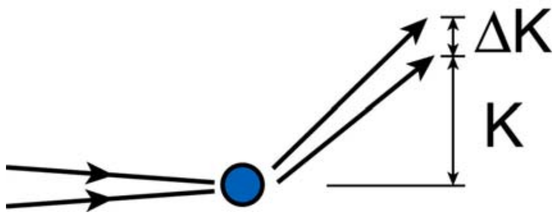
Spatial spread



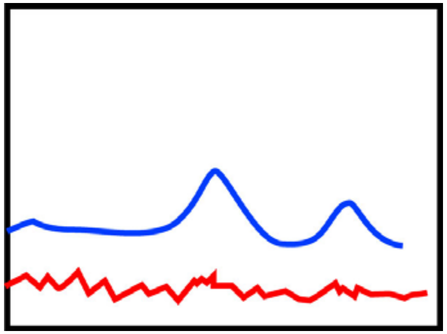
Time structure



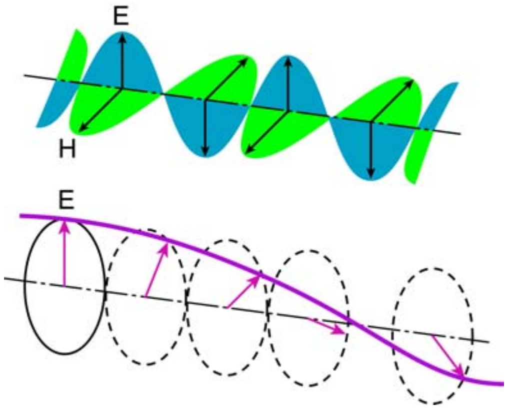
Angular divergence



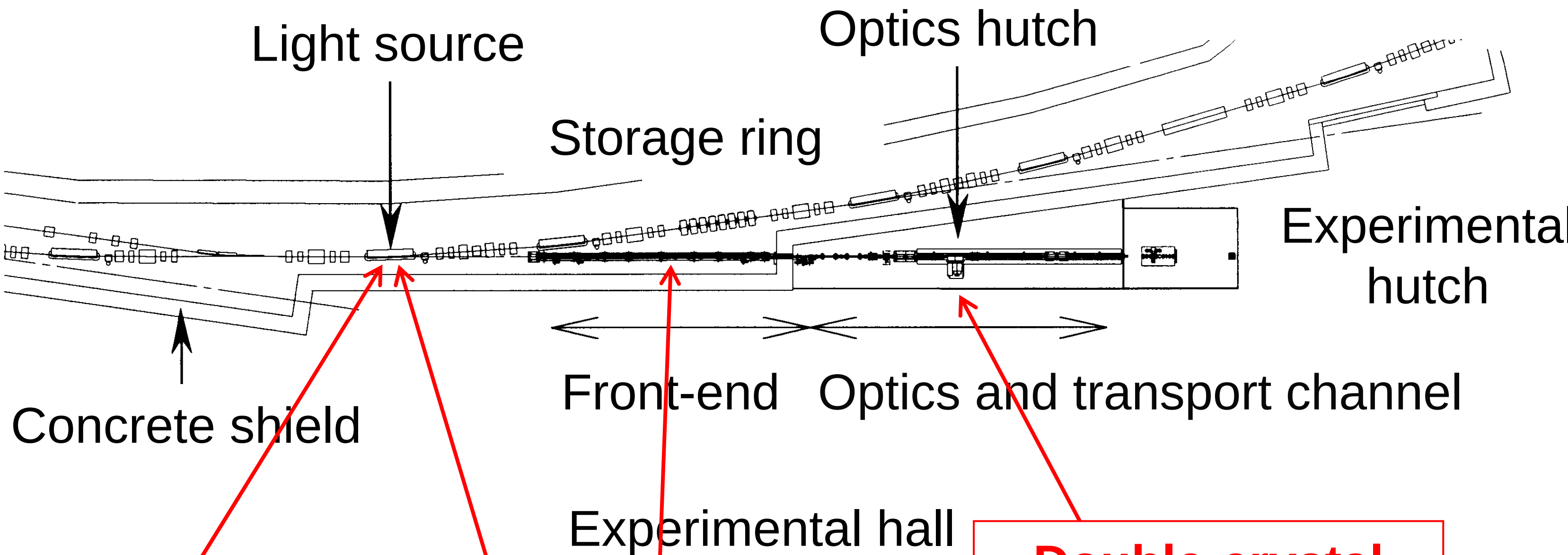
intensity

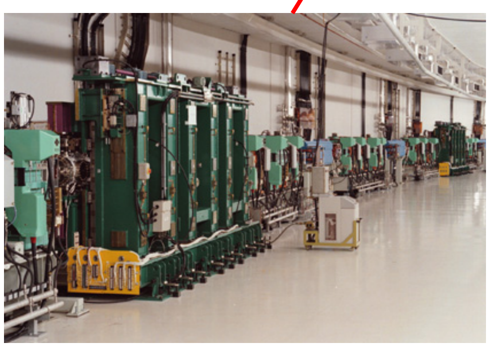


polarization

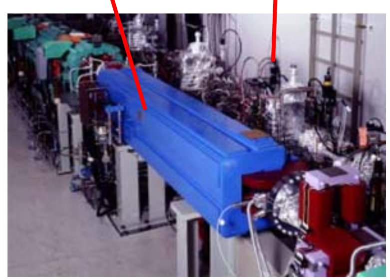


Example of beamline structure @SPring-8





Undulator



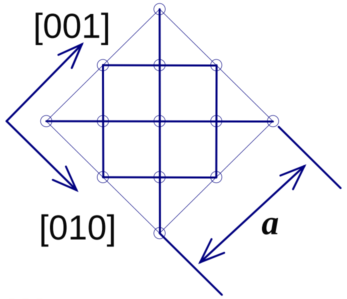
Bending magnet & front-end

Double-crystal monochromator

Lattice planes of silicon

a_0 = 5.43095 Å

Top view



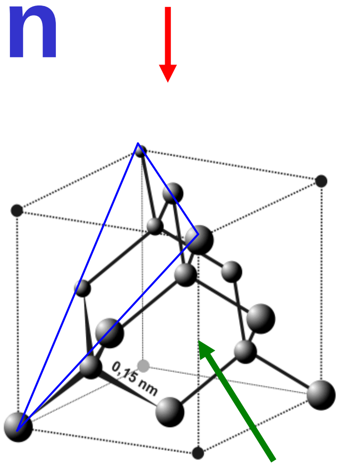
d-spacing

(400) : 1.3578 Å

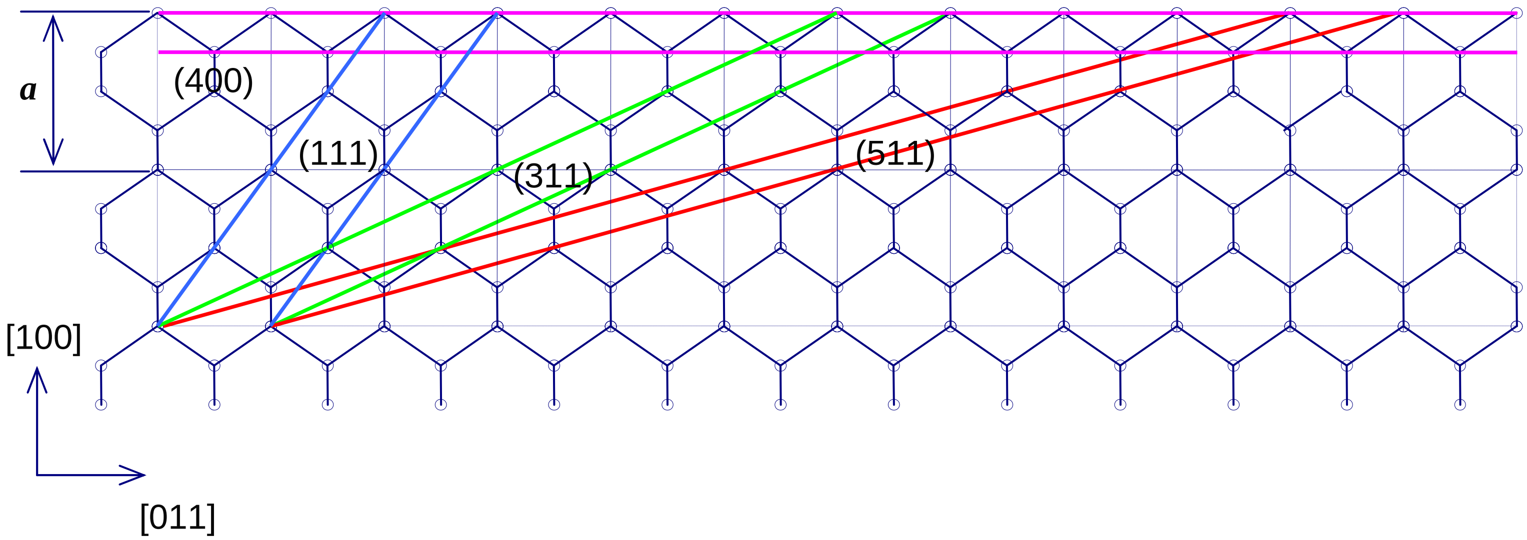
(111) : 3.1356 Å

(311) : 1.6375 Å

(511) : 1.0452 Å



Side view

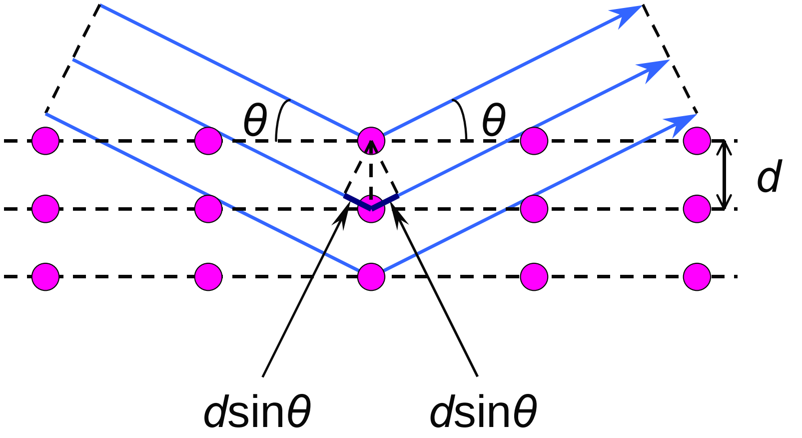


Crystal monochromators

Bragg's law of difraction

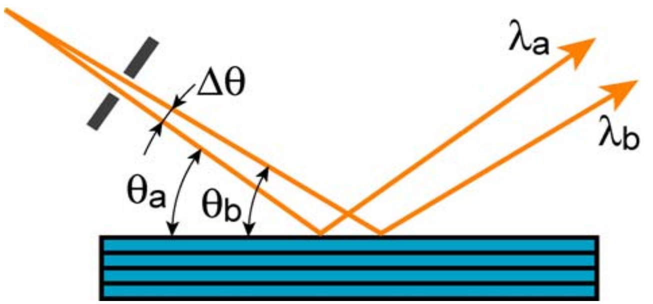
$$\boxed{2d(\text{Å})\sin(\theta) = n\lambda(\text{Å}) = n\frac{12.4}{E(keV)}}$$

d: Latiice *(d)*-spacing,
θ: glancing angle,
λ: X-ray wavelength
10 keV : 1.24 Å,
1 Å : 12.4 keV



Energy (wavelength) resolution

$$\frac{\Delta E}{E} = \frac{\Delta\lambda}{\lambda} = \Delta\theta \cot\theta$$

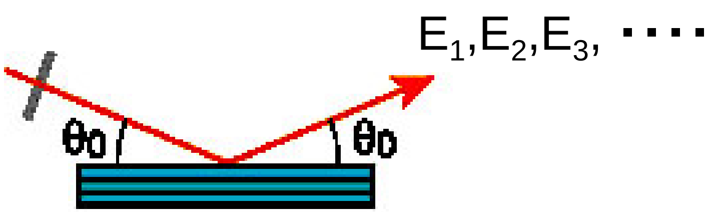


Higher harmonics

E_1 = 10 keV (n = 1)

E_2 = 20 keV (n = 2)

E_3 = 30 keV (n = 3)



Energy range of SPring-8 standard monochromator

e.g. For SPring-8 standard monochromator

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

→ **Reflection**

Si 111 : d = 3.1356 Å

Si 311 : d = 1.6375 Å

Si 511 : d = 1.0452 Å

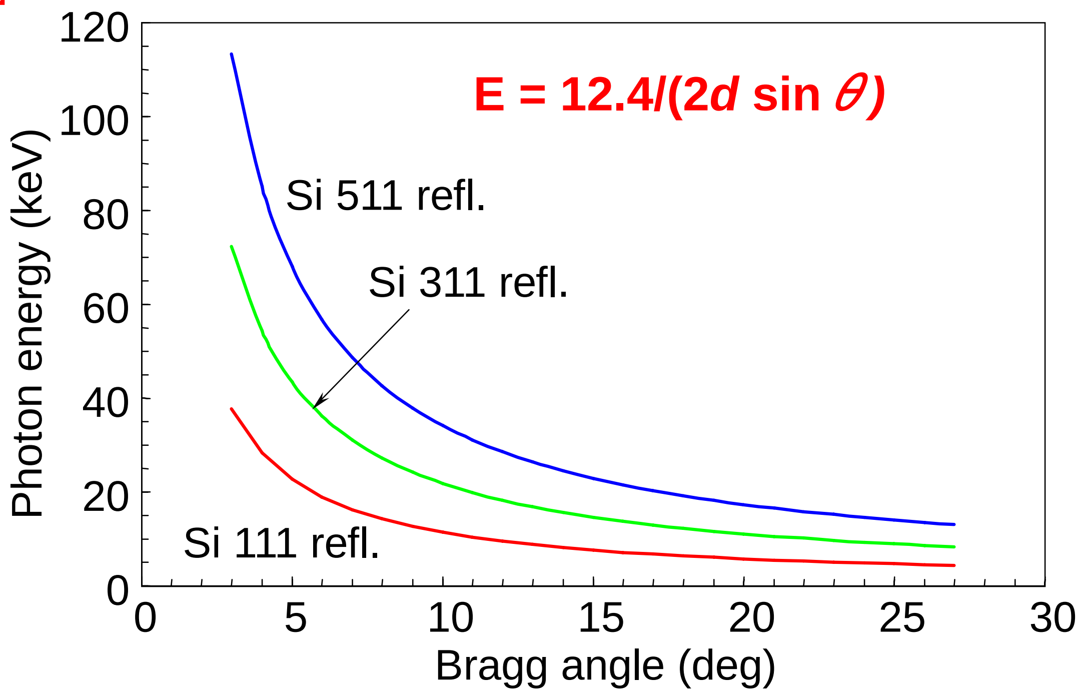
…..

→ **Bragg angle**

3～27°

→ **Energy range**

4.4～110 keV



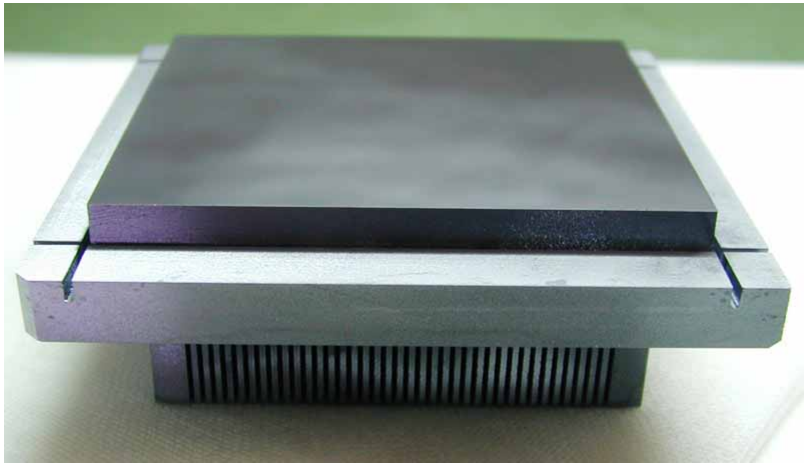
Photon energy (wavelength) can be selected

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)



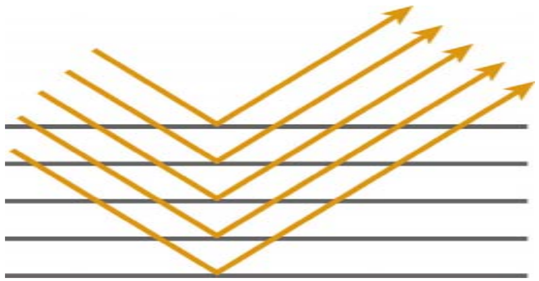
Courtesy of Sharan Instruments Co. Ltd.

