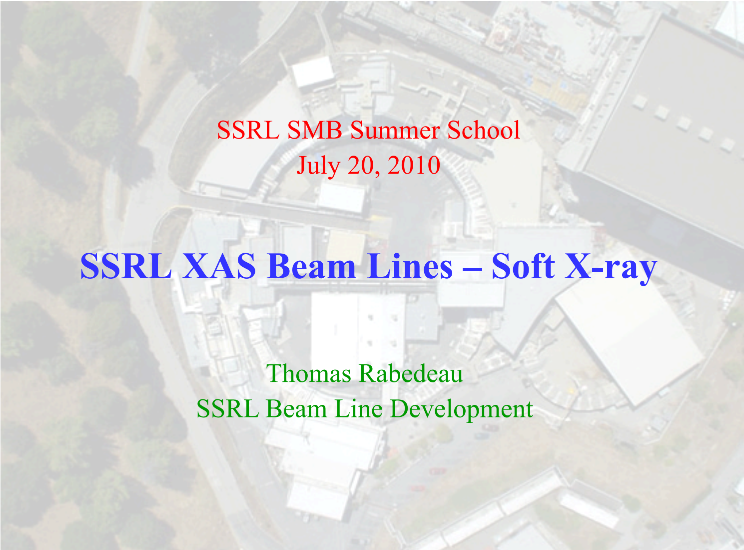


SSRL SMB Summer School
July 20, 2010

SSRL XAS Beam Lines – Soft X-ray

Thomas Rabedeau
SSRL Beam Line Development

Objective/Scope

Objective -

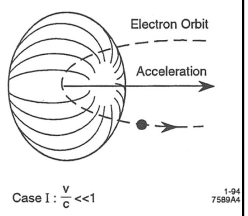
- *develop a better understanding of the capabilities and limitations of SSRL soft x-ray beam lines to facilitate quality XAS data collection*

Scope –

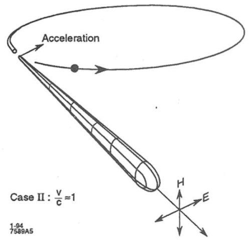
- *lightening review of synchrotron source characteristics*
- *relationship between the source characteristics and sample requirements*
- *x-ray mirrors*
- *crystal monochromators*
- *practical application on SSRL beam lines*

Synchrotron Radiation
Emission Cone

Electrons in circular motion are also undergoing acceleration



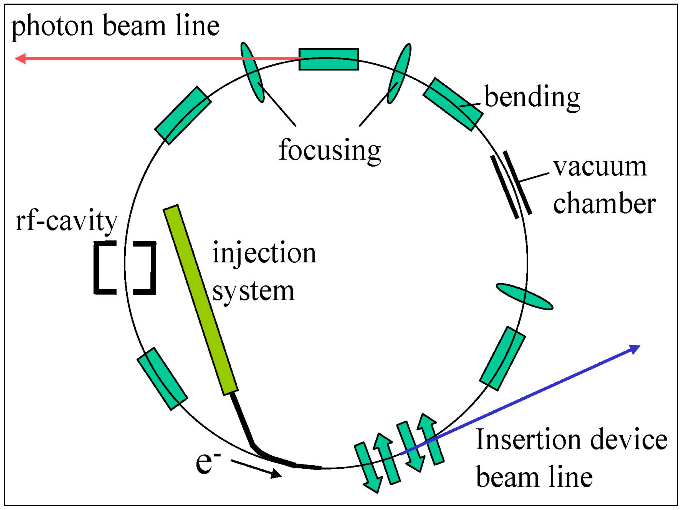
When electrons are moving slowly the radiation comes out in ***doughnut pattern***



When the electron speed gets close to the speed of light, the radiation comes out only in a narrow forward cone; ***a laser-like concentrated stream***

- In SPEAR electron speed is 99.999999% speed of light
- Radiated power increases at higher velocities; spectrum Doppler shifted
- Radiation collimated, opening angle = 1/γ ($\gamma = E/m_0c^2 = 5871$).

Synchrotron Radiation
Synchrotron Ring

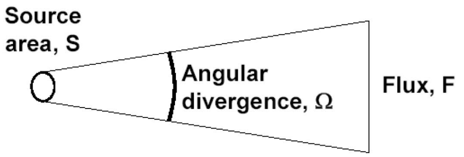


Synchrotron Radiation
Flux and Brightness

$$\textbf{Flux} = \frac{\#\text{ of photons in given } \Delta\lambda/\lambda}{\text{sec}}$$

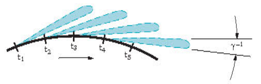
$$\textbf{Brightness} = \frac{\#\text{ of photons in given } \Delta\lambda/\lambda}{\text{sec, mrad } \theta\text{, mrad } \varphi\text{, mm}^2}$$

(a measure of concentration of the radiation)

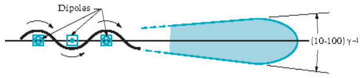


$$\textbf{Brightness} = \textbf{constant} \times \frac{\textbf{F}}{\textbf{S} \times \Omega}$$

Synchrotron Radiation
Bends, Wigglers, Undulators



bending magnets - a "sweeping searchlight", BLs 1, 2, 8, 14 e_c= 7.78 keV



wiggler - incoherent superposition of radiation from an array of magnet poles
BL6 ε_c= 5.39 keV, BL7 ε_c= 12.2 keV

undulator - coherent interference of radiation from an array of magnet poles

bend magnets & wigglers

- continuous spectrum with half-power point "critical energy"

