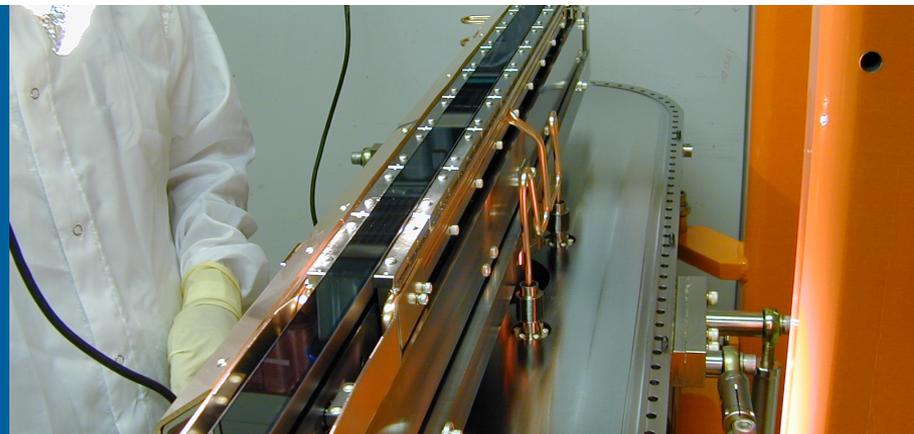


OPTICAL COMPONENTS & DETECTORS FOR HARD X-RAY SOURCES



DENNIS MILLS
Advanced Photon Source

National School for Neutron and X-ray Scattering
July/August 2024

Outline of Presentation

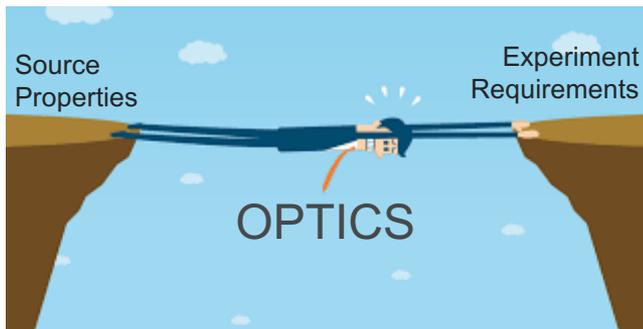
1. Why Do We Need Optics?
2. X-ray Mirrors (Reflective Optics)
3. X-ray Lenses (Refractive Optics)
4. Single Crystal, Multilayers & Zone Plates (Diffractive Optics)
5. High Heat Load Optics
6. Detectors

I will not be discussion gratings as they are used in the soft x-ray region of the spectrum and the focus of this talk will be hard x-ray optics.

OPTICS BRIDGE THE SOURCE AND EXPERIMENT

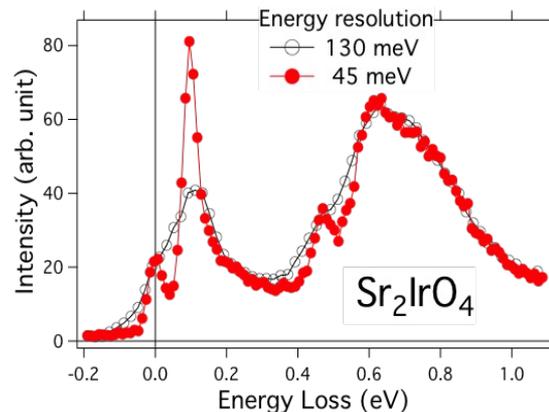
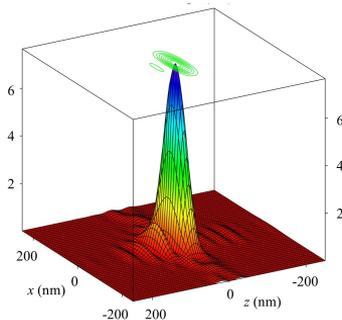
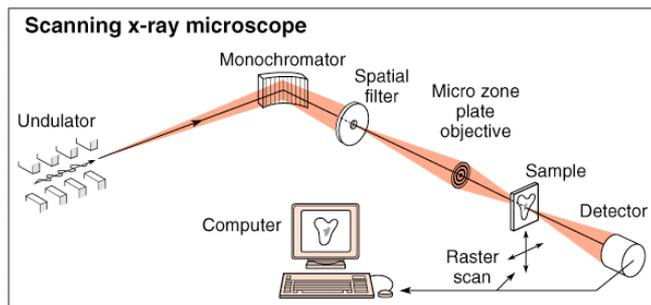
Source Properties

- x-ray energy ranges from BMs and IDs
- x-ray intensity
- emittance of the source
- degree of coherence
- polarization*



Experiment Requirements

- photons/sec on sample
- beam size
- collimation
- energy resolution ($\Delta E/E$)
- coherence
- polarization*



*Production of polarized (linear, circular, etc) beams is often accomplished by using specialized insertion devices and will not be covered in this talk.