X-ray diffraction by a single crystal

Kinematical diffraction

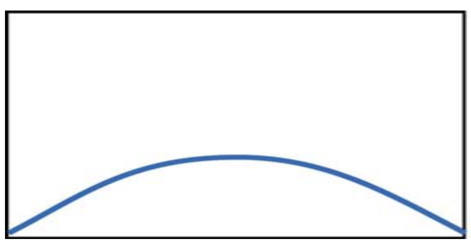
(imperfect crystal,

small crystal)



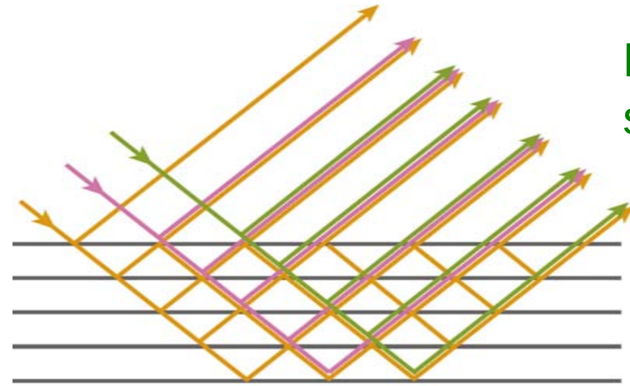
Single scattering

X-rays are scattered only once in the crystal.



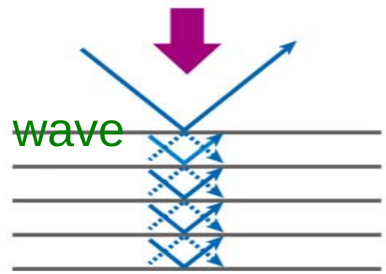
Dynamical diffraction

(nearly perfect crystal)



Multiple scattering

stationary wave field



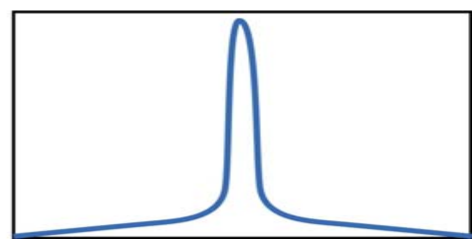
Silicon

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

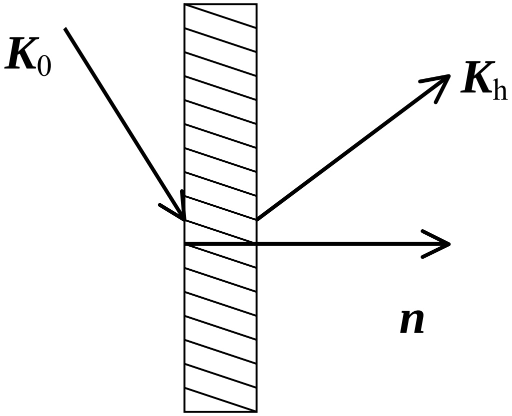
B. W. Batterman and H. Cole, Rev. Mod. Phys. 36, 681 - 717 (1964)
Dynamical Diffraction of X Rays by Perfect Crystals

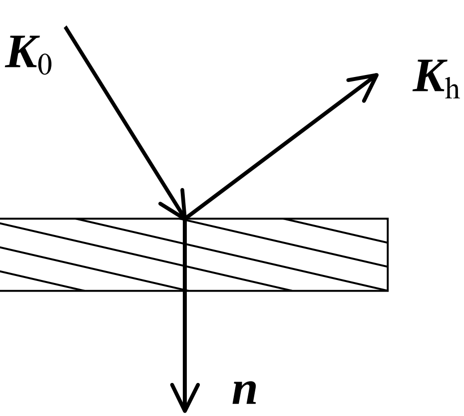
A. Authier, Dynamical Theory of X-Ray Diffraction, International Union of Crystallography Monographs on Crystallography No. 11. Oxford: Oxford University Press, 2001

R. W. James: The Dynamical Theory of X-Ray Diffraction, in Solid State Physics (Seitz and Turnbull) vol.15 (1963), Academic Press

M. von Laue: *Roentgenstrahlen Interferenzen* , 1941

Laue case and Bragg case





Laue case **Bragg case**

$$F_{0r} = \sum_j f_j \exp(-ik\, r_j)$$

Reflectivity

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

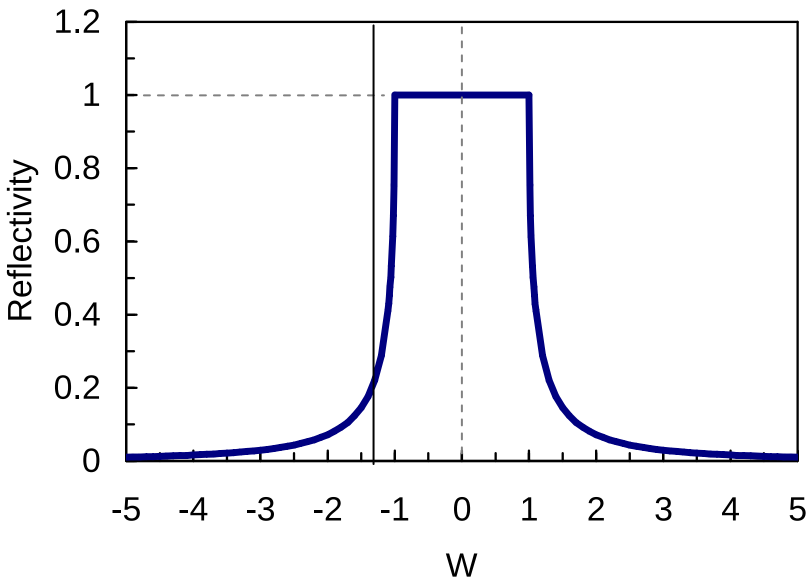
$$\chi_{hr} = \frac{e^2\lambda^2}{\pi mc^2 V} F_{hr} e^{-M}$$

$$R = \frac{|\gamma_h|}{\gamma_0}\left|\frac{E_h}{E_0}\right|^2 = \left(W + \sqrt{W^2 - 1}\right)^2 \quad (W < -1)$$

$$R = 1 \quad (-1 \le W \le 1) \qquad \leftarrow \text{Total reflection}$$

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

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



For symmetric Bragg case, sigma polarization:

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

Darwin width → Δ W= 2

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

Shift of Bragg angle due to refraction:

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

Silicon single crystal, E = 10 keV

hkl	Braqg angle (degree)	ω (arc sec)	ω (μrad)
111	11.403	5.476	26.55
220	18.836	3.984	19.32
311	22.246	2.273	11.02
400	27.167	2.495	12.10
422	34.001	1.886	9.142
333	36.379	1.228	5.952
440	40.22	1.543	7.479

χ0h on the web!!!

http://sergey.gmca.aps.anl.gov/x0h.html

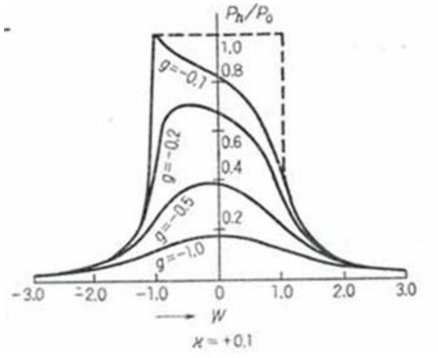
For thick absorbing crystal in the Bragg-case (reflection geometry), the reflectivity is given by

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

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

$$\kappa = \frac{\chi_{hi}}{\chi_{hr}}$$

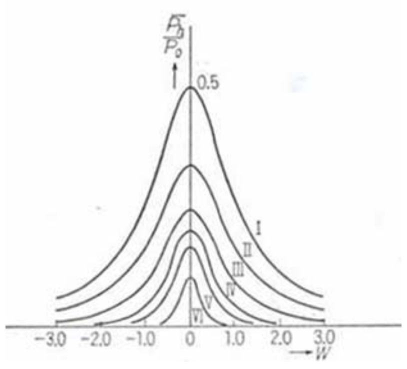
$$g = \frac{\chi_{0i}}{|\chi_{hr}|}$$



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

$$R_T = \frac{\exp(-\mu t/\gamma)}{(1 + W2)}\left[\sin^2(A\sqrt{1+W^2}) + \sinh^2\frac{\kappa A}{\sqrt{1+W^2}}\right]$$

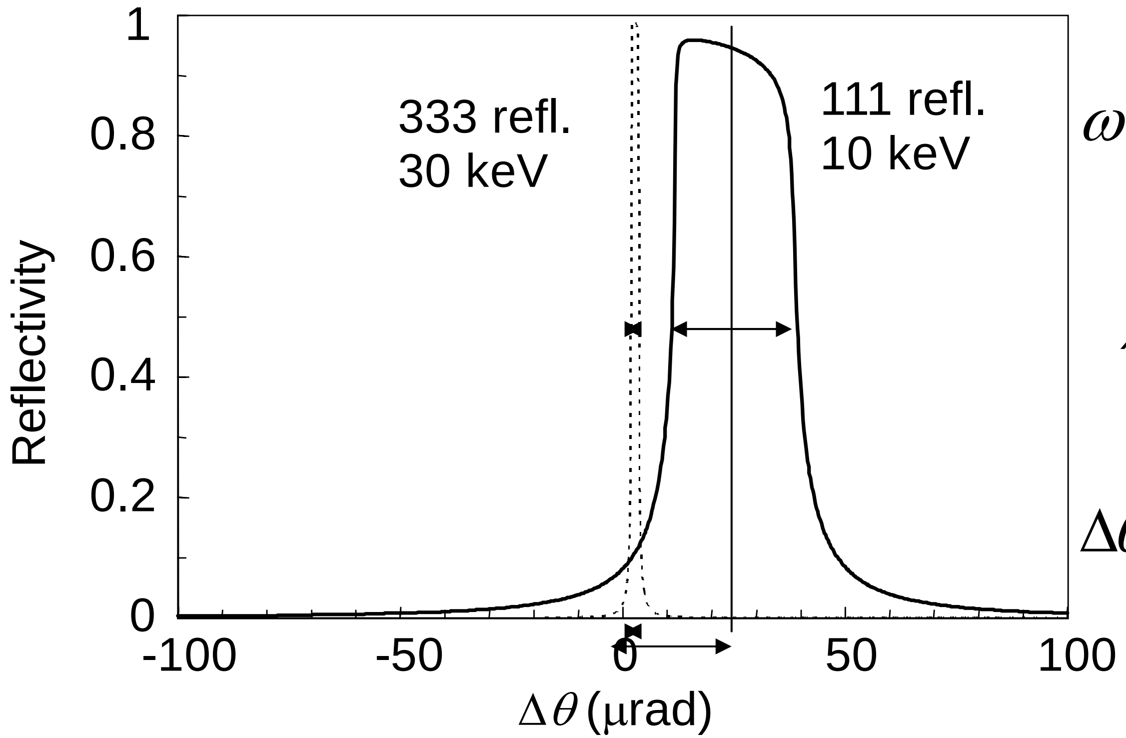
$$A = \pi k\,|\chi_{hr}|\,t/\gamma$$



Intrinsic rocking curve for silicon

Based on the dynamical theory for perfect crystal

for thick crystal and absorption considered:



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

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

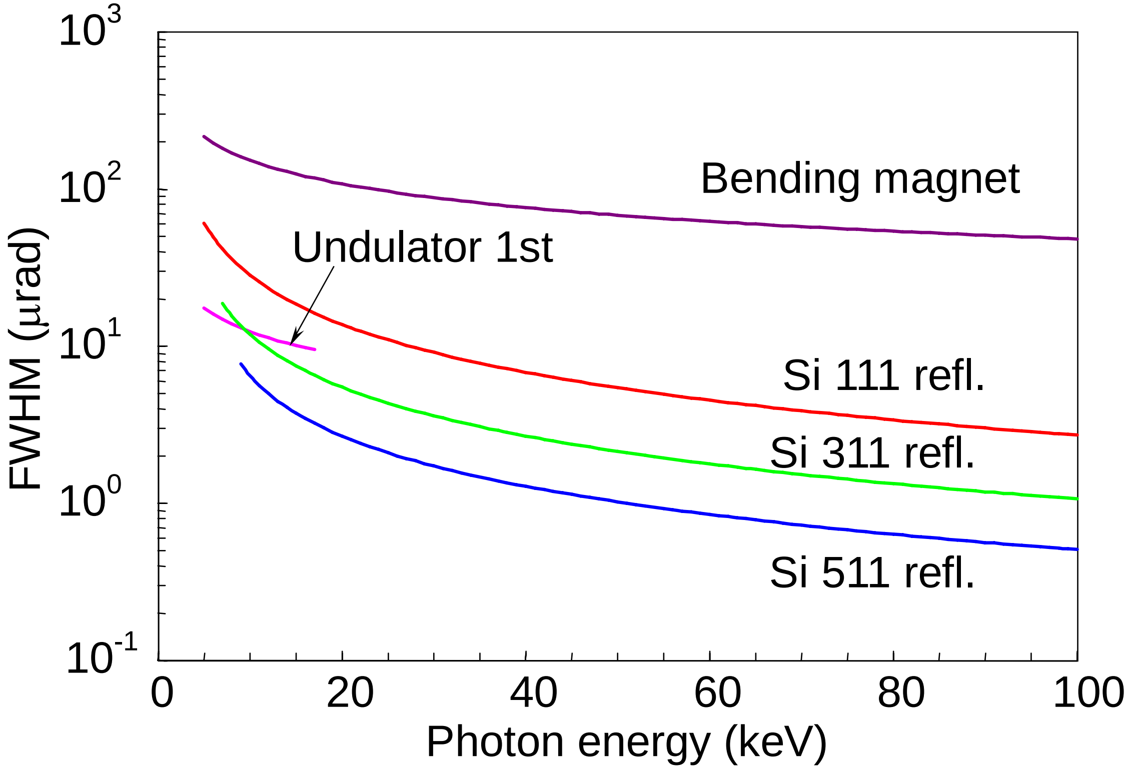
$$\Delta\theta_{refraction} = -\frac{\chi_{0r}}{\sin 2\theta_{BK}}$$

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

Features:

- Diffraction width (Darwin width) of 0.1～100 μrad
- Peak reflectivity of ~1 for low absorption case

Angular divergence of sources and diffraction width



SPring-8 bending magnet

$$\sigma_{\mathrm{r}} \approx \frac{1}{\gamma} \approx 60\,\mu\mathrm{rad}$$

Undulator (N= 140)

$$\sigma_{\mathrm{r}} \approx \frac{1}{\gamma\sqrt{N}} \approx 5\,\mu\mathrm{rad}$$

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

Energy resolution

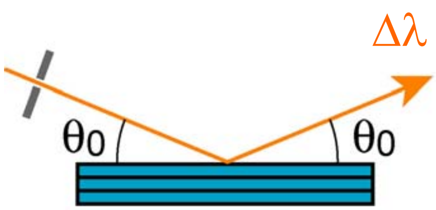
$\Delta E/E = 10^{-5} \sim 10^{-3}$

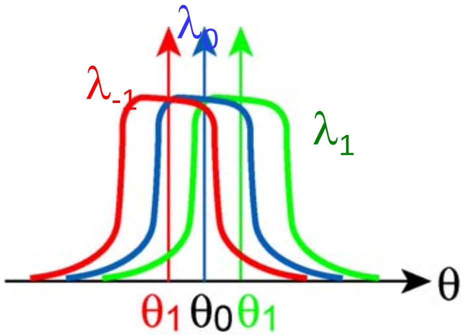
$$\frac{\Delta E}{E} = \cot\theta_B \sqrt{\omega^2 + \Delta\theta^2}$$