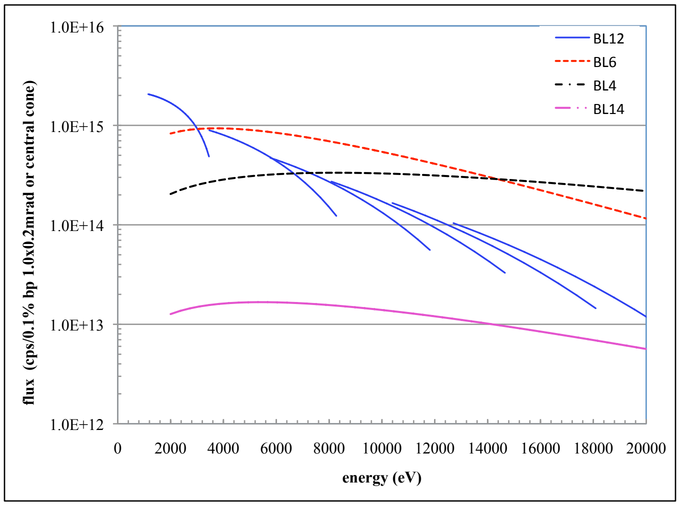
ε_c(keV) = 0.665*B(T)E^2(GeV)

- intensity ~ N_{poles}
- broad horizontal fan

undulator

- quasi-monochromatic spectrum consisting of fundamental and higher harmonics
- intensity ~ $(N_{poles})^2$
- narrow horizontal emission cone

Synchrotron Radiation
Flux of Typical SSRL Sources



Source Characteristics & Sample Requirements
Two Sides of the Same Coin?

source (beam) characteristics:

- *size (x, y)*
- *angular divergence (x', y')*
- *energy content*
- *stability*
- *polarization*
- *time domain*
- *coherence*

sample (beam) requirements:

- *focus size (x, y)*
- *angular convergence (x', y')*
- *energy content*
- *stability*
- *polarization*
- *time domain*
- *coherence*

The job of x-ray optics is to transform the source beam characteristics to provide the best possible match to the sample requirements.

For most of the SMB experiments conducted at SSRL the first four characteristics listed are the central concern, so this talk with concentrate on how optics can manipulate these characteristics to best advantage.

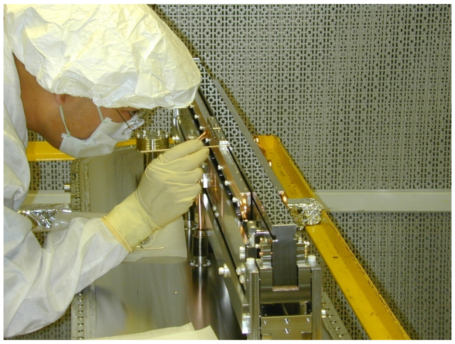
SSRL Source Characteristics
Current Generation of SSRL SMB Facilities

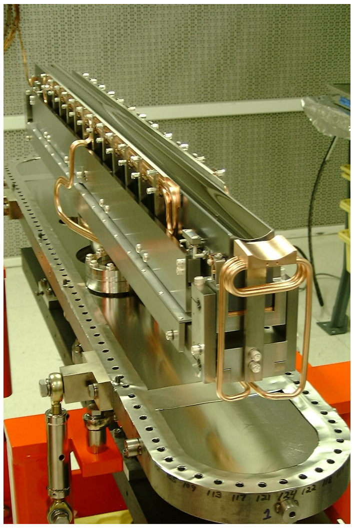
- *source size (typical excluding undulator BL12-2)*
  - *typical ID 311um x 8.1um rms (730um x 19um fwhm)*
  - *bend 141um x 13.8um rms (332um x 32.5um fwhm)*
- *angular divergence (typical excluding undulator BL12-2)*
  - *horizontal divergence limited by slits to 1-3mrad typical*
  - *vertical divergence is energy dependent - typical x-ray divergence ~110urad rms (250urad fwhm)*
- *broad (white) energy content*
- *stability - ~20um horz x ~5um vert (rms over hour time scales)*
- *polarization – dominantly horizontal*
- *time domain – fast pulsed (~250MHz)*
- *coherence – very slight*

X-ray Mirrors
Function

- focusing
  - *condense beam to source dimensions on sample (1:1 focusing)*
  - *demagnify source image to better couple photons on small sample at the expense of greater angular convergence on sample (n:1 demagnification results in n-fold convergence)*
- collimation
  - *collimate divergent beam to improve energy resolution of a crystal monochromator as discussed below*
- power filter
  - *absorb waste power at low power density on grazing incident optic rather than high power density on crystal monochromator*
- harmonic filter
  - *suppress higher energy contamination of beam (low pass filter)*

X-ray Mirrors



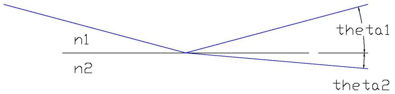


above: 1.0m Si flat, side-cooled M0 mirror employed for beam collimation

right: 1.2m Si cylindrical, side-cooled, M0 mirror employed for beam focus

---

X-ray Mirrors
Reflectivity at Grazing Angles

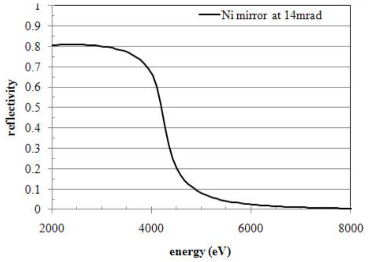


refractive index

$$n = 1 - r_0 \rho \lambda^2 / 2\pi - i \mu\lambda / 4\pi$$

where

- r_0 *is classical* e^- *radius (2.82e-13cm)*
- ρ *is electron density*
- μ *is linear absorption coefficient*