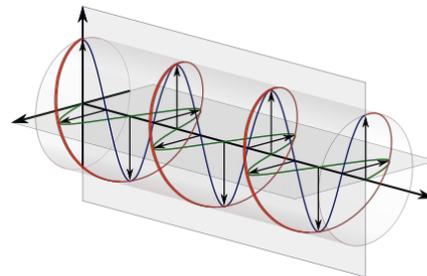
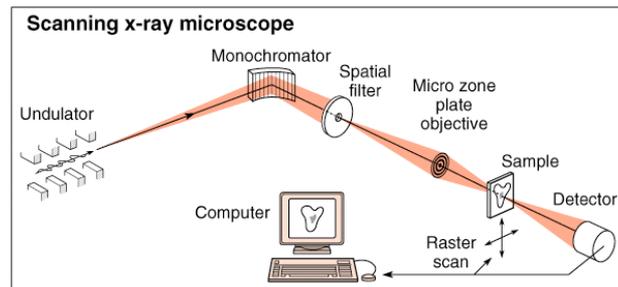
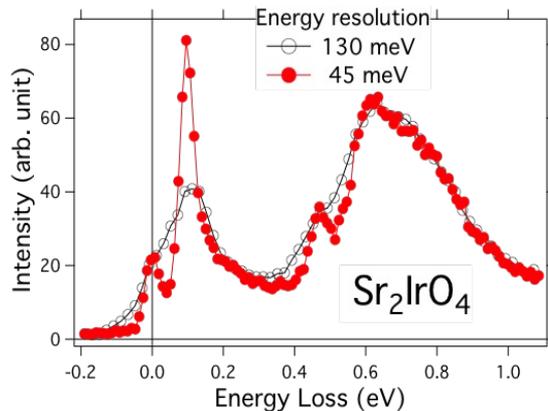
WHY X-RAY OPTICS?

- Control the energy (E) and bandwidth (ΔE) of the beam.
 - $\Delta E = 1\text{-}2 \text{ keV @ } 10 \text{ keV}$; $\Delta E/E = 10^{-1}$ (wide bandpass to increased flux for time-resolved studies – lectures latter this week)
 - $\Delta E = 1\text{-}2 \text{ eV @ } 10 \text{ keV}$; $\Delta E/E = 10^{-4}$ (typical diffraction exp.)
 - $\Delta E = \text{a few milli-eV @ } 10 \text{ keV}$; $\Delta E/E = 10^{-7}$ (inelastic scattering – lecture on Monday)

- Control the size/divergence of the beam (often related).
 - Micro- or nano-beams (spot sizes microns to 10's of nanometers)
 - Highly collimated beams

- Control the polarization of the beam.
 - Linear
 - Circular (magnetic x-ray scattering or spectroscopy)

Current trend is to design the ID to produce circularly polarized light and not to convert linear to circular polarization via optics.



REFLECTIVE OPTICS: X-RAY MIRRORS

SNELL'S LAW WITH X-RAYS

- The index of refraction for x-rays is less than one:

$$n = 1 - (n_e r_e / 2\pi) \lambda^2 = 1 - \delta (+ i\beta)$$

β is related to μ , the linear absorption coefficient, through $\beta = \lambda\mu/4\pi$, with $(I = I_0 e^{-\mu t})$. We'll ignore β hereafter.

where $r_e = (e^2/mc^2)$ is the classical radius of the electron (2.82×10^{-13} cm) and n_e the number of electrons per unit volume.

- Plugging in the values for r_e and n_e into the expression for δ , you find that:

$$\delta \approx 10^{-5} \text{ to } 10^{-6}.$$

- So the index of refraction for x-rays is less than one, but only by a few ppm.
- From Snell's Law [$n_1 \sin(\phi_1) = n_2 \sin(\phi_2)$], when $\phi_2 = 90^\circ$ we have the condition for **total external reflection**:

$$\cos(\theta_c) = n_2 \cos(0) \quad (\theta = 90^\circ - \phi)$$

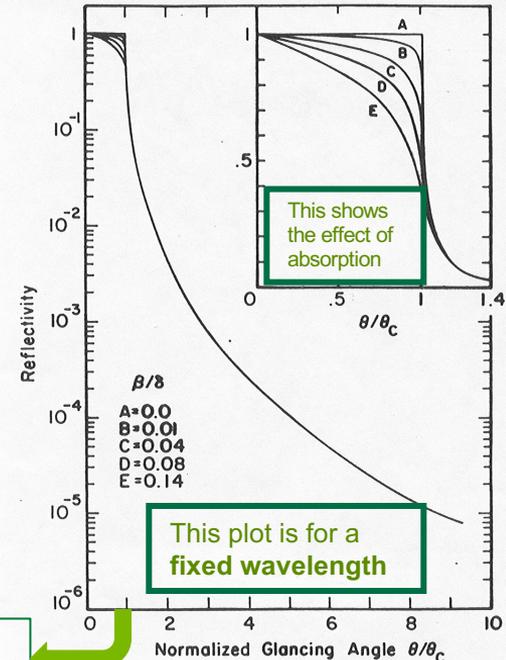
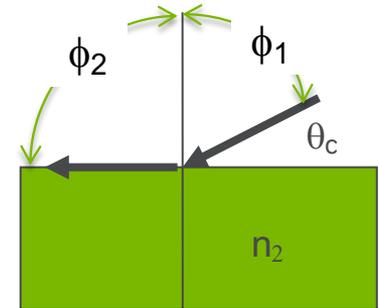
- Expanding the cosine of a small angle and substituting $1 - \delta$ for n_2 in the above equation gives:

$$1 - \frac{1}{2}(\theta_c)^2 = 1 - \delta$$

$$\theta_c = (2\delta)^{1/2}$$

- $\theta_c = (2\delta)^{1/2}$ and since δ is 10^{-5} to 10^{-6} , the critical angle will be about 10^{-3} or a few milliradian.

Air ($n_1 = 1$)



ENERGY CUTOFF FOR A FIXED ANGLE-OF-INCIDENCE MIRROR

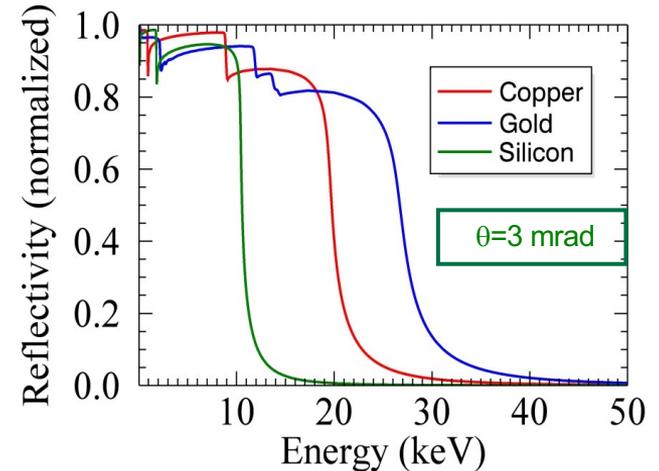
- Often mirrors are used as first optical components. This means a **polychromatic incident beam strikes the mirror at some fixed angle**.
- The relationship for the critical angle and wavelength can be re-written, for a **fixed angle of incidence θ** , in terms of a cut-off wavelength, $\lambda_{\text{cut off}}$, where wavelengths above $\lambda_{\text{cut off}}$ are reflected and those below $\lambda_{\text{cut off}}$ are not. Since $E = hc/\lambda$, I can re-write this and get a relationship for a **fixed incident angle, θ** , and determine the maximum, or cut-off energy, $E_{\text{cut off}}$, that will be totally reflected by the mirror.

$$\theta_c = (2\delta)^{1/2} \approx \lambda(n_e r_e / \pi)^{1/2}$$

$$E_{\text{cut off}} = hc/\lambda_{\text{cut off}} = (hc / \theta) (n_e r_e / \pi)^{1/2}$$

- Low-pass filters
 - mirrors can be used to effectively suppress high energies
 - mirrors are designed so that the cutoff energy, $E_{\text{cut off}}$, can be varied by having several different coatings deposited on the mirror substrate

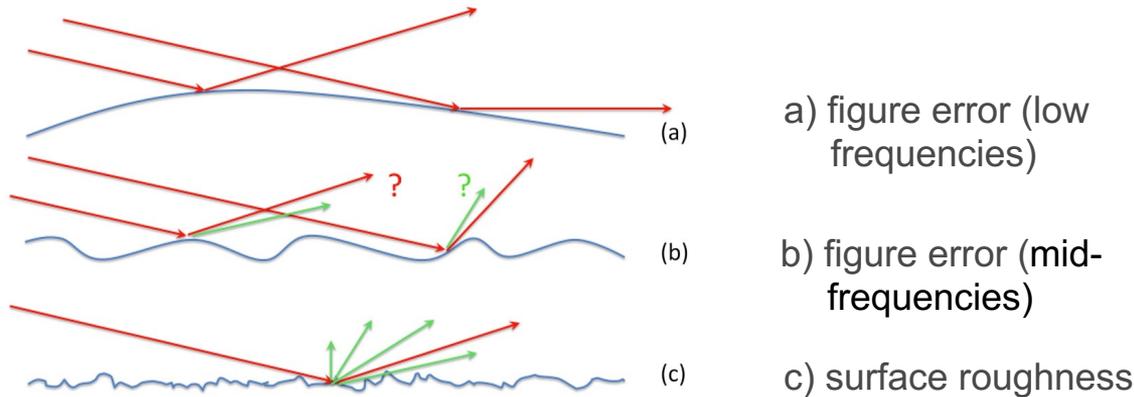
Cut-off energy, $E_{\text{cut off}}$ for fixed angle θ



For a fixed angle of incidence, you can vary the cut-off energy by coating the mirror with materials of different electron densities, n_e .

GRAZING INCIDENCE X-RAY MIRRORS

- Because the incidence angle are small (a few milliradians) to capture the full extent of the beam (about 1 mm or so), x-ray mirrors tend to be very long (sometimes over a meter).