ω: intrinsic angular width of diffraction

$\Delta\theta$: angular divergence of X-ray beam

(1) ω





$2d \sin(\theta_0) = \lambda_0$

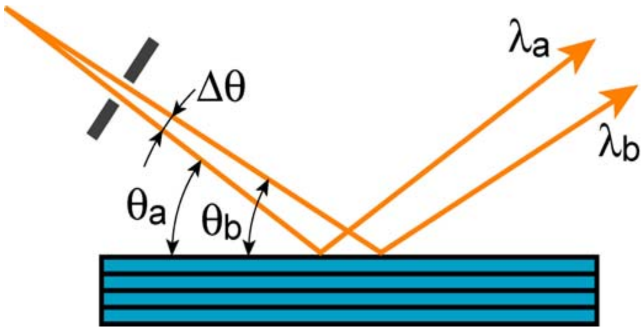
$2d \sin(\theta_{-1}) = \lambda_{-1}$

$2d \sin(\theta_1) = \lambda_1$

For Si 111, ω = 2.66 x 10^{-5} rad at 10 keV

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

(2) $\Delta\theta$



source-to-slit distance = 30 m

slit width = 1 mm

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

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

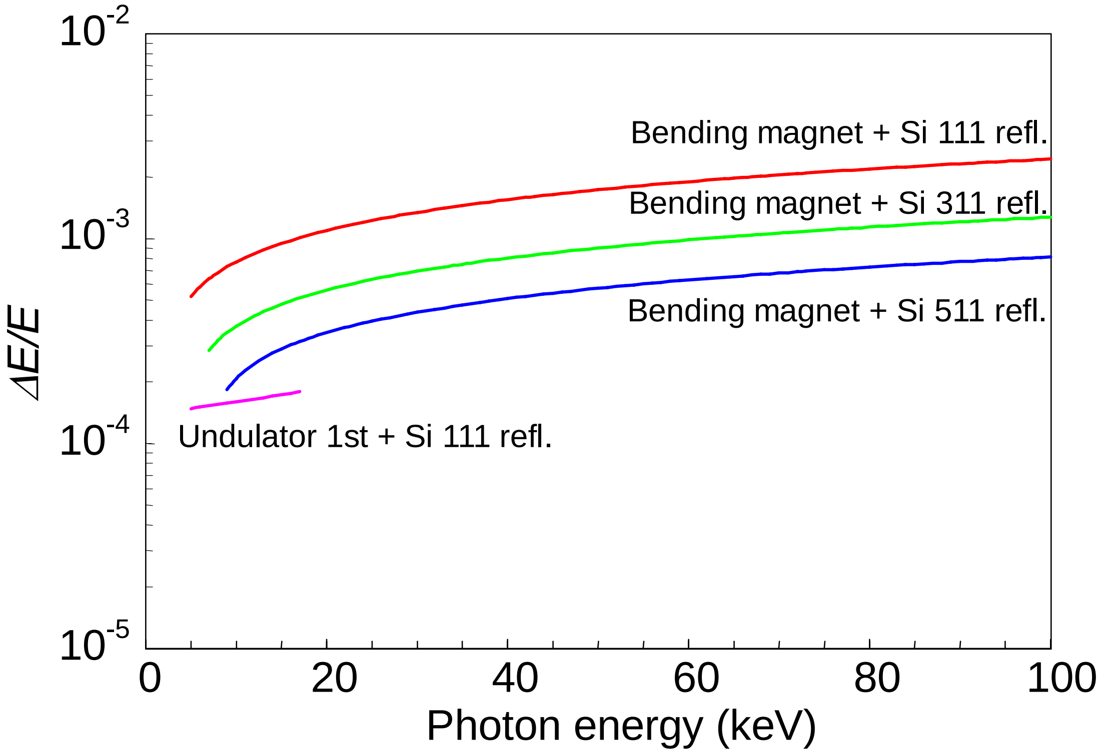
$\Delta E/E = \Delta\theta \cot\theta = 1.6 \times 10^{-4}$

Energy resolution

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

Ω: divergence of source

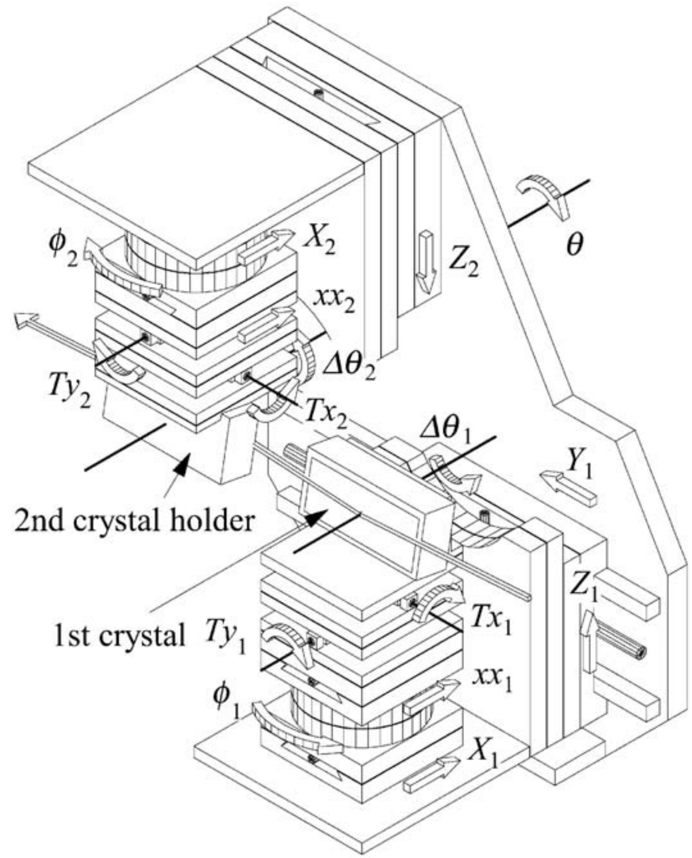
ω: diffraction width



Using slit, collimator mirror,.. we can reduce Ω_{eff},

$\Delta E/E = 10^{-5} \sim 10^{-3}$

SPring-8 standard DCM



$3^o < \theta_B < 27^o$

Offset *h*= 30 mm

Adjustment stages

for undulator beamline DCM

Sub-micron, sub-μrad control

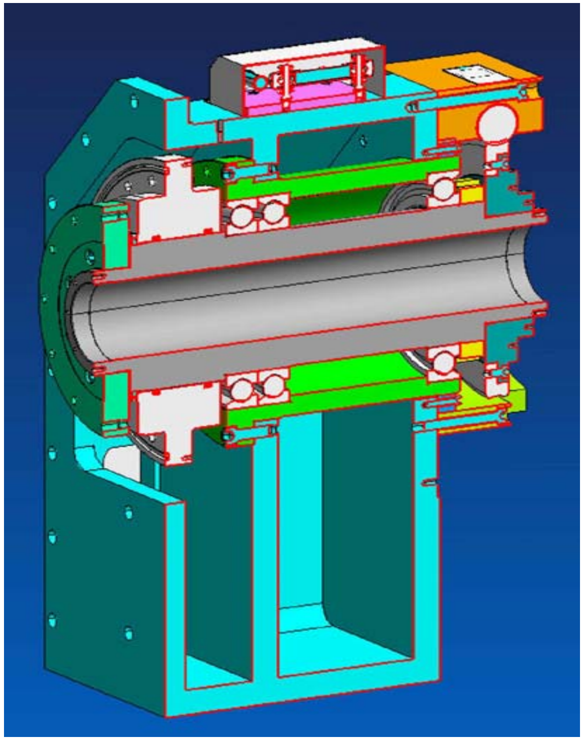
Goniometer and angle accuracy

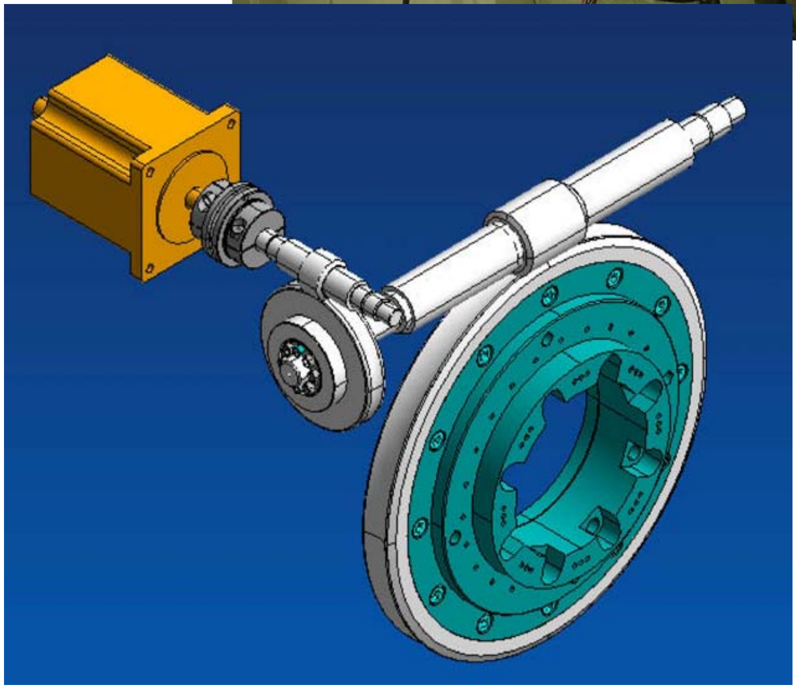
Energy scan (Si 111 d = 3.119478 Å)

E_0: 10.000 keV $\theta = 11.46246^\circ$

E_1: 10.001 keV $\theta = 11.46130^\circ$

$\Delta\theta = 0.001162^\circ$ = 4.18 arc s = 20.3 μrad





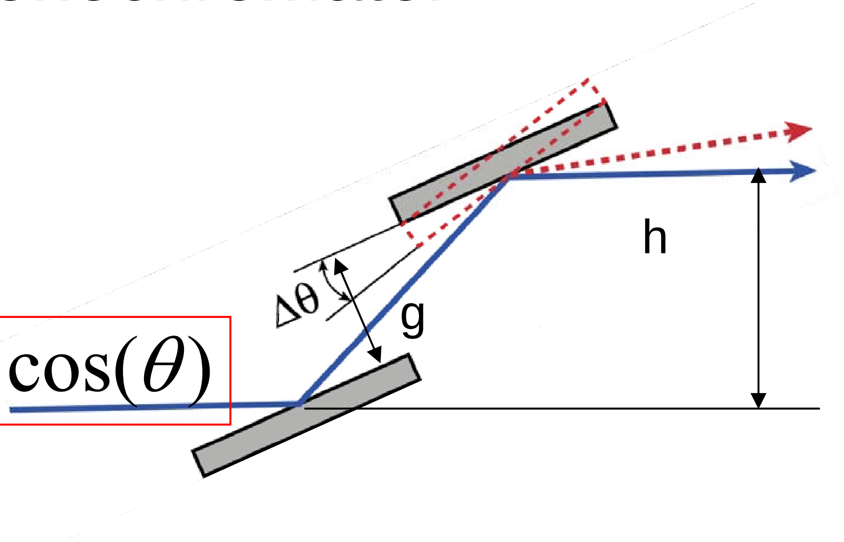
0.2 arc s / step (1 μrad / step)

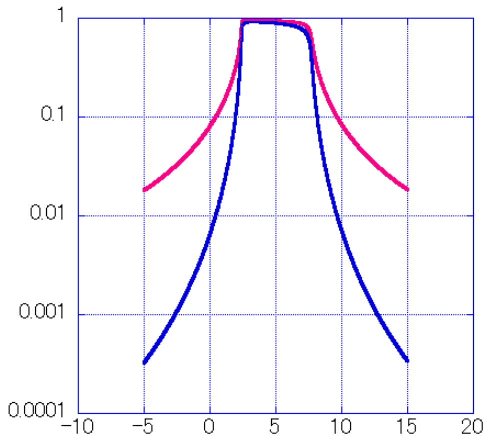
Courtesy of Kohzu Seiki Co. Ltd.

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.

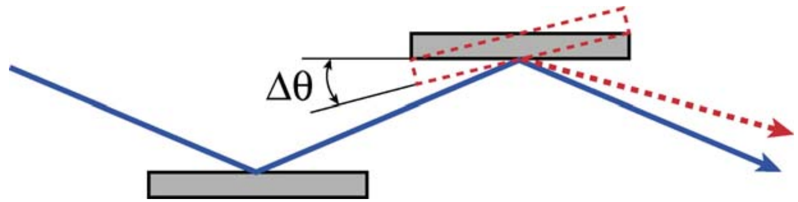
$$h = 2g\cos(\theta)$$



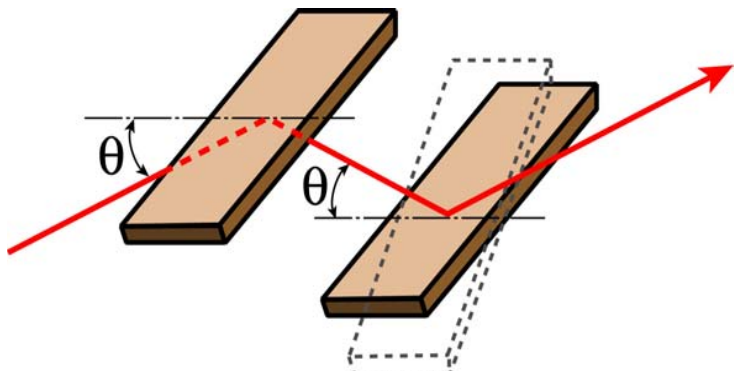


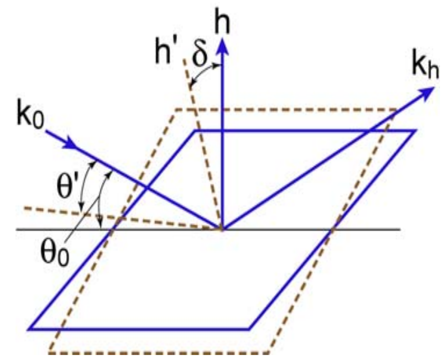
Alignment of a double crystal monochromator

(1) Parallelity in the scattering plane: sub-μrad



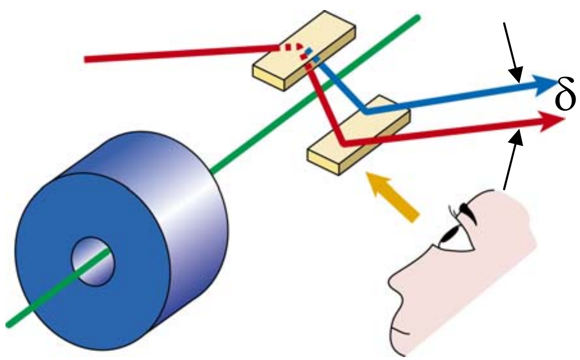
(2) Parallelity normal to the scattering plane: ~10^{-3} - 10^{-4} rad