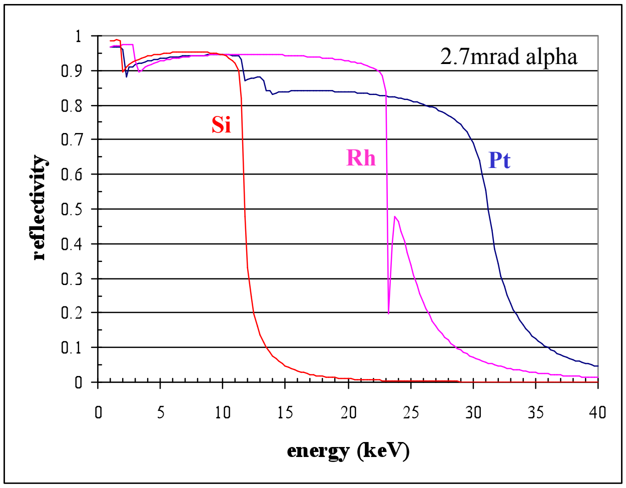


By Snell's law [$n_1 \cos(\theta_1) = n_2 \cos(\theta_2)$ *with* θ *the grazing angle] in the absence of absorption, we find total external reflection for angles less than*

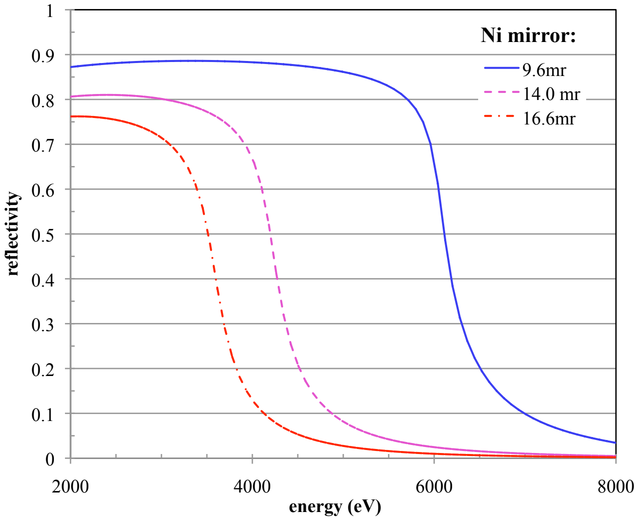
$$\theta_c \approx \lambda(r_0 \rho / \pi)^{1/2}$$

θ_c *is typically a few mrad for x-ray mirrors. As a consequence x-ray mirrors tend to be quite long. For example, a 250urad fwhm beam intercepted at 15m by a mirror at 3mrad results in 1250mm beam footprint.*

X-ray Mirrors
Reflectivity vs. Composition



X-ray Mirrors
Reflectivity vs. Angle



X-ray Mirrors
Figure

X-ray mirrors are either polished or bent to obtain desired figure.

- *elliptical figure provides point to point focusing*
- *parabolic figure collimates beam from source point or focuses parallel beam to a point*
- *focusing equations*

  $R_{tangential} = 2 F_{in} F_{out} / (F_{in} + F_{out}) \alpha$

  $R_{sagittal} = 2 F_{in} F_{out} \alpha / (F_{in} + F_{out})$

Most SMB BL mirrors at SSRL fall into two classes:

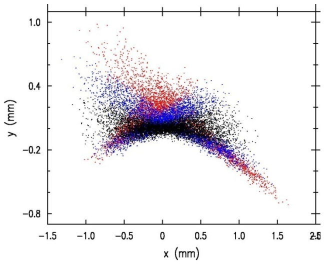
- *polished flats bent to approximate an ellipse or parabola to provide one dimensional beam shaping*
- *polished cylinders bent into a toroidal figure to provide two dimensional beam shaping*

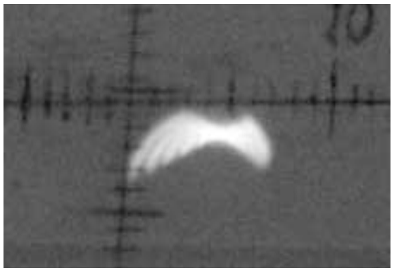
Typical radii of curvature:

- $R_{tangential}$ *= 2-8 km*
- $R_{sagittal}$ *= 35-100 mm*

X-ray Mirrors
Non-idealities

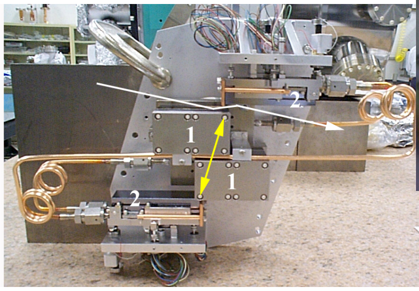
- grazing incidence optics introduce focus aberrations particularly when used to focus in horizontal and vertical planes simultaneously (function of accept.)
- toroidal mirrors located upstream of a crystal monochromator can significantly limit the energy resolution of the mono as discussed below (eg., BL10-2 or BL4-2 discussed below)
- mirror polish errors introduce focus blowup (eg., 2ur rms error on mirror 15m from focus broadens beam 60um rms)
- absorbed power can distort mirror surface resulting in focus degradation and time dependent focus changes
- beam stability crucially dependent upon mirror stability (eg., 1um differential motion at mirror ends can steer beam 20-30um at sample)

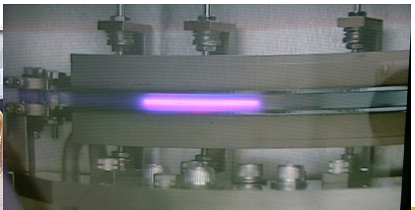


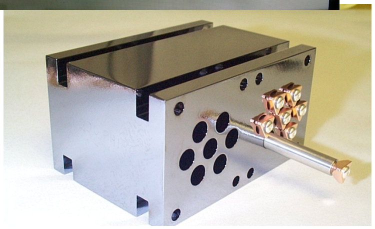


X-ray Monochromators
Function

- select a narrow energy band pass from the broad spectrum synchrotron source; typical crystal mono energy resolution ~1e-4 (or better) while multilayers (BL4-2) offer ~1% energy resolution