- Mirrors can effectively remove a considerable amount of the heat from the incident (polychromatic) beam and reduce the thermal loading on downstream optics.

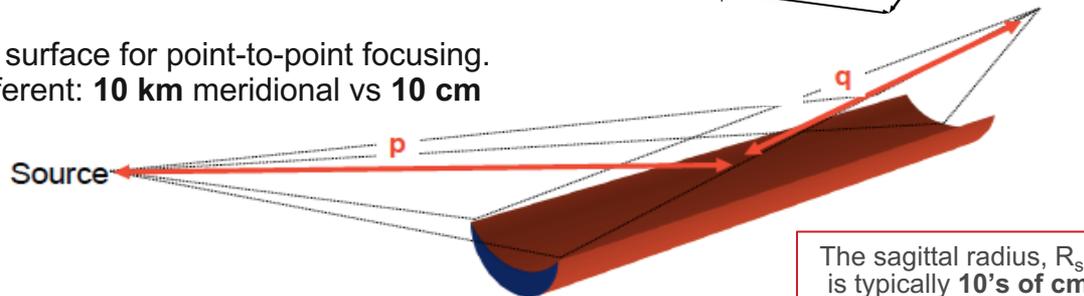
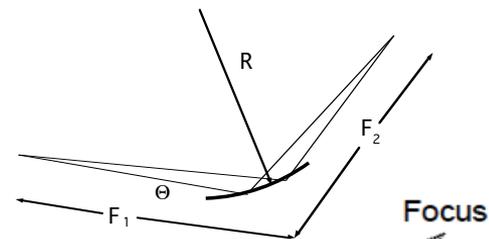


Most mirrors are made from silicon coated with one or multiple stripes of high-Z material after polishing.

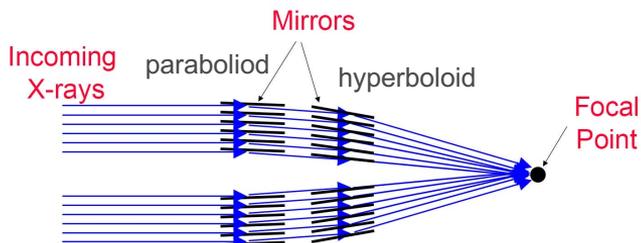
MIRRORS AS FOCUSING ELEMENTS

- **One-dimensional focusing**, collimating, etc.
 - An ellipse is the ideal shape for a reflecting surface for point-to-point focusing. (A source at one foci of the ellipse will be imaged at the other foci.)
 - In many cases cylindrically shaped mirrors are used rather than ellipses since they are considerably easier to fabricate (but may have so-called spherical aberrations).
- **Two -dimensional focusing**
 - An ellipsoid is the ideal shape for a reflecting surface for point-to-point focusing.
 - However, the radii of curvature are widely different: **10 km meridional** vs **10 cm sagittal**
 - Very difficult to fab a mirror like this

Typical meridional radius, R , is around **10's of kilometers**.



The sagittal radius, R_s , is typically **10's of cm**



Chandra X-ray Observatory, launched by NASA in 1999, is still operational as of 2024



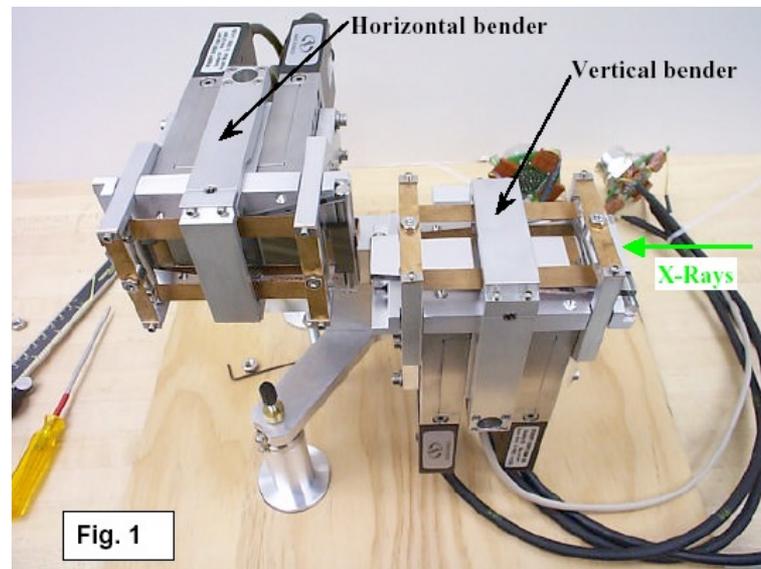
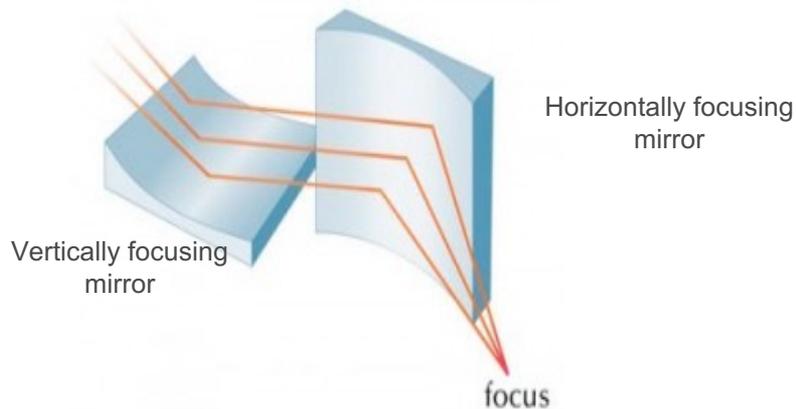
SNR 1181: Stunning Image of 800-year-old Supernova Remnant