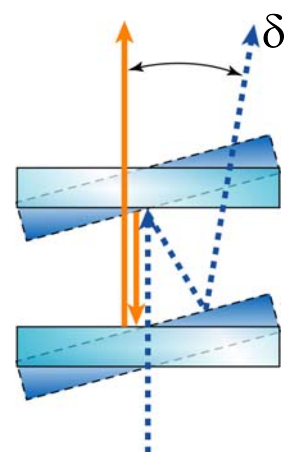




$$\Delta\theta = -\frac{\phi^2}{2}\tan\theta$$

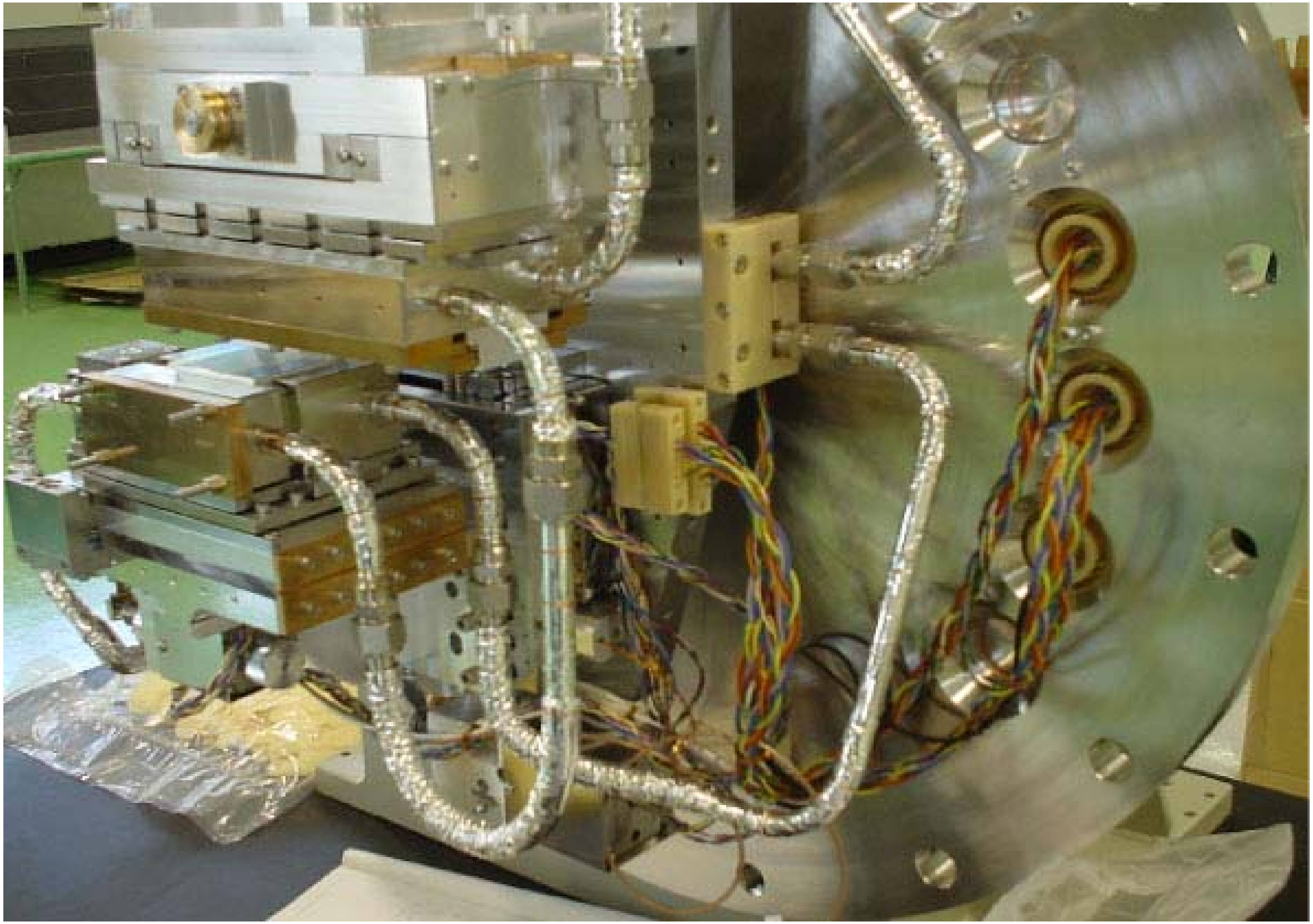
(3) Parallelity to the rotating axis :





ψ: 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

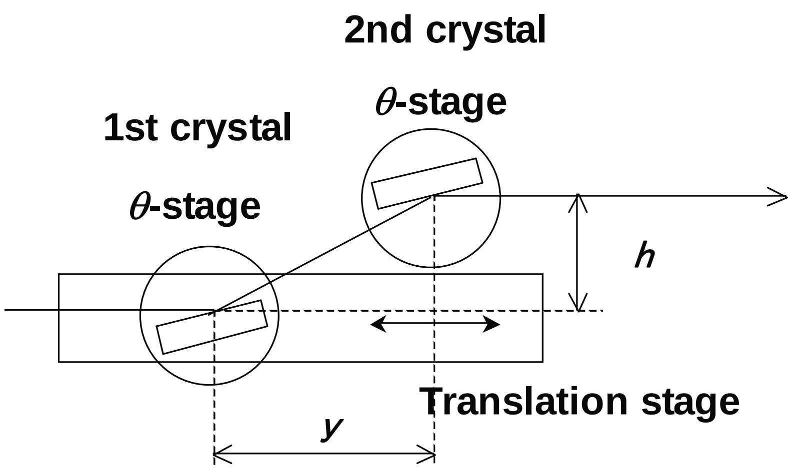
Heat load by the radiation itself

Vibration

floor, cooling water, vacuum pump

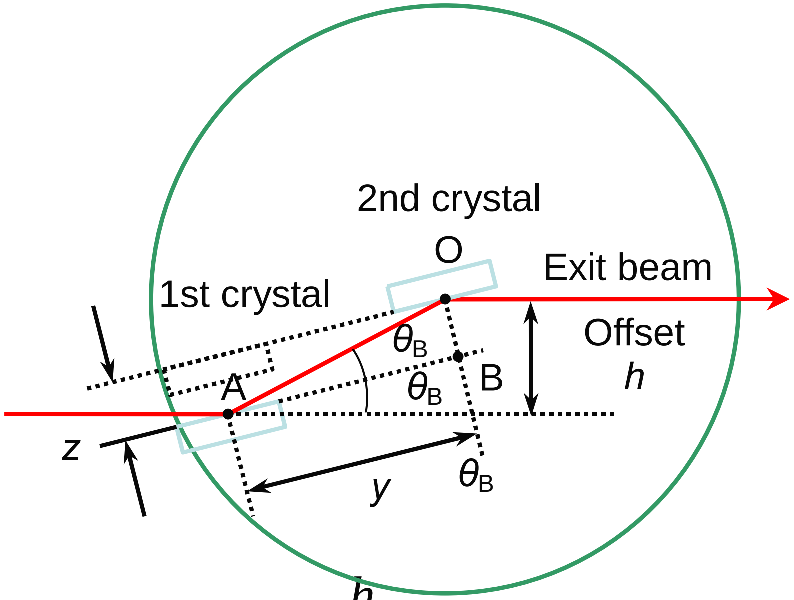
Fixed-exit DCM

(1) θ_1 + translation + θ_2 link



$$y = \frac{h}{\tan 2\theta_B}$$

(2) θ + two translation link

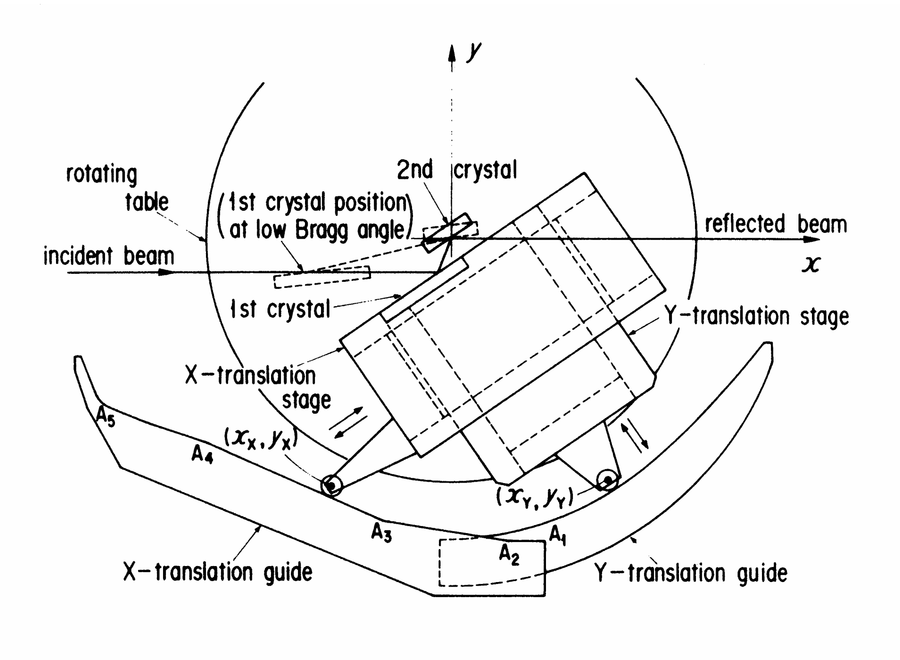


$$z = \mathrm{OB} = \frac{h}{2\cos\theta_B} \qquad y = \mathrm{AB} = \frac{h}{2\sin\theta_B}$$

$$(y^2 - h^2/4)(z^2 - h^2/4) = h^4/16$$

A number of different schemes have been developed to realize a fixed-exit beam.

θ + two translation (KEK-PF)



PF BL-4C..

Matsushita et al., NIM A246 (1986)

h= 25 mm, θ_B= 5～70°

Two cams for two translation-stages

Rotation center at 2nd crystal

Heat load on 1st Crystal

Heat load on the mochromator 1st crystal:

→ For SPring-8 bending magnet source
100 W & 1 W/mm^2

→ For SPring-8 standard undulator source
~500 W & ~ 500 W/mm^2

cf.	**Hot plate**	**: ~ 0.02 W/mm^2**
	CPU	**: ~ 0.3 W/mm^2**

Crystal cooling

Why crystal cooling？

Qin (Heat load by SR) = Qout (Cooling + Radiation,..)

→ with temperature rise ΔT

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

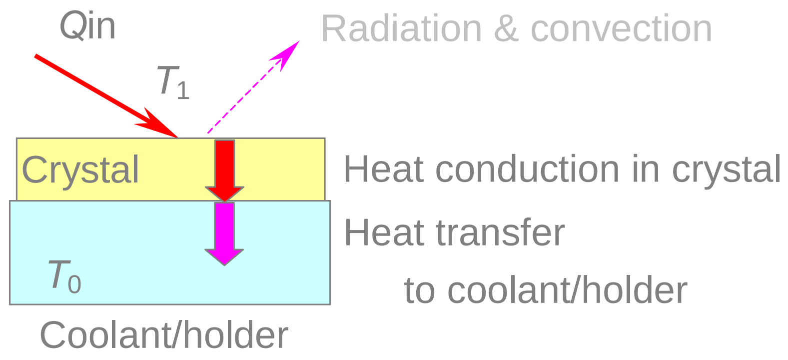
α: thermal expansion coefficient

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 → 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) κ / α → Larger

(S-2) Large contact area between crystal and coolant/holder

→ larger

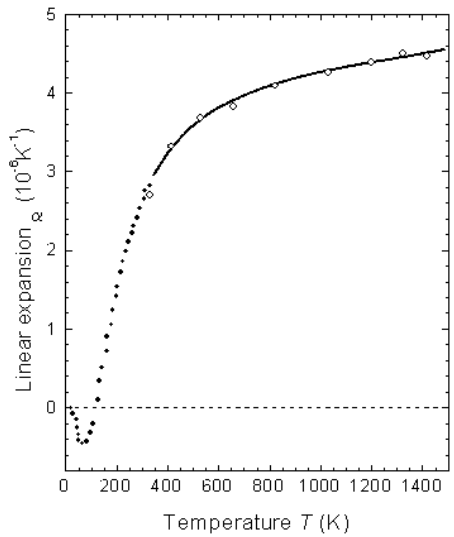
(S-3) Irradiation area → Larger, and power density → smaller

Figure of merit

	Silicon 300 K	Silicon 80 K	Diamond 300 K	Copper 300 K
κ (W/m/K)	150	1000	2000	401
α (1/K)	2.5×10^{-6}	-5×10^{-7}	1×10^{-6}	16.5×10^{-6}
κ / α $\times10^{6}$	60	2000	2000	24

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

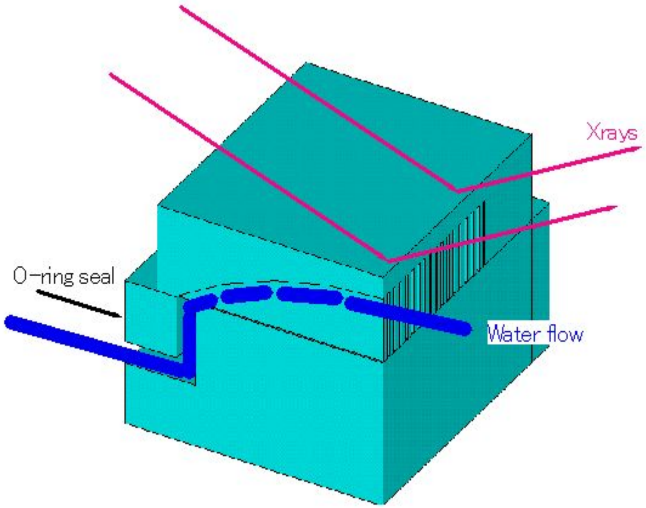
Liquid N_2 : 77.36° K (-195.79° K)

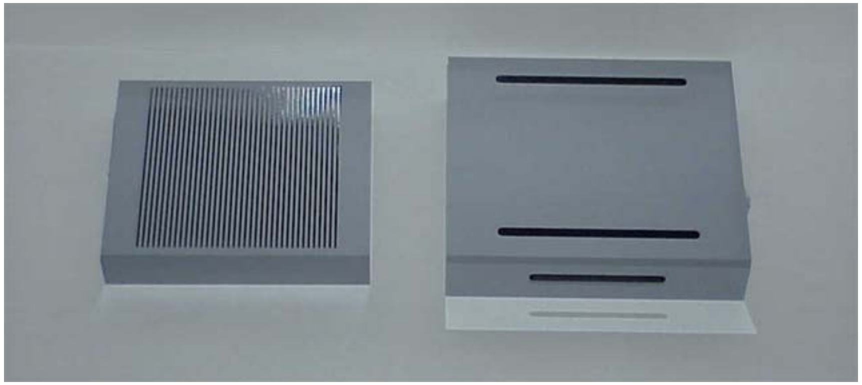


Direct cooling with fin crystal

Improvement of fin-cooling crystal

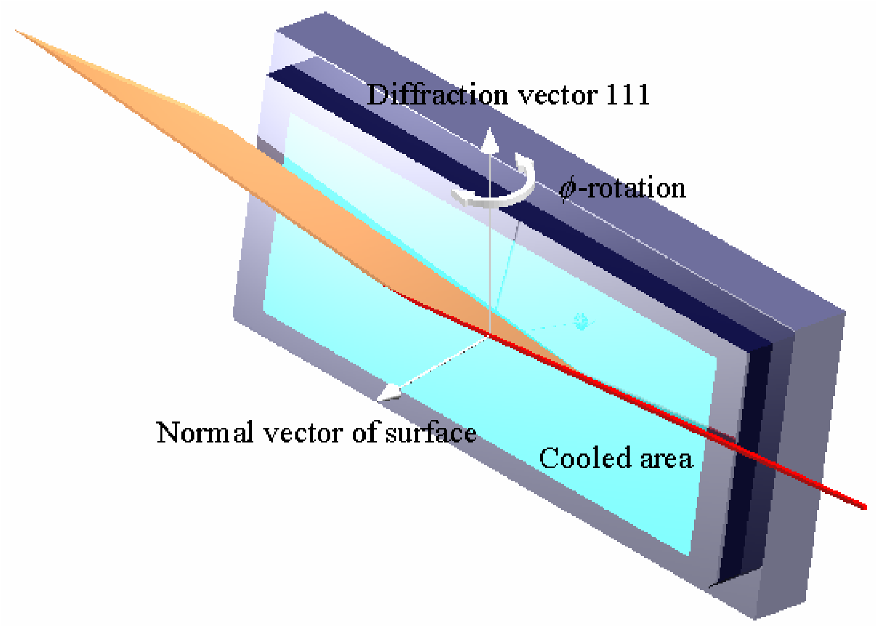
Reduce radiation damage of rubber O-ring