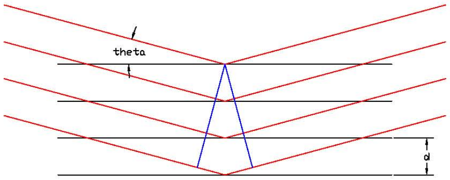


above left - LN mono crystal mount plate
above right – side scattering mono
lower right – LN mono first crystal with cooling channel bundle

---

X-ray Crystal Monochromators
Bragg Equation

- diffraction from a crystal is obtained when radiation scattered from successive atomic planes adds constructively (ie., $n\lambda$ path difference)
- Bragg Equation (real space)
  $2d \sin\theta = n\lambda$
- Bragg Equation (k space)
  $(4\pi / \lambda) \sin\theta = q$ where
  $q = (2\pi / a_0)(h^2 + k^2 + l^2)^{1/2}$
  a_0 is unit cell dimension &
  h, k, l are reciprocal lattice vectors



- consequence of the Bragg equation ($\varepsilon \sin\theta \sim q$) … crystal monochromators pass not only the fundamental energy of interest but also allowed higher order harmonics, so harmonic rejection becomes important function of optics

X-ray Crystal Monochromators
Energy Resolution

- *the function of the monochromator is to select a narrow energy band pass from the broad spectrum synchrotron source; typical energy resolution ~1e-4 (or better)*
- *energy resolution obtained by taking derivative of Bragg equation wrt θ, divide by Bragg eq., and rearrange terms …*

  $\delta\lambda / \lambda = \delta\,\theta / \tan\theta = \delta\varepsilon / \varepsilon$
- *better energy resolution obtained by using higher index reflections to obtain larger θ for a given energy*

  $\sin\theta = (\lambda / 2a_0)(h^2 + k^2 + l^2)^{1/2}$
- *so what contributes to δ θ?*
  - *beam divergence or convergence on monochromator*
  - *crystal rocking width or Darwin width*

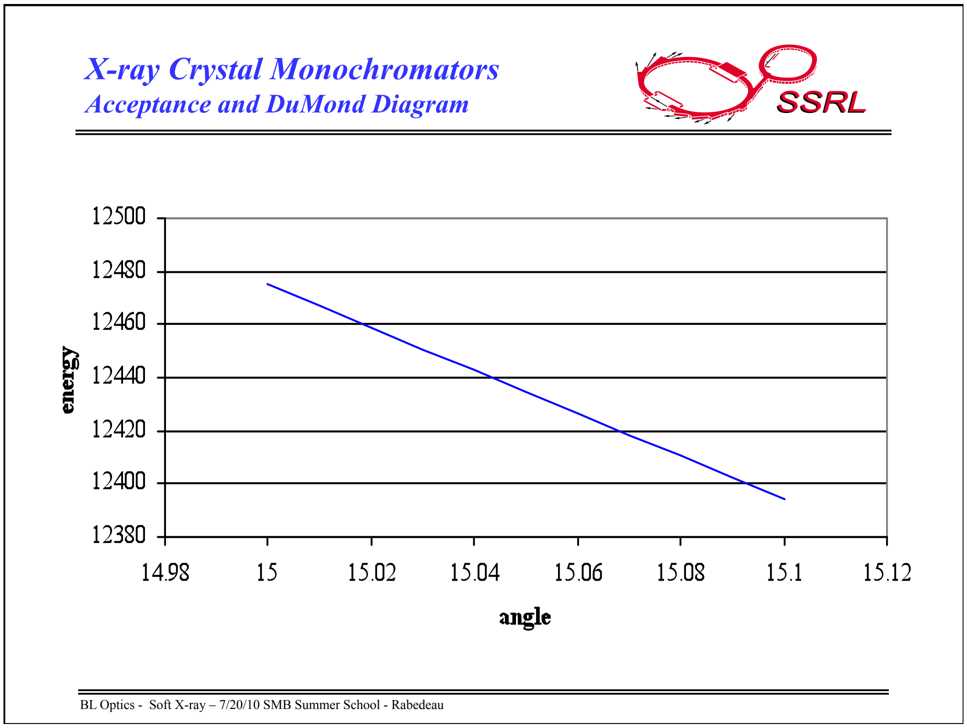
X-ray Crystal Monochromators
Darwin Width

- *simple Bragg picture of diffraction doesn't properly account for multiple reflections within crystal*
- *full dynamical theory of diffraction indicates Bragg diffraction occurs over a small range of angles (Darwin width)*