MIRRORS AS FOCUSING ELEMENTS

▪ Kirkpatrick – Baez (KB) geometry

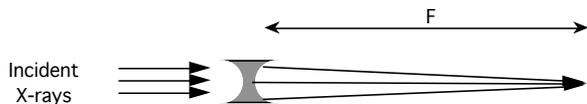
- Another system that focuses in two dimensions consists of a set of **two orthogonal singly focusing mirrors**, off which incident X-rays reflect successively, as first proposed in 1948 by Kirkpatrick and Baez (KB).
- This system allows for easier fabrication of the mirrors and is used frequently at synchrotron sources.

Mirrors are achromatic, i.e. the focal length is independent of x-ray wavelength.



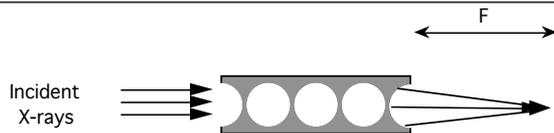
REFRACTIVE OPTICS: X-RAY LENSES

COMPOUND REFRACTIVE LENSES: ONE DIMENSIONAL FOCUSING



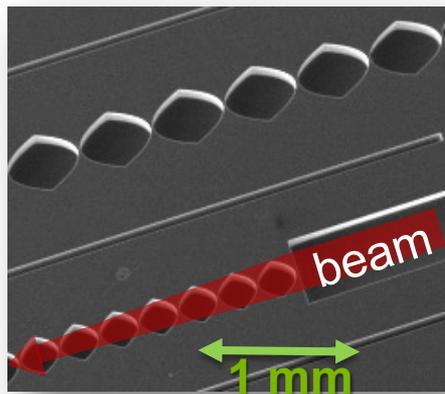
Single Refractive Lens

Note that since the index of refraction for x-rays is < 1 , concave lenses will focus the beam.



Compound Refractive Lens

Parabolic lenses etched 400 μm deep into Si wafer made at CNM and tested at APS. The gray shaded area is one lens.



- The Lens Maker's Equation for x-rays still applies:

$$1/F = \delta (1/R_1 + 1/R_2 + \text{etc.})$$

- For a single lens:

$$1/F = \delta(1/R + 1/R) \text{ or } F = R / 2\delta$$

this is for a lens with 2 curved surfaces

- If we have N surfaces, all with radius R:

$$F = R/2N\delta$$

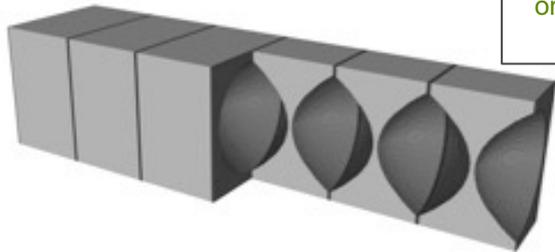
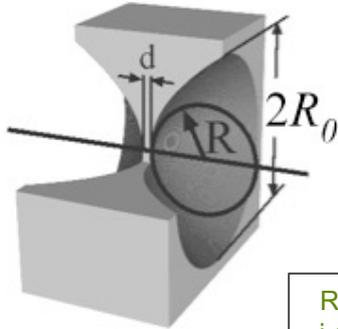
- Using the same numbers as before but with 50 lenses, i.e.:

$$R = 1 \text{ mm} \quad \delta \approx 10^{-5} \quad N = 50$$

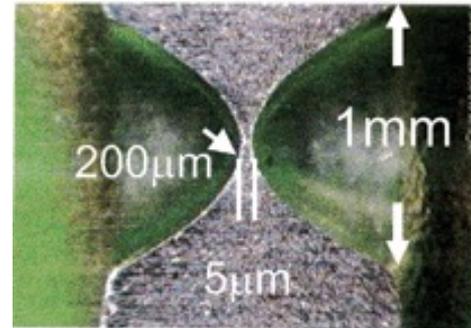
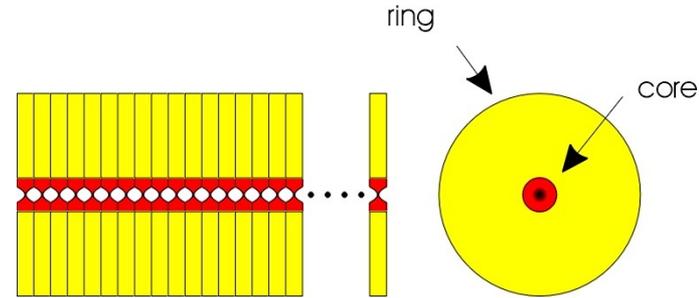
- Then the focal length, F, would be at 1 m.
- These lenses focus at rather larger distances and are well adapted to the scale of synchrotron radiation beamlines.