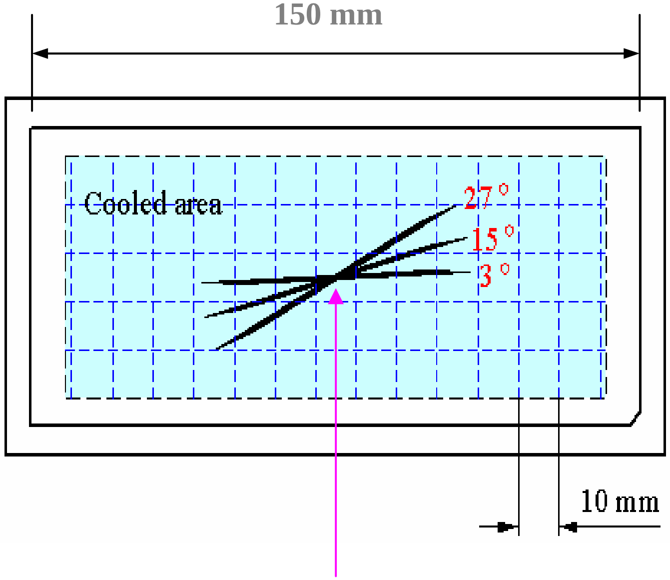




Au-Si eutectic bonding

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





Irradiation area

Inclination angle β= 80°

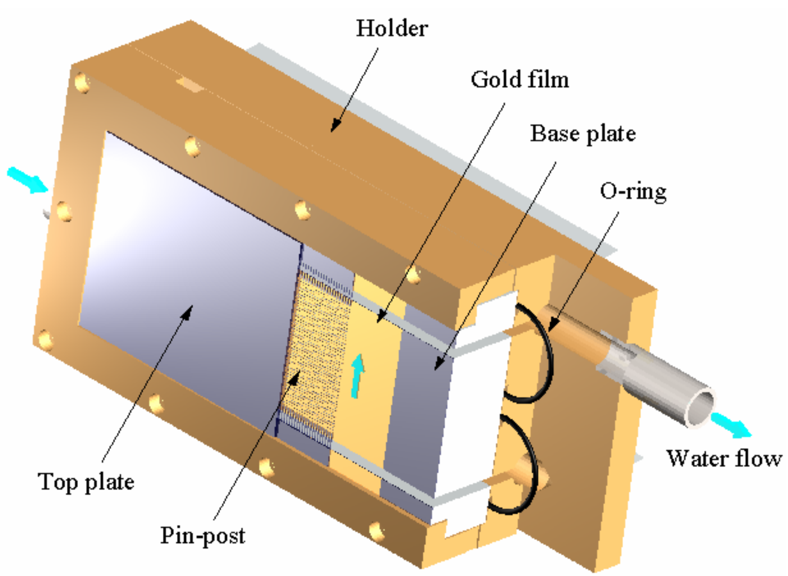
Grazing angle down to 1° using ϕ-rotation

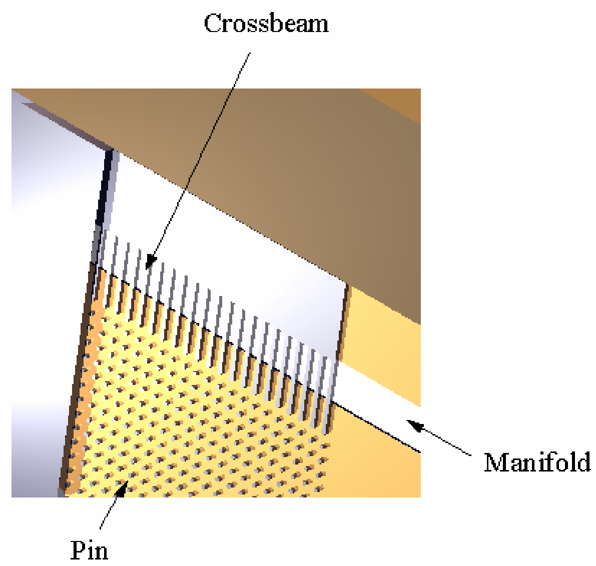
Irradiation area enlarged to x50, power density reduced to 1/50

Applied to undulator beamline

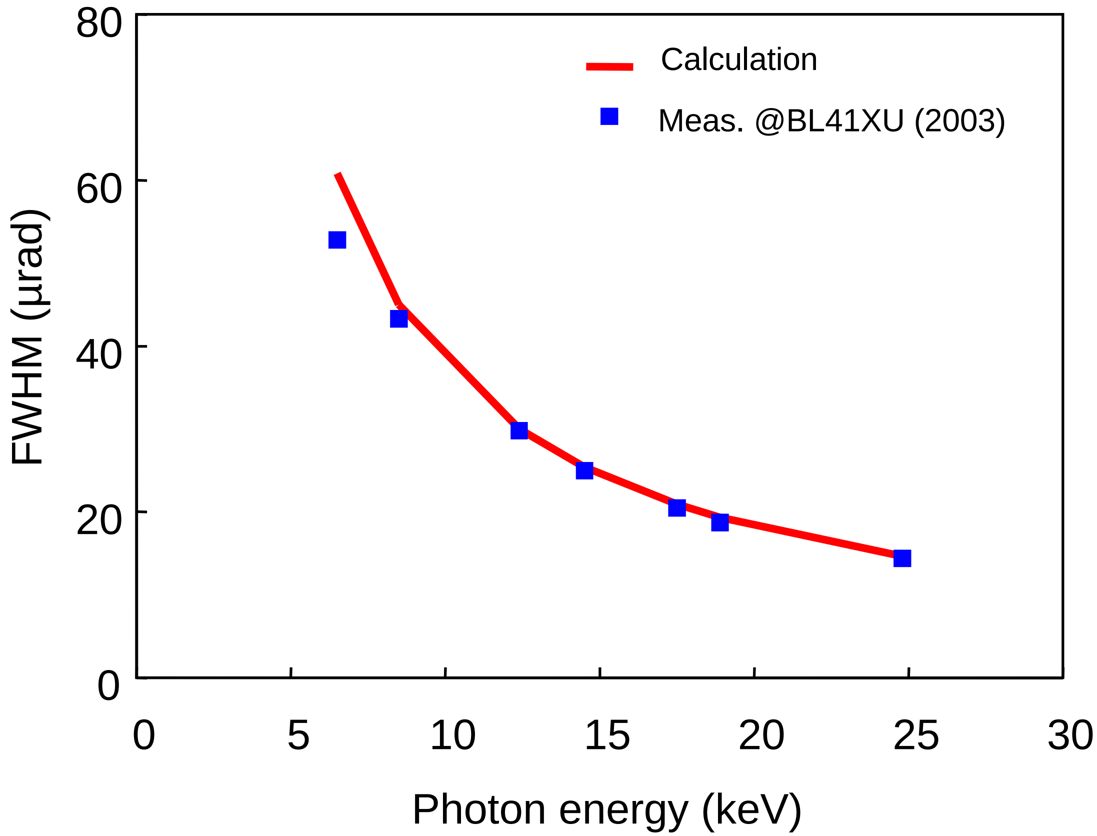
Structure of pin-post crystal

Top plate with pin-post is bonded to base plate (manifold) using Au-Si eutectic bonding.





Performance of pin-post crystal

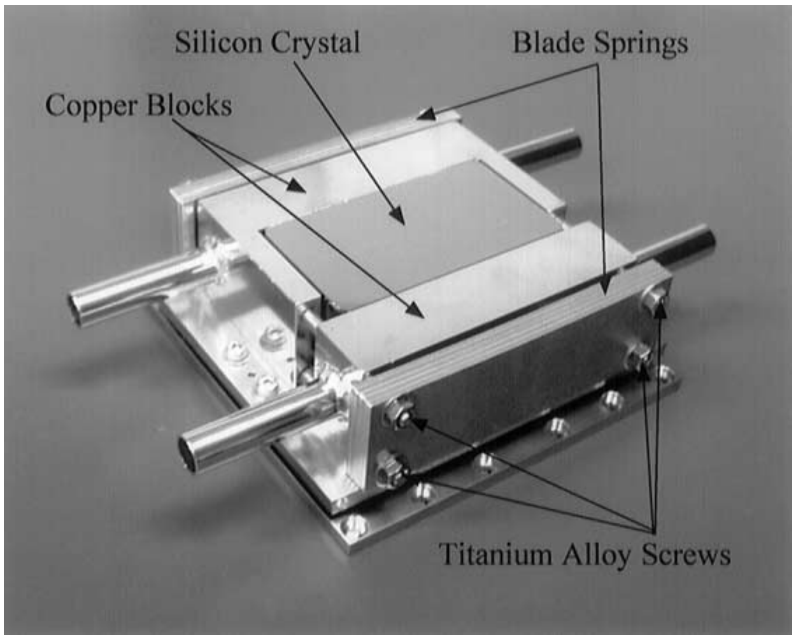


Rocking curve widths agree well.

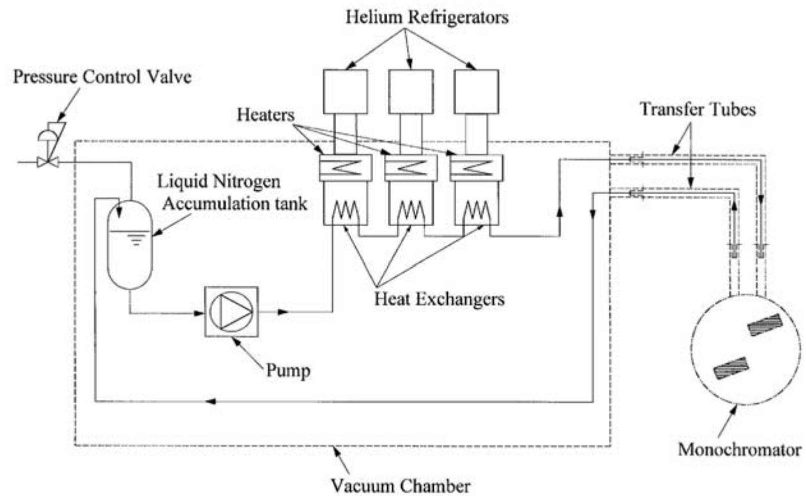
A rocking curve is obtained by rocking (rotating) the second crystal of the double crystal arrangement and recording the diffracted intensity.

Cryogenic cooling

Indirect side cooling



LN_2 circulator with He refrigerator



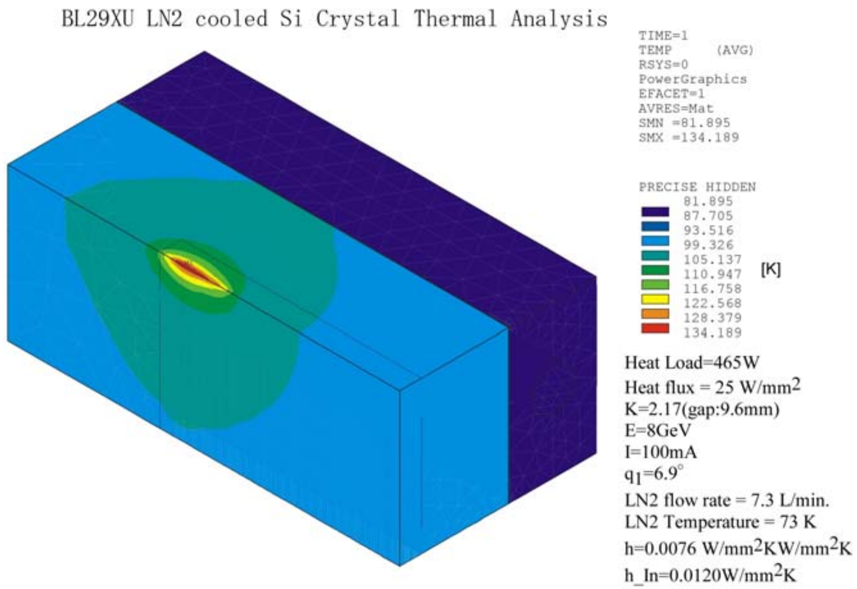
Applied to undulator beamline

Silicon, Liquid Nitrogen Cooling

465W, θ=6.9° 25 W/mm^2

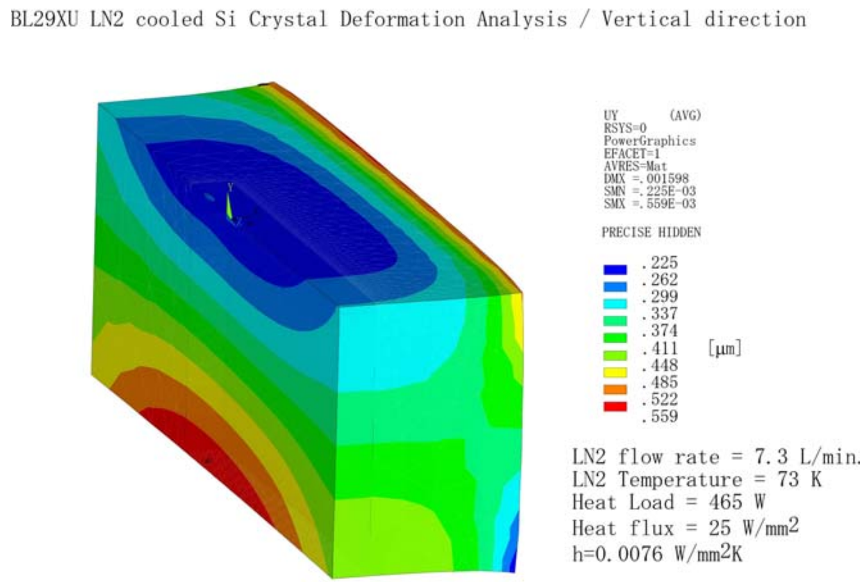
Spring-8 undulator beamline

Temperature distribution



prepared for SRI2000,T. Mochizuki et.al, Cryogenic cooling monochromators for the SPring-8 undulator beamlines,

Deformation distribution

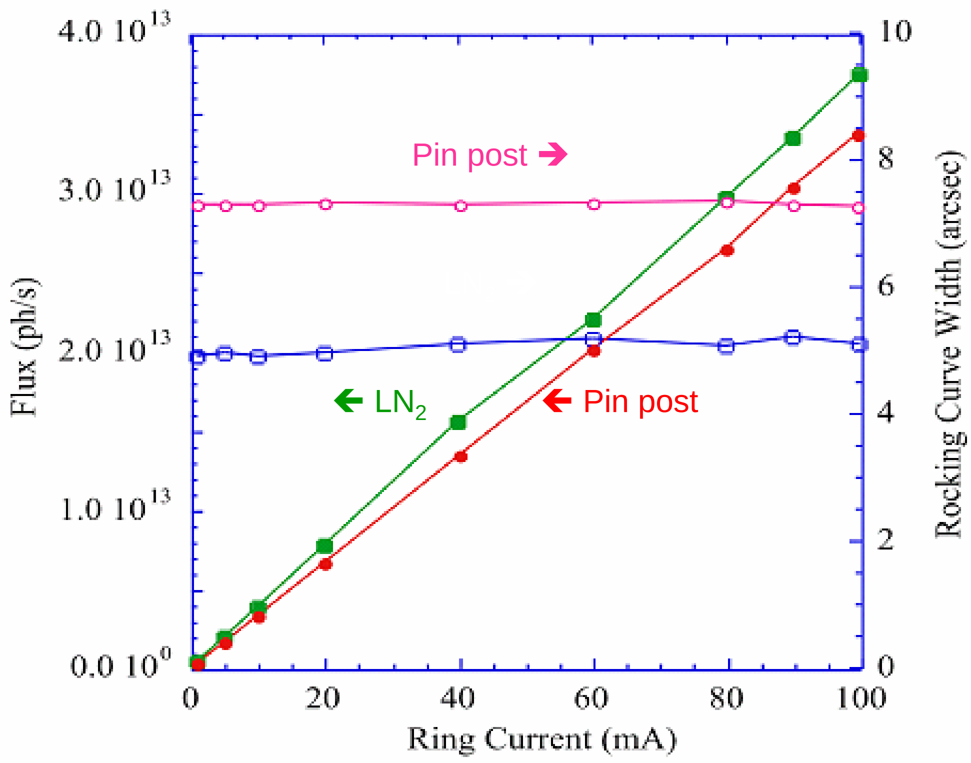


prepared for SRI2000,T. Mochizuki et.al, Cryogenic cooling monochromators for the SPring-8 undulator beamlines,