  $\delta\theta_{Darwin} \sim S_{hkl} / (\varepsilon^2 V \sin 2\theta)$

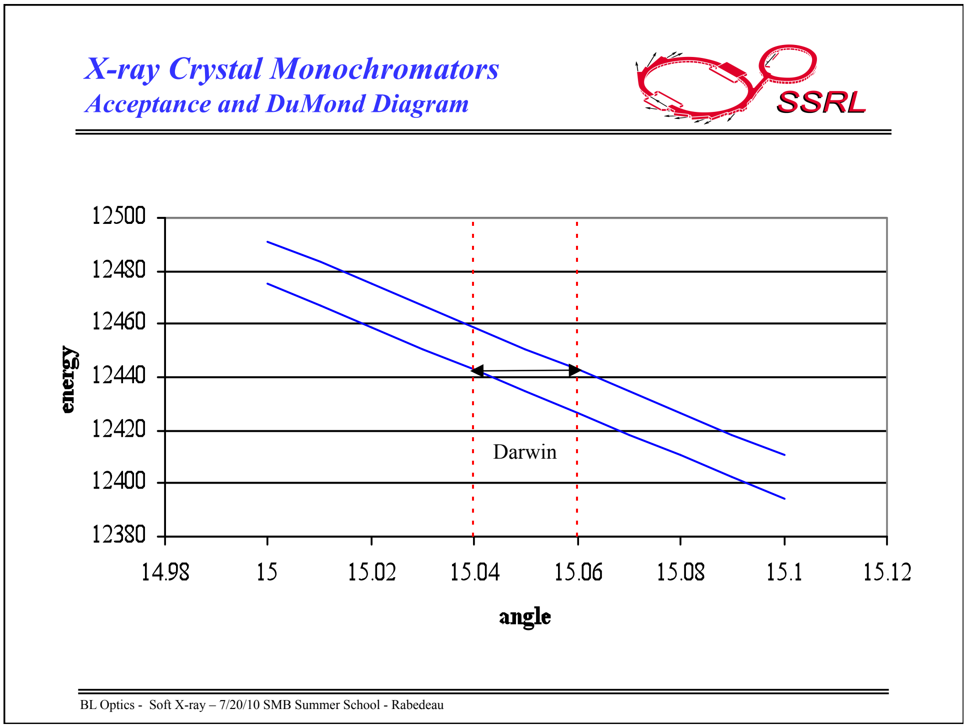
  where S_{hkl} is the Bragg peak structure factor and V is the unit cell volume
- *higher order peaks are narrower thus providing better energy resolution as seen in the following example for Si at 12keV*

index	theta (deg)	Darwin (urad)	δε/ε
220	15.607	15.99	5.72E-05
440	32.554	5.95	9.31E-06
660	53.817	3.27	2.39E-06