FOCUSING IN TWO DIMENSIONS WITH REFRACTIVE LENSES

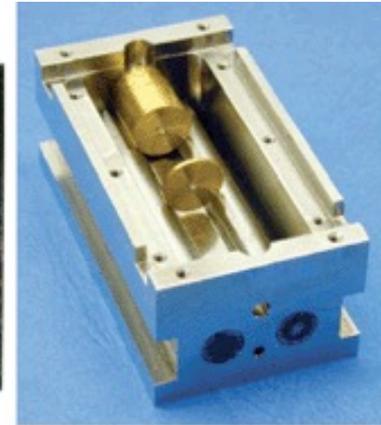
- 2-D lenses typically “embossed” and made from Be, Al or Ni
- Spherical lenses are easy to make but suffer from spherical aberrations.
- Paraboloids eliminate spherical aberrations.



Refractive lenses are chromatic, i.e. the focal length is dependent on x-ray wavelength since δ is a function of λ .



a

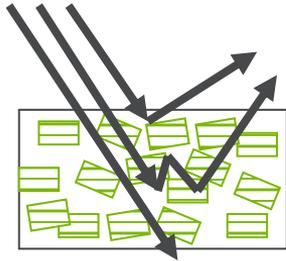


b

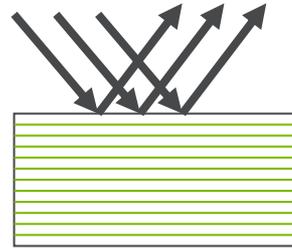
DIFFRACTIVE OPTICS: CRYSTALS, ZONE PLATES & MULTILAYERS

DIFFRACTION FROM PERFECT CRYSTALS

- The theory that describes diffraction from perfect crystals is called dynamical diffraction theory (as compared with kinematical theory, which describes diffraction from imperfect or mosaic crystals) first proposed in 1914 by C. G. Darwin in two seminal papers.



Mosaic crystal model



atomic planes

Perfect crystal model



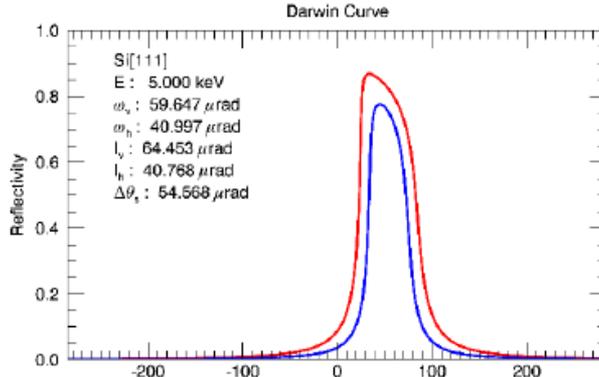
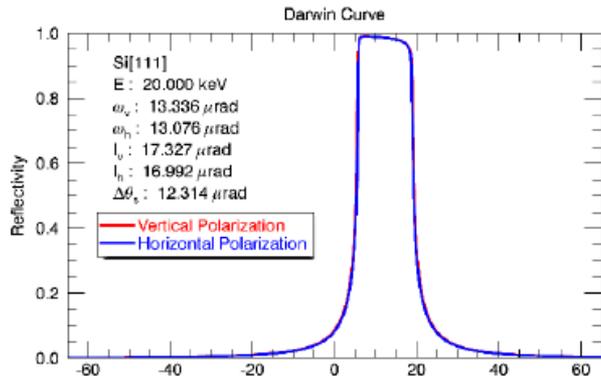
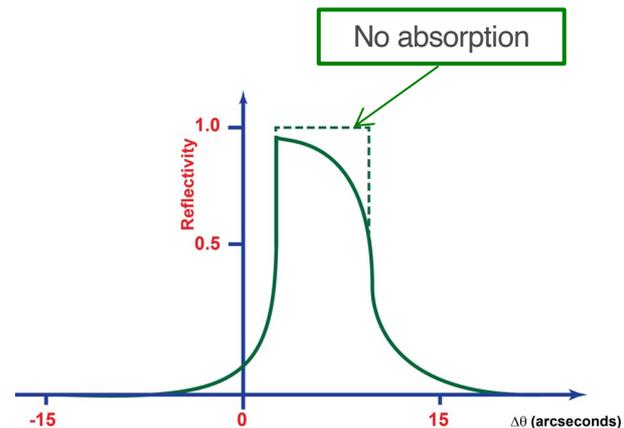
<http://www.eoht.info/page/C.G.+Darwin>

- In the case of a strong reflection from a perfect crystal of a monochromatic x-ray beam, the penetration of the x-rays into the crystal is not limited by the (photoelectric) absorption, but the beam is attenuated due to the reflecting power of the atomic planes. (This type of attenuation is called “extinction”.) *“if the crystal is perfect all the radiation that can be reflected is so, long before the depth at which the rays at a different angle are appreciably absorbed.”*

Aside: C. G. Darwin was the first to calculate the index of refraction for x-rays. Charles G. Darwin was the grandson of the “more famous” Charles Darwin of evolution fame.