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:

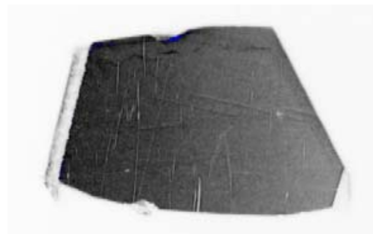
- Good thermal properties → Capability of indirect cooling
- Higher resolution (←→ Less throughput (30～40% of Si))

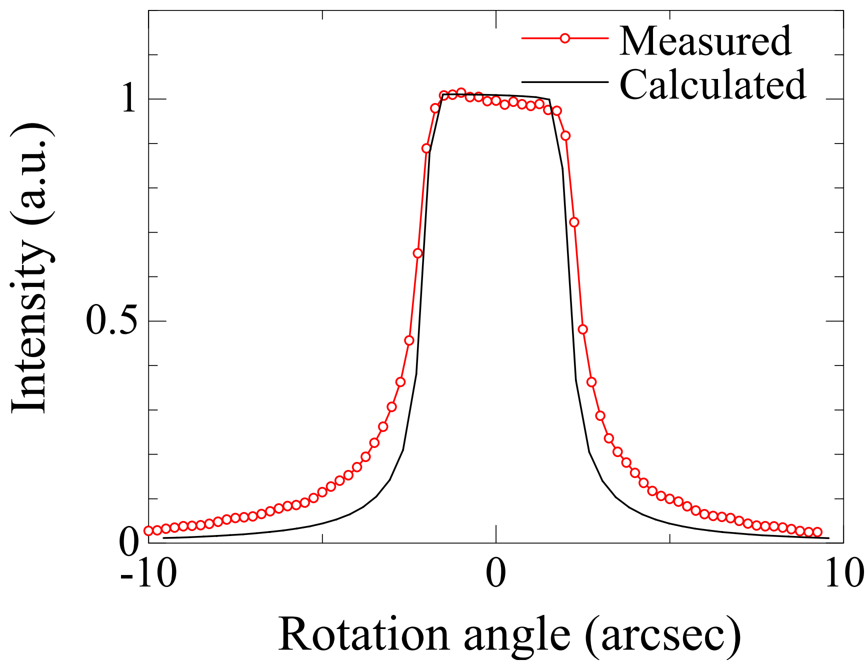
Issues:

- Perfection of crystal → HPHT IIa diamond (Sumitomo)…

  Successive upgrade is crucial !
- Holding of crystal → X-ray topograph, Zygo
- Optimization of thermal contact → New process with In insert
- Small crystal (< 10 mm□)
- Alignment: using CCD camera, PIN photodiode, thermocouple

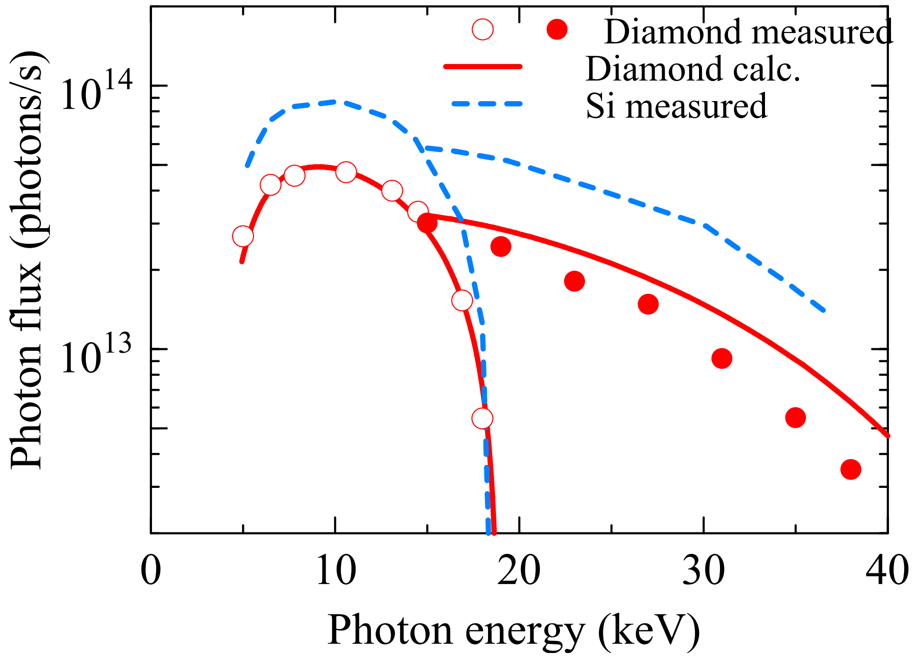
Topograph and rocking curve

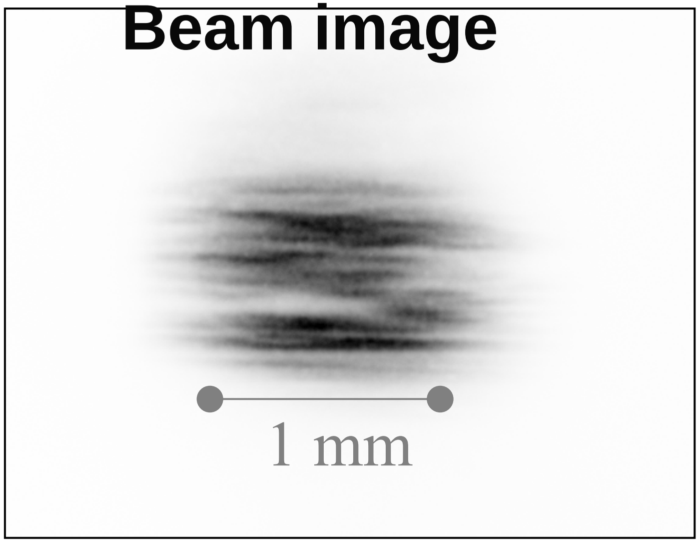




Characterization of diamond monochromator

Photon flux





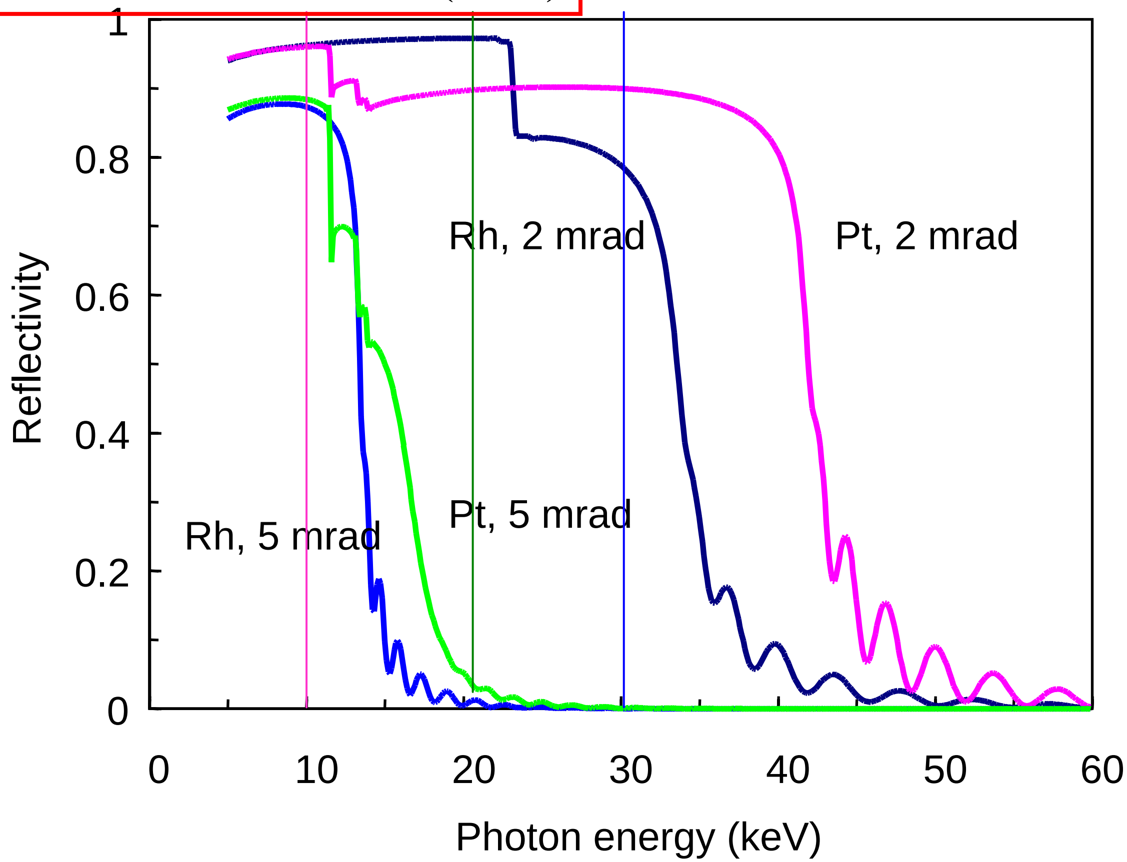
ε = 3 nm. rad, I_b = 100 mA
Front-end slit aperture = 0.7 (v) x 1.0 (h) mm^2

Low energy: 50~60% of Si DCM
High energy: Infinite size effect ?

Improvement of crystal growth and surface finish is needed.

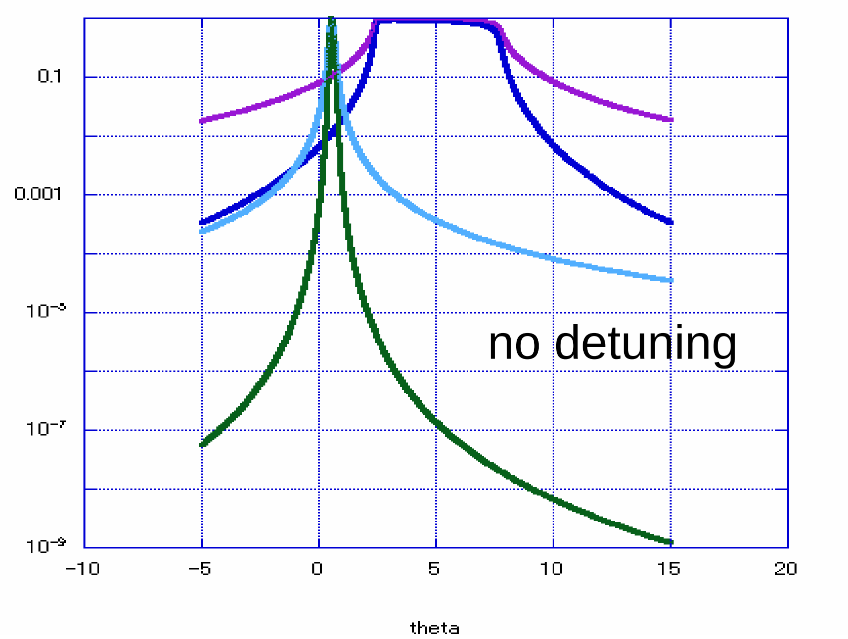
Higher harmonics rejection
- total reflection mirror -

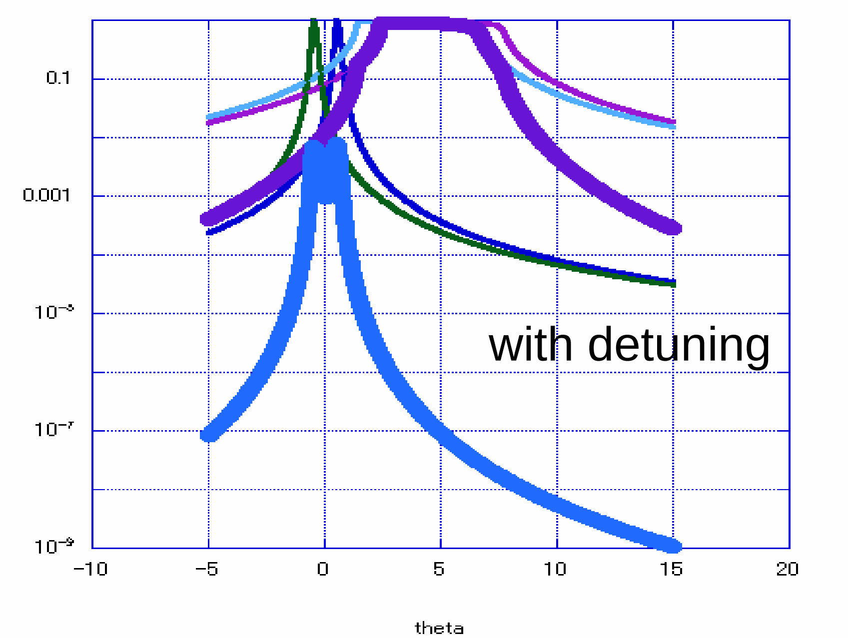
$$\boxed{2d(\text{Å})\sin(\theta) = n\lambda(\text{Å}) = n\frac{12.4}{E(keV)}}$$



Film thickness: 50 nm
Surface roughness: 1 nm

Higher harmonics rejection
- Detuning of DCM -



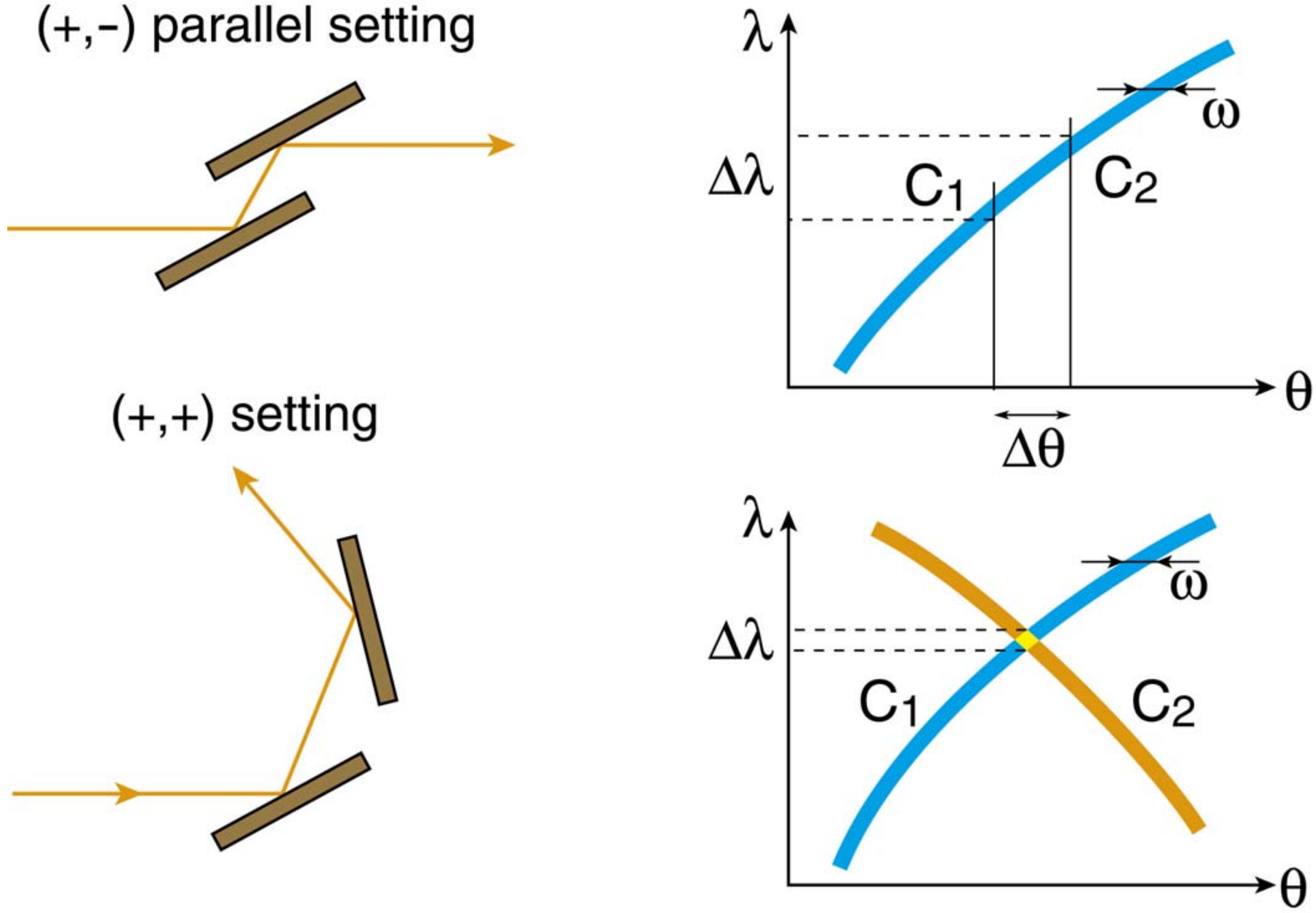


e.g. $\Delta\theta$ = 12 μrad
→ 70% of peak intensity for fundamental (111 refl. @ 10 keV)
→ 0.3% of peak for 3rd harmonics (333 refl. @30 keV)