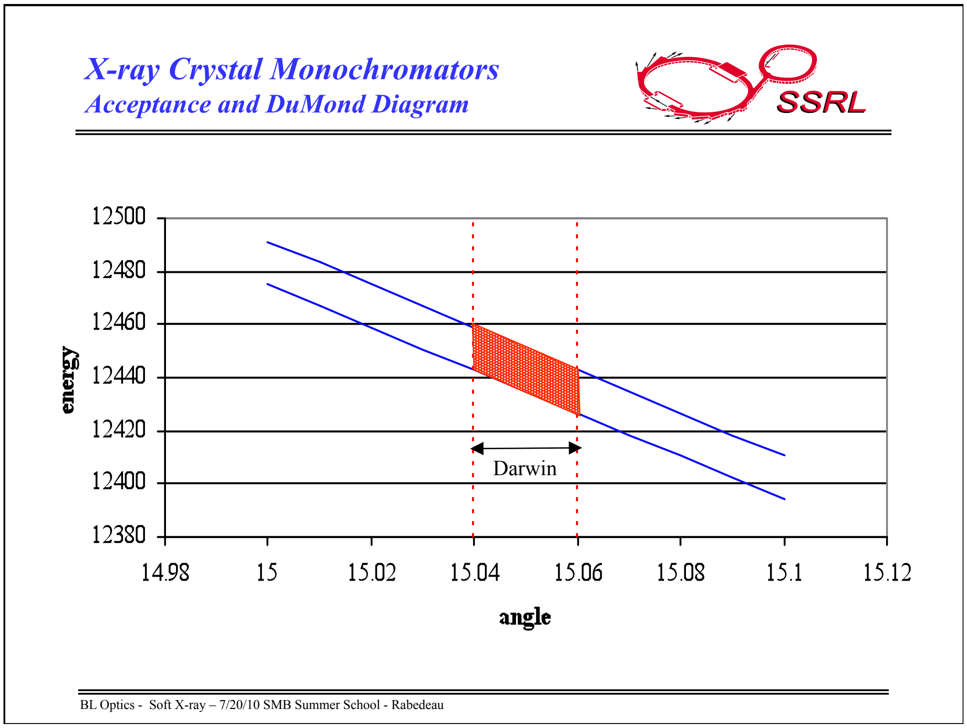
X-ray Crystal Monochromators

Acceptance and DuMond Diagram



X-ray Crystal Monochromators

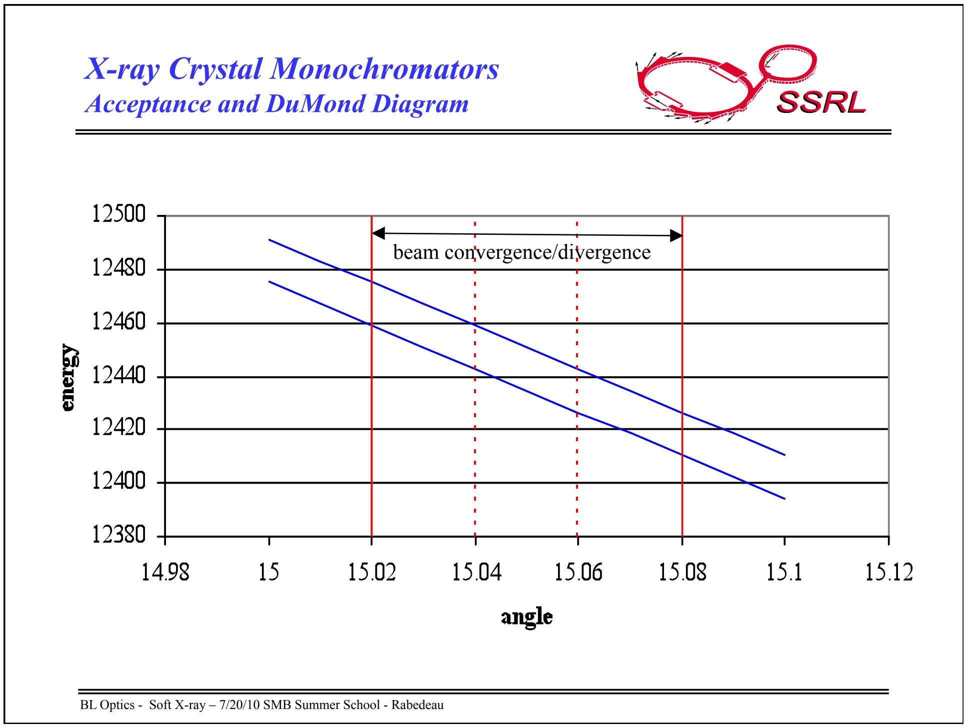
Acceptance and DuMond Diagram



X-ray Crystal Monochromators

Acceptance and DuMond Diagram

BL Optics - Soft X-ray – 7/20/10 SMB Summer School - Rabedeau

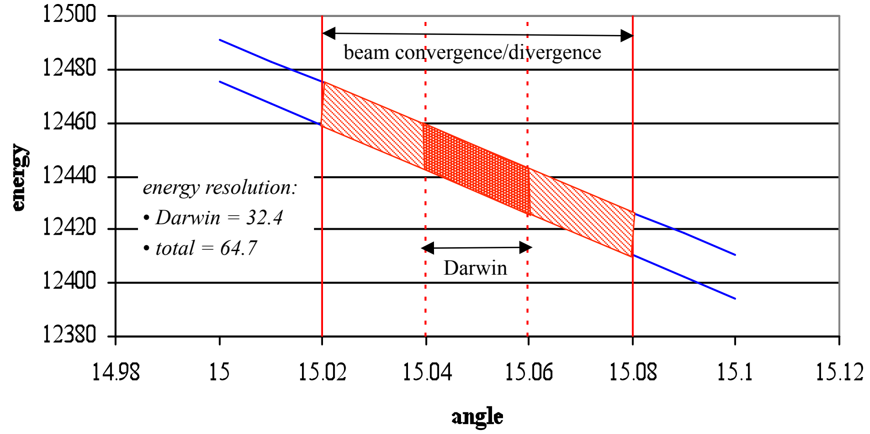


X-ray Crystal Monochromators

Acceptance and DuMond Diagram

BL Optics - Soft X-ray – 7/20/10 SMB Summer School - Rabedeau

X-ray Crystal Monochromators
Acceptance and DuMond Diagram



X-ray Crystal Monochromators
Improving Energy Resolution

$$\delta\varepsilon / \varepsilon = \delta\,\theta / \tan\,\theta_{Bragg}$$

- *employ higher index monochromator crystal (1/tan θ scaling), but not feasible for lower energy XAS applications*
- *use collimating mirror upstream of monochromator to reduce vertical angular spread to near Darwin limit or less (collimating M0 mirrors are available on BL4-1, **4-3**, **6-2**, 7-3, 9-2, 9-3, 11-2, 14-1, and **14-3**), unfortunately this is less effective at low energy since Darwin width is quite large (e.g., Si(111) at 2472eV is 177urad whereas synchrotron fan rms opening angle is 226urad on BL4 and 180urad on BL14)*
- *reduce vertical angular acceptance*
  - *BL w/o mirror optics upstream on mono (BL2-3)*
  - *BL w/ collimating mirrors to reduce mirror aberration effects*

X-ray Crystal Monochromators
Harmonic Content

- *crystal monochromators pass not only the fundamental energy of interest but also allowed higher order harmonics since*
  $\sin\theta = (\lambda / 2a_0)(h^2 + k^2 + l^2)^{1/2}$
- *fortunately the reduced Darwin width of higher order harmonics decreases the diffracted intensity as a function of peak index*
- *Si(111) example with fundamental at 2472 eV (53.13 deg):*

index	energy (eV)	Darwin (urad)	δε/ε
111	2472	177.0	1.33e-4
333	7416	12.2	9.1E-06

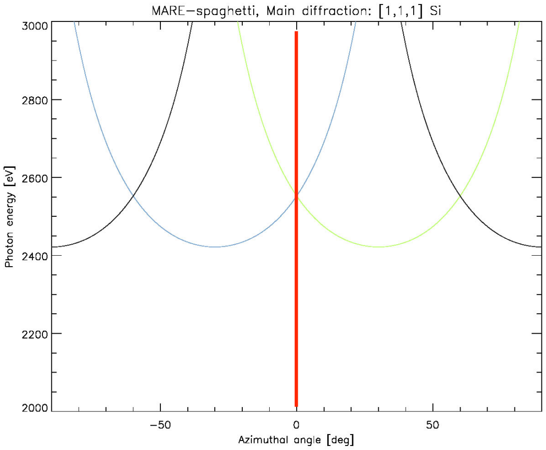
- *narrower rocking curves also facilitate slightly detuning double crystal pair in monochromator to suppress diffraction from harmonics while retaining most of diffracted intensity of fundamental*
  - *detuning maximizes mono sensitivity to crystal angular misalignment!*
  - *it is always better to use mirrors to harmonic reject when feasible (variable incidence M0 on BL4-1, **4-3**, **6-2**, 7-3, 9-2, 9-3, 11-2, 14-1, & **14-3**)*

X-ray Crystal Monochromators
Glitches

What's a glitch in the context of x-ray optics?