TWO CONSEQUENCES OF LIMITED PENETRATION IN DIFFRACTION FROM PERFECT CRYSTALS

- The limited penetration due to extinction (reflection by the atomic planes) means at the Bragg condition, the x-ray beam is limited in the number of atomic planes it “experiences”.
- Consequence #1:
 - There is a finite angular width over which the diffraction occurs. This is often called the ***Darwin width***, ω_D
 - Depends on the strength of the reflection, $F(hkl)$, and square of the wavelength, λ^2 .
- Consequence #2:
 - The reflectivity over this narrow angular width is nearly unity, even in crystals with a finite absorption.



Using modern notation, Darwin width, ω_D , can be written as:

$$\omega_D = 2r_e F(hkl) \lambda^2 / \pi V \sin(2\theta)$$

$F(hkl)$ = structure factor and
 V = volume of unit cell

PERFECT CRYSTAL X-RAY MONOCHROMATORS

- Simply use Bragg's Law to select a particular wavelength (or energy)

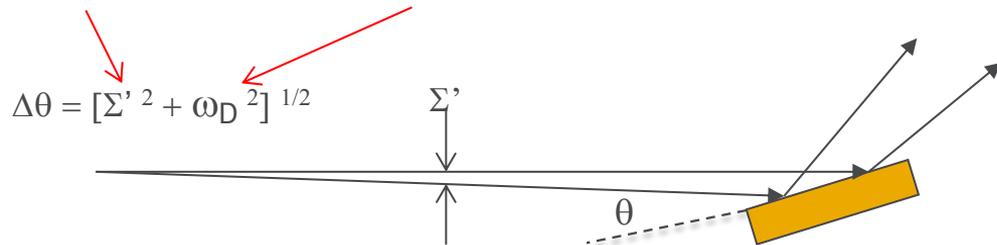
$$\lambda = 2d \sin(\theta).$$

- If we differentiate Bragg's Law ($\Delta\lambda = 2d \cos(\theta) \Delta\theta$), divide this by the original equation we can determine the energy resolution of the monochromator.

$$\Delta\lambda / \lambda = \Delta E / E = \cot(\theta) \Delta\theta$$

X-ray divergence (source)

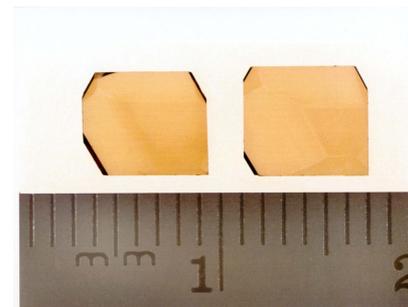
Darwin width (optic)



- At 8 keV (1.5\AA) for Si(111) $\omega_D \approx 40$ microradians. For an APS undulator, the opening angle of the central cone is about 5-10 microradians.
- In this case the energy resolution of the mono is determined by the crystal. Plugging in the values you get $\Delta E / E = 10^{-4}$. So for at 8 keV x-ray the bandwidth (or ΔE) would be about 0.8 eV.



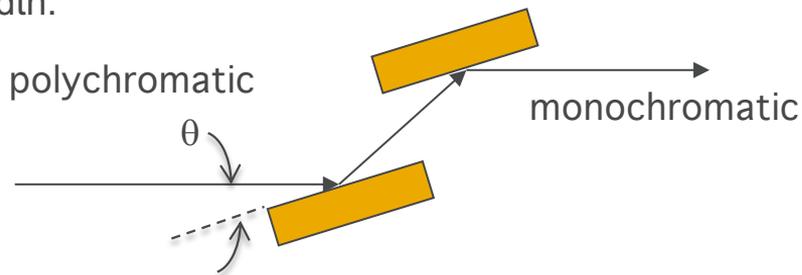
Silicon is used for monochromator crystals as it can be easily and cheaply obtained.