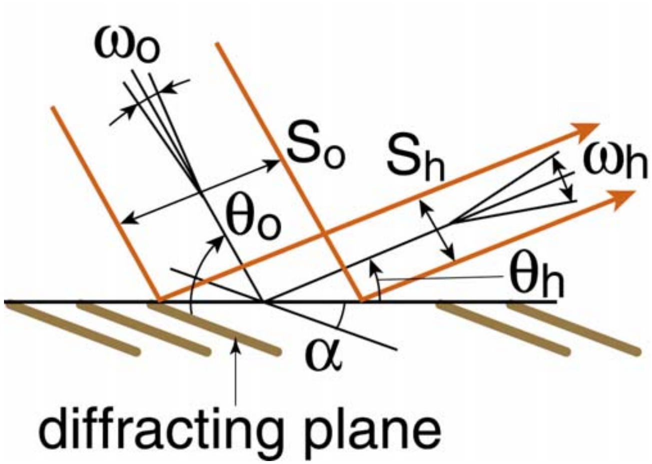
e.g. $\Delta\theta_2$= 10 μrad → Angle change of exit beam = $2\Delta\theta_2$= 20 μrad
Beam position change of 0.2 mm @10 m from DCM.
We should recognize the beam position change by DCM detuning!

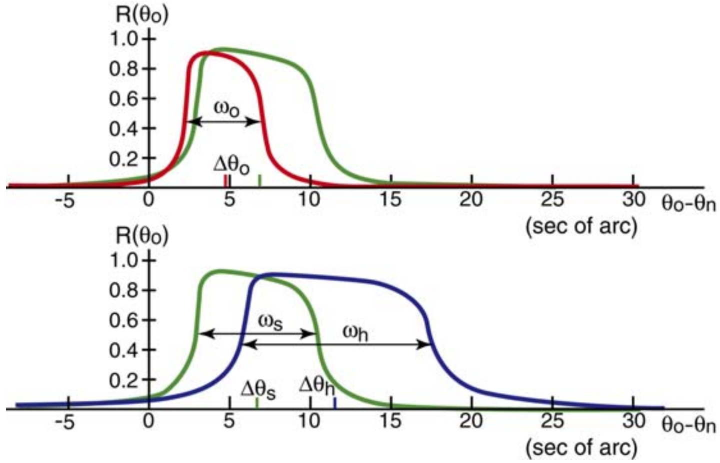
High resolution monochromator

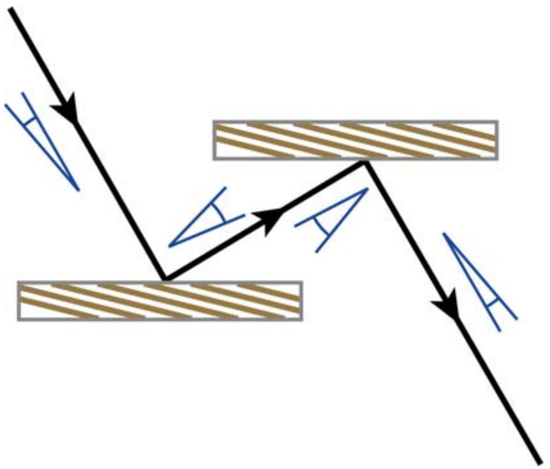
double crystal arrangements and the DuMond diagram

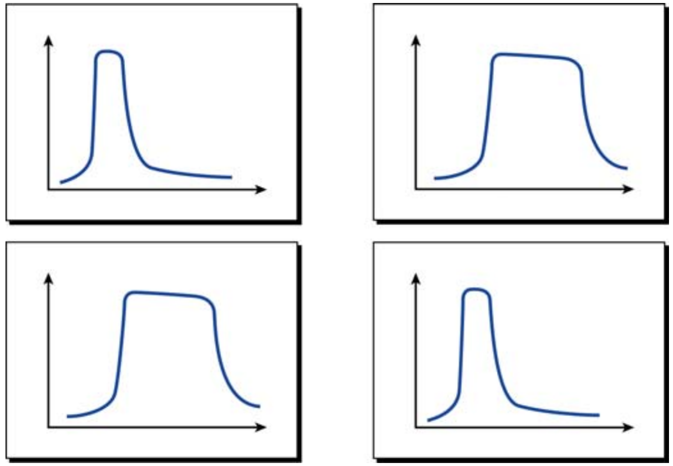


Asymmetric diffraction for high resolution monochromator

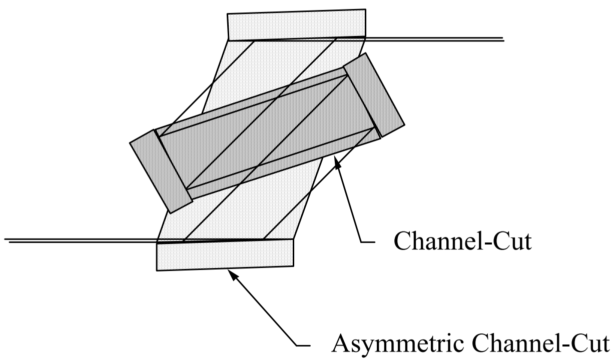


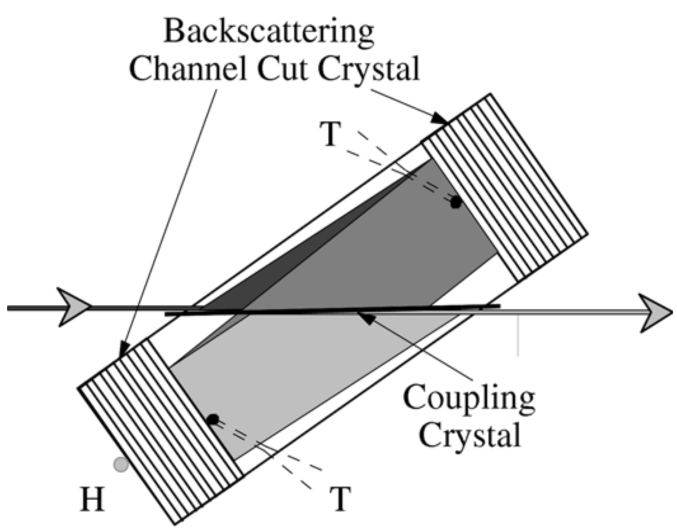






Extremely High-Resolution X-Ray Monochromators





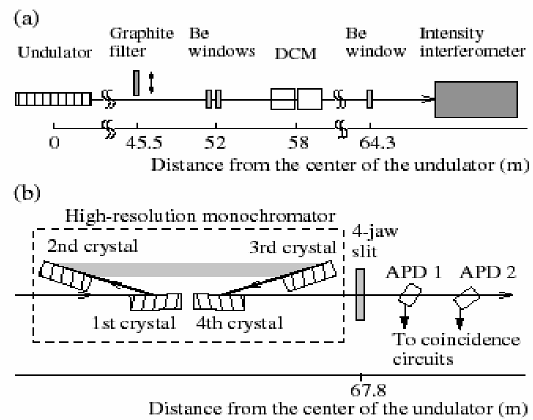
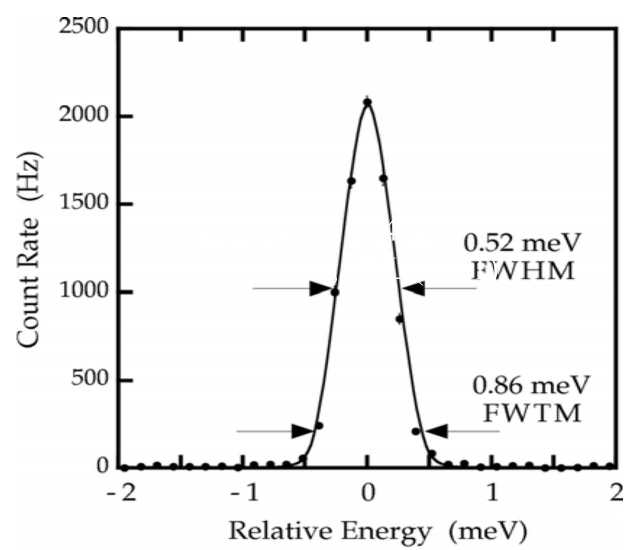


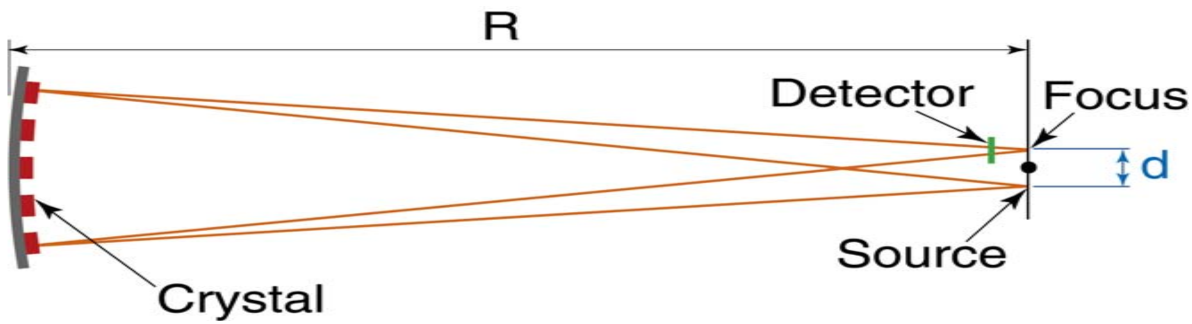
FIG. 1. Schematic top view of the experimental setup (a), and that of the intensity interferometer (b). The interferometer consists of a high-resolution monochromator using four separated crystals, a precision four-jaw slit, two semitransparent avalanche photodiodes (APDs), and coincidence circuits.



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

Spherical Diced Crystal Energy Analyzer

- meV resolution -



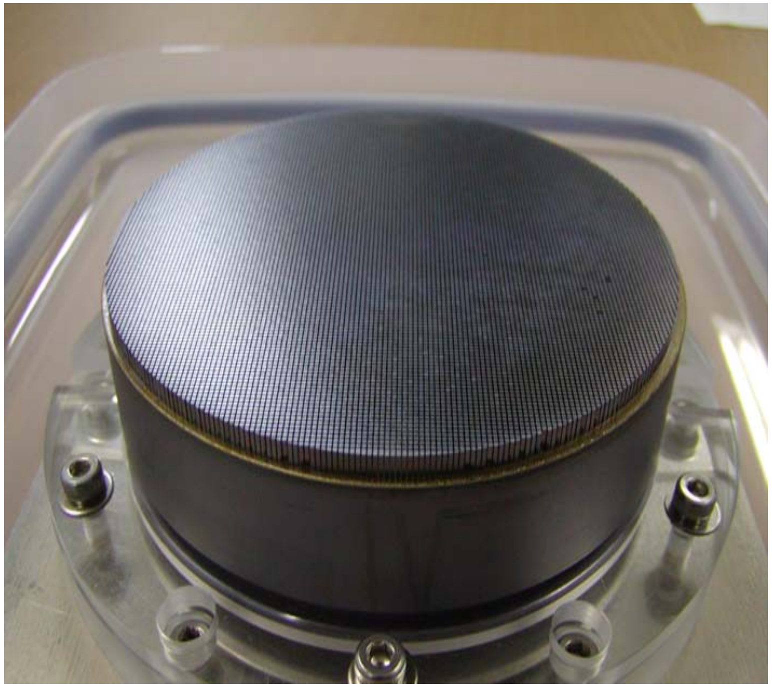
$$\frac{\Delta E}{E} = \sqrt{\omega^2 + \Delta\theta^2}\,\cot\theta$$

When $\theta = 89.97^\circ$

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

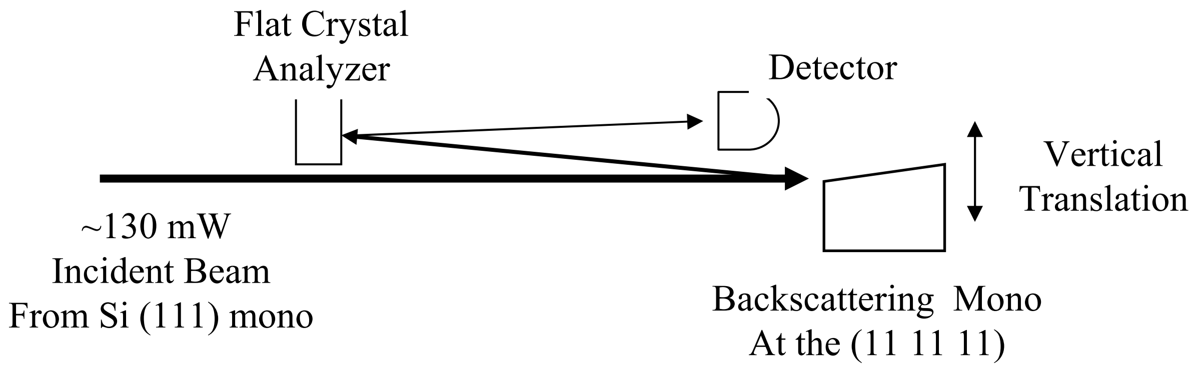
If $\sqrt{\omega^2 + \Delta\theta^2} \sim 10^{-3}$ -10^{-4}