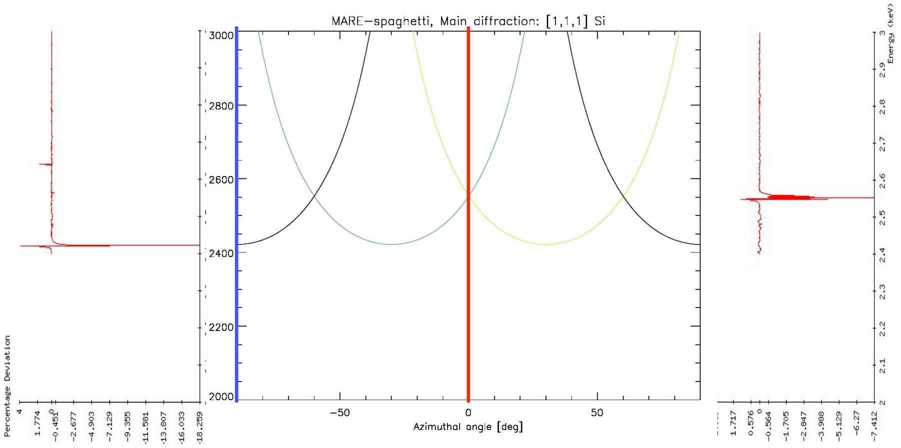
Bragg diffraction condition is met for multiple reflections at the same energy.

Intensity of the fundamental is altered as the mono energy is scanned through the glitch as some of the photons are scattered into these spurious Bragg peaks. Example at right shows Si(111) crystal aligned with Si(2-20) along crystal surface and beam direction (aka SSRL "phi 90").



Si(111) "phi90" crystal glitches 2-3keV
(note - SSRL employs 90 rotation in phi definition)

X-ray Crystal Monochromators
Glitches – Si(111) example



Si(111) phi 0

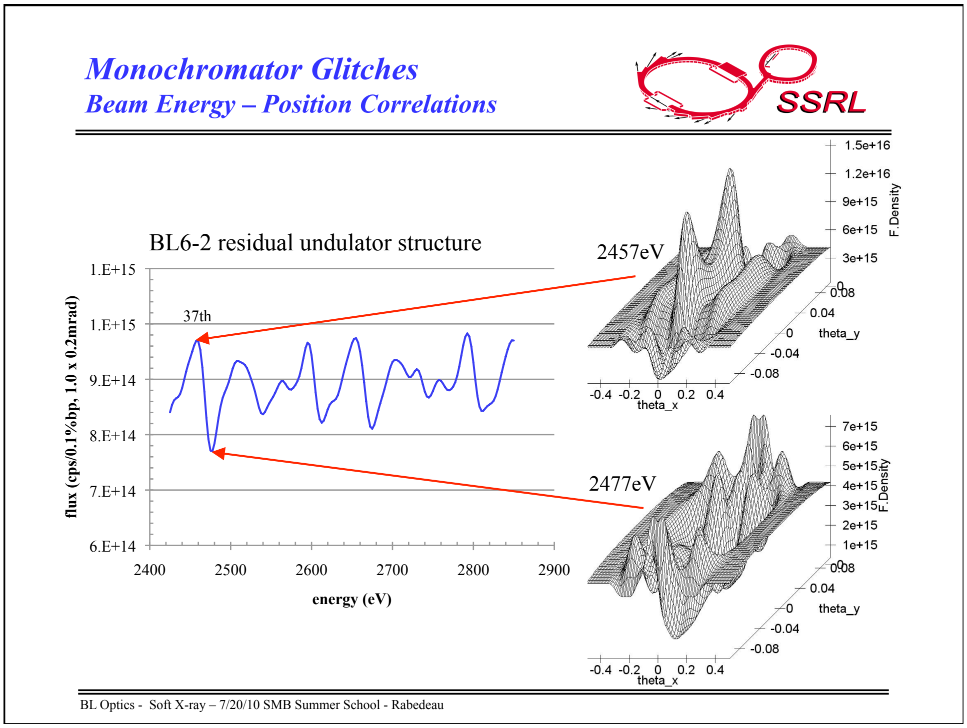
Si(111) phi 90

(remember - SSRL employs 90 rotation in phi definition wrt MARE)

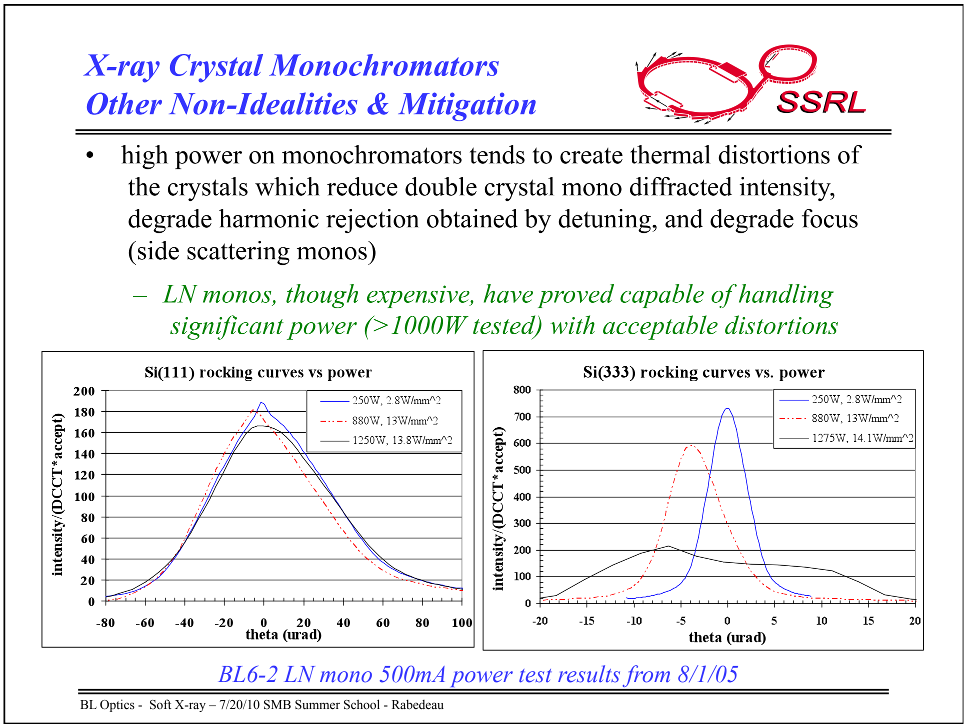
X-ray Crystal Monochromators
Glitches

- Won't glitches just ratio out of the data?
  - *The glitches may ratio out, but consider…*
  - *Monochromatic beams tend to possess energy – position correlations (more in a moment). As the mono tunes through the glitch, the resulting flux change may only occur in a localized portion of the beam which marches across the beam as the energy is scanned across the glitch.*
  - *Any sample non-uniformity, detector response non-uniformity, I0 monitor response non-uniformity, etc can result in the glitch not normalizing out of the data.*
- What can I do to reduce the impact of glitches?
  - *Plan in advance! Glitch spectra are available online at*

    http://www-ssrl.slac.stanford.edu/~smangane/smbxas/
  - *With advance warning crystal sets in SSRL double crystal monos can changed remotely (LN monos) or manually (all other DCM) to provide the crystal set that has the least problematic glitch characteristics.*



Monochromator Glitches
Beam Energy – Position Correlations



X-ray Crystal Monochromators
Other Non-Idealities & Mitigation

- high power on monochromators tends to create thermal distortions of the crystals which reduce double crystal mono diffracted intensity, degrade harmonic rejection obtained by detuning, and degrade focus (side scattering monos)
  - *LN monos, though expensive, have proved capable of handling significant power (>1000W tested) with acceptable distortions*

BL6-2 LN mono 500mA power test results from 8/1/05

X-ray Crystal Monochromators Other Non-Idealities & Mitigation

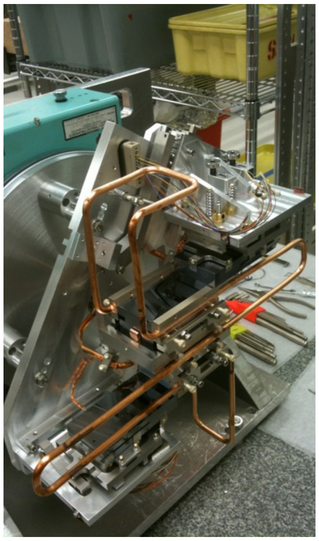
- SSRL employs quasi channel cut double crystal monochromators as this approach tends to make for quieter monos; however…
  - *diffracted beam height varies as 2*channel height*cos θ so hutch table and/or downstream optics need to compensate for beam motion as a function of energy*
- Roll misalignment between the first and second crystal in a double crystal monochromator results in beam horizontal motion with energy
  - *roll misalignment is particularly troublesome when the mono is followed by a toroidal focusing mirror as beam horizontal motion results in a focusing mirror yaw error such that the focus shape changes with energy*
  - *the LN monochromators employed on ID beam lines include a remote roll adjustment capability*
  - *the crystals in the older double crystal monochromators have been polished to minimize miscuts and carefully aligned such that the first and second crystal surfaces are parallel (eg., BL2-3)*

SSRL Low Energy XAS BL

- *BL6-2:*
  - *57 pole, 70mm period, 0.9T (5.4keV Ec) wiggler*
  - *Rh-coated M0 (collimating) & M1 (focusing) mirrors, 5-17keV M0 mirror cutoff , Rh L-edges 3004-3412eV*
  - *Si(111) phi 0 & Si(311) LN-cooled mono, 2.4-17 keV*
  - *high power density (11.1kW/mr^2 unfiltered)*
  - *high demand for fixed instruments (microscope and XRS) and power management difficulties so low energy XAS being moved to BL4-3 and BL14-3*
- *BL4-3:*
  - *20 pole, 230mm period, 1.9T (11.4keV Ec) wiggler*
  - *Ni-coated M0 (collimating) mirror, 4-14keV variable cutoff, Ni k -edge 8333eV*
  - *Si(111) phi 0 & Si(111) phi 90 LN-cooled mono, 2.4-14keV*
  - *medium power density (8.3kW/mr^2 unfiltered)*

SSRL Low Energy XAS BL

- *BL14-3: (commissioning this fall)*
  - *bend magnet 1.25T source (7.5keV Ec)*
  - *Ni-coated M0 (collimating) & M1 (focusing) mirrors, 3.6-6.0 keV M0 mirror cutoff*
  - *Si(111) phi 0 & Si(111) phi 90 water -cooled mono, 2.05-5 keV*
  - *low power density (0.29kW/mr^2 unfiltered)*
  - *~0.2 x 0.2 mm focus (anticipated) with micro-focus KB mirrors to be installed on downstream optical bench using 0.2x0.2mm focus as virtual source*



A Few Closing Thoughts -
Some Keys to Optimal BL Performance

- *your BL request should reflect careful consideration of the experiment requirements vs. the source/optics capabilities (ie., the best BL for a given experiment isn't always the most familiar BL)*
- *plan/communicate needs in advance such that the BL is configured optimally for your experiment (mirror cutoffs, mono crystal cuts, etc.)*
- *avoid depositing waste power in optics, rather use slits and filters to best advantage!*
- *utilize the BL mirrors to optimize performance*
  - *power filtering*
  - *harmonic rejection*
  - *collimation for energy resolution*
  - *beam focusing for increased flux on smaller samples*
- *to maximize mono stability avoid mono detuning whenever possible*