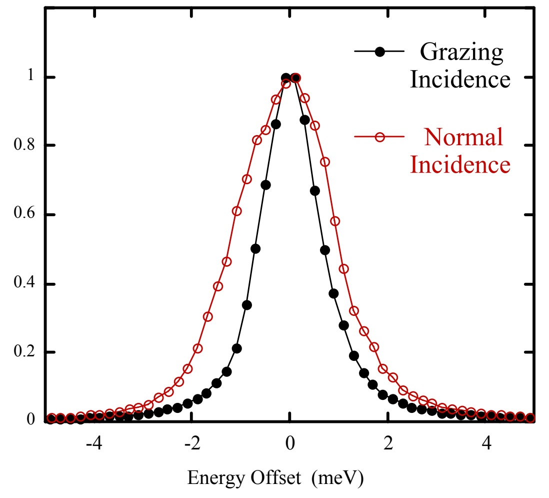
$\Delta E/E \sim 10^{-7} - 10^{-8}$



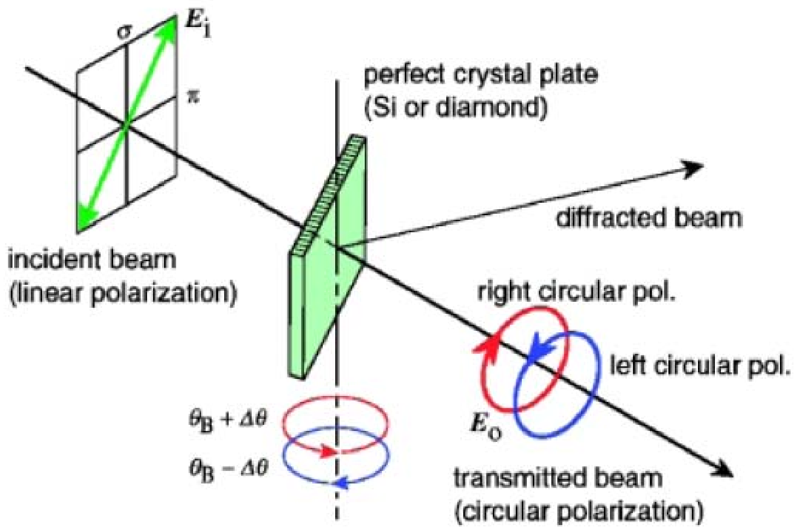
R=9.8 m Spherical Diced Crystal Analyzer

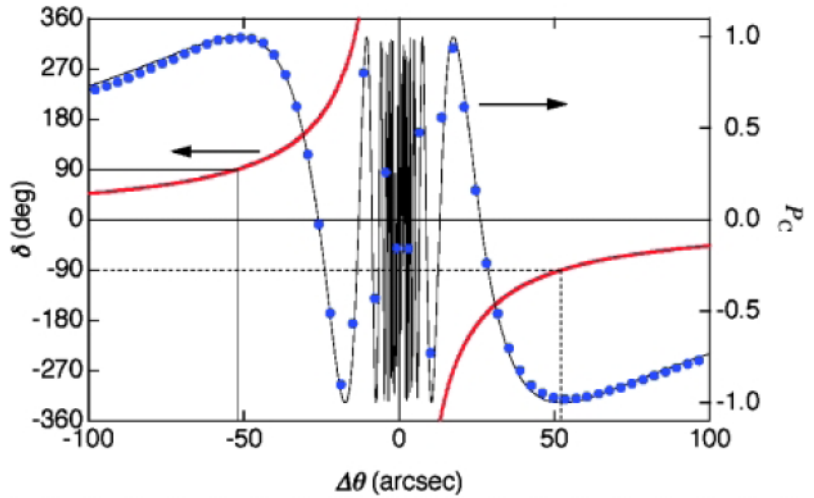
Power Load Effects on Backscattering Monochromator





phase retarder, polarization conversion
- transmission through a thin crystal -



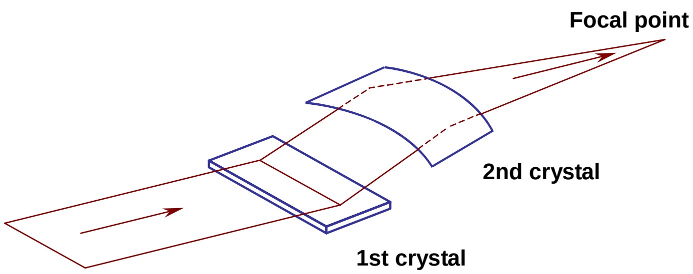


Concept of dispersion surfaces should be utilized to understand.

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

Sagittal focusing

Principle of sagittal focusing



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

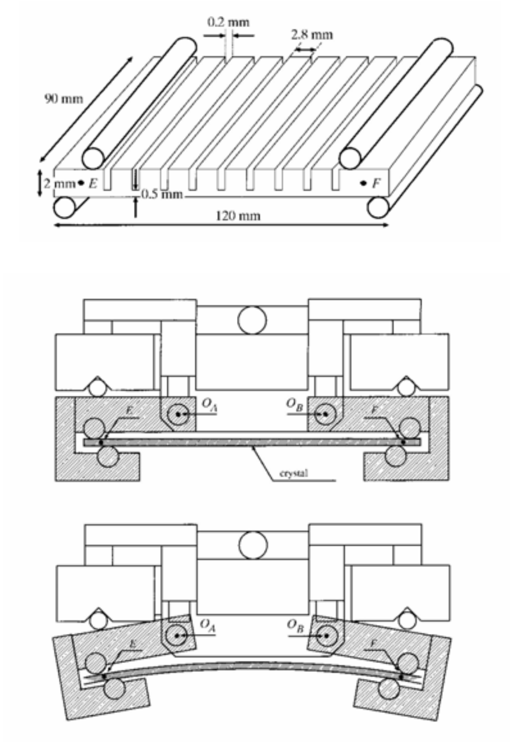
r: radius of 2nd crystal

θ: Bragg angle

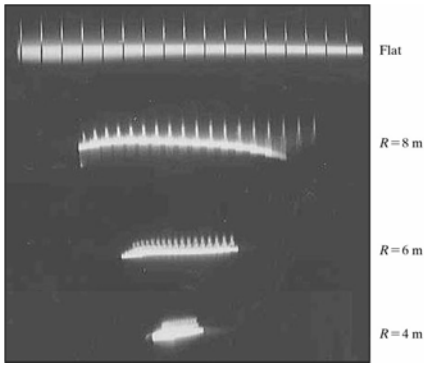
p: source ~ crystal distance

q: crystal ~ focal point distance

Bending mechanism for SPring-8 sagittal focusing



e.g. Sagittal focusing images



50 mm

Si 311 refl.

40 keV

Source～Crystal= 36.5 m

Crystal～focal point= 16.5 m

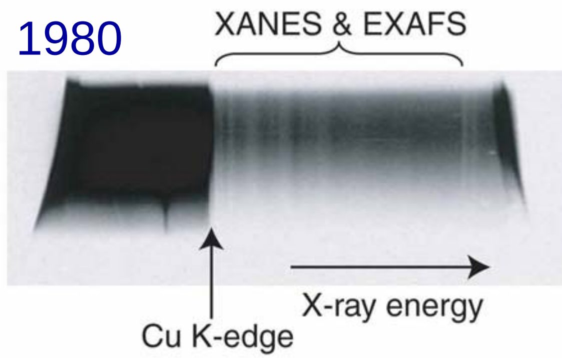
Applied for bending magnet beamline

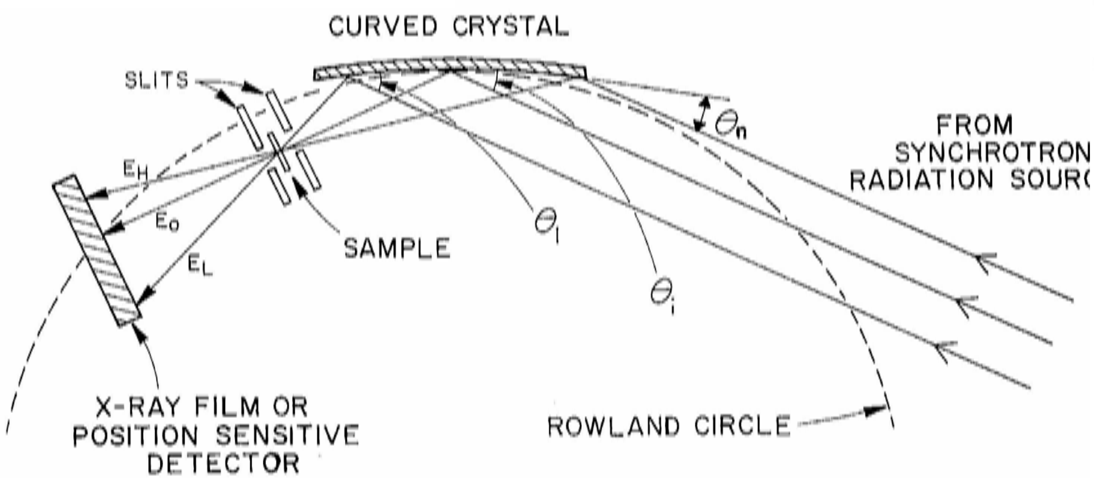
Dispersive X-ray Absorption Spectroscopy

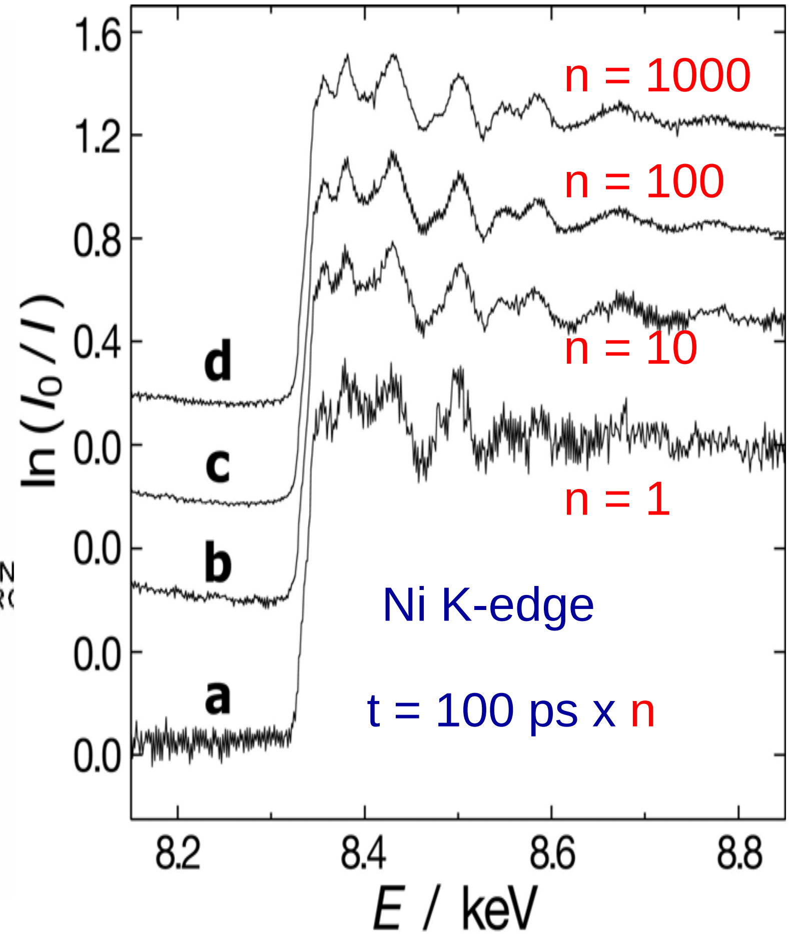
T. Matsushita & R. P. Phizackerley:
Jpn. J. Appl. Phys. 20, 2223-2228 (1981)

R = 100~300 cm
E_H-E_L = ~ 1 keV

1980







稲田、丹羽、野村：放射光、20, 242 (2007)

Multiwavelength Dispersive X-Ray Reflectometry

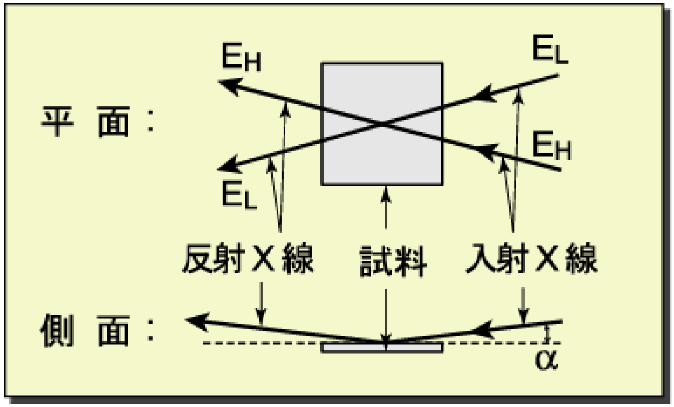
$$Q = 4\pi \sin(\alpha)/\lambda$$

$$\sim E \sin(\alpha)$$

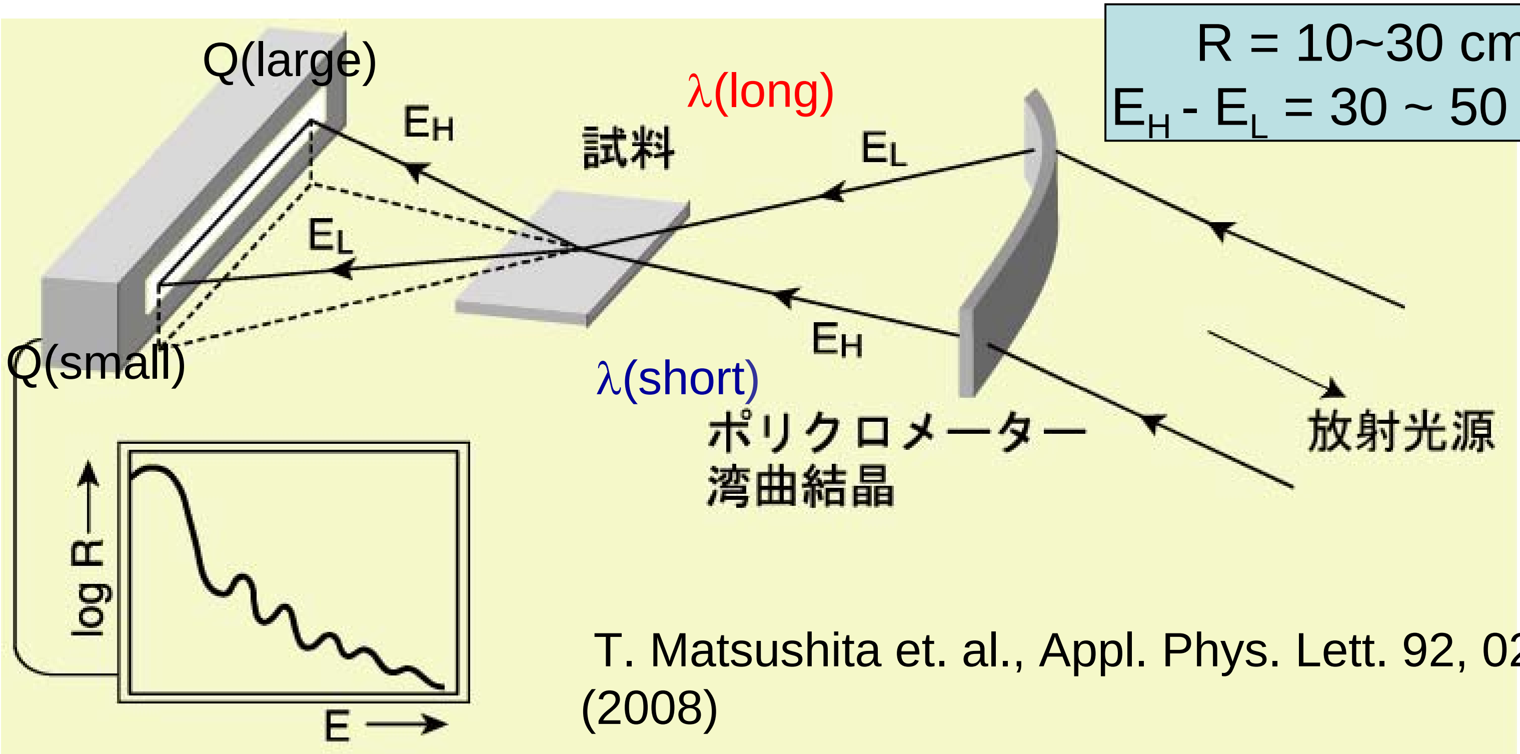
Q: 0.08~0.8 Å^{-1}

$Q_{max}/Q_{min}=10$

$E_{max}/E_{min}=10$



R = 10~30 cm
$E_H - E_L$ = 30 ~ 50 keV



T. Matsushita et. al., Appl. Phys. Lett. 92, 024103 (2008)

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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