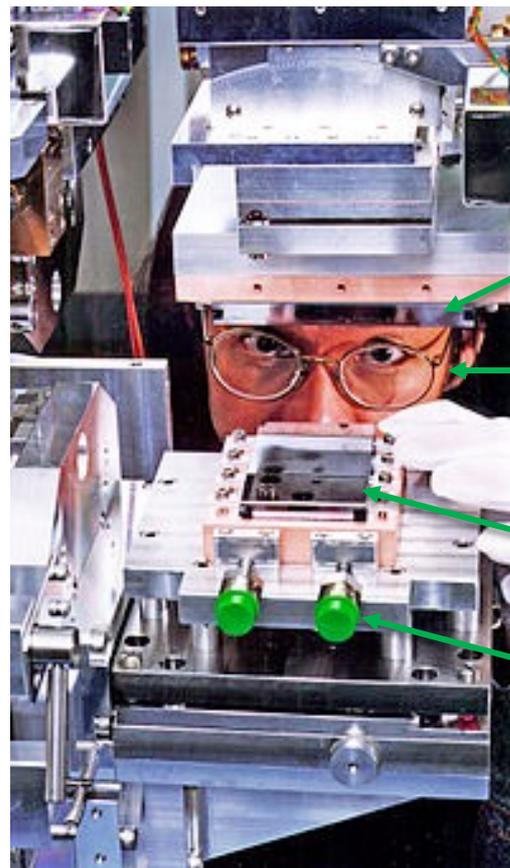
Synthetic diamonds are also a good choice but much harder to find with the required quality

DOUBLE CRYSTAL MONOCHROMATORS

- The most common arrangement for a monochromator is the double-crystal monochromator (DCM). It:
 - is **non-dispersive**, that is all rays that diffract from the first crystal simultaneously diffract from the second crystal (if same crystals with same hkl's are used)
 - **keeps the beam parallel to the incident beam** as the energy is changed (by changing the Bragg angle, θ).
- There is little loss in the throughput using two crystals because the reflectivity is near unity over the Darwin width.



- Monochromators need to be cooled to maintain the desired properties. More on that later.



edge of
2nd Si
crystal

APS staff
trying to look
immersed in
his work

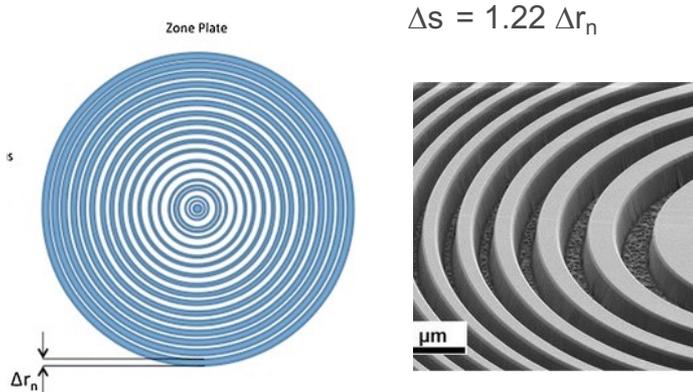
cooled 1st
Si crystal

coolant
connections

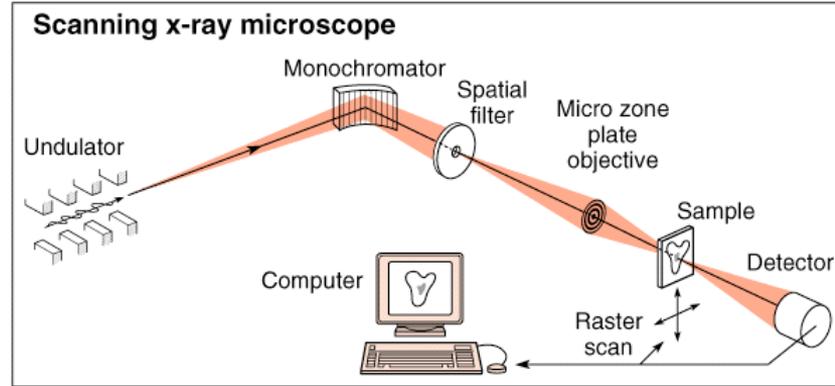
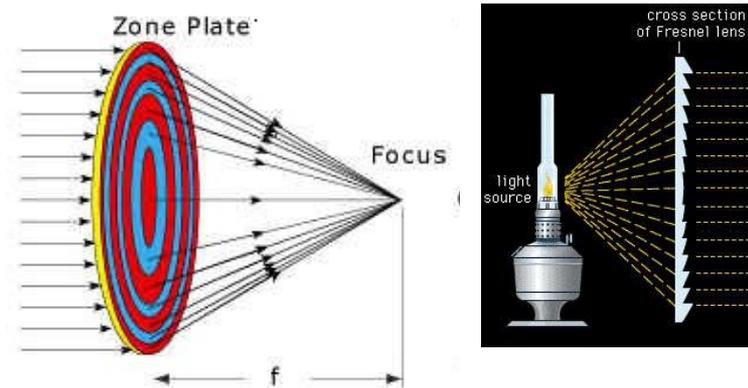
polychromatic beam going into the slide

FRESNEL ZONE PLATES

- The focusing capability is based on constructive interference of the wavefront modified by passage through the zone plate. The wave that emerges from the zone plate is the superposition of spherical waves, one from each of the zones.
- Zone plates satisfy the condition that the pathlength varies by $\lambda/2$ for each ring. They are composed of alternating concentric zones of two materials. To get rid of the spherical waves that are out of phase, every other ring must block (absorb) the x-rays – these are called amplitude zone plates.
- In general, the size of the **focal spot from the zone plate**, Δs , is determined by the width of the outermost ring, Δr_n , and is given by:



$$\Delta s = 1.22 \Delta r_n$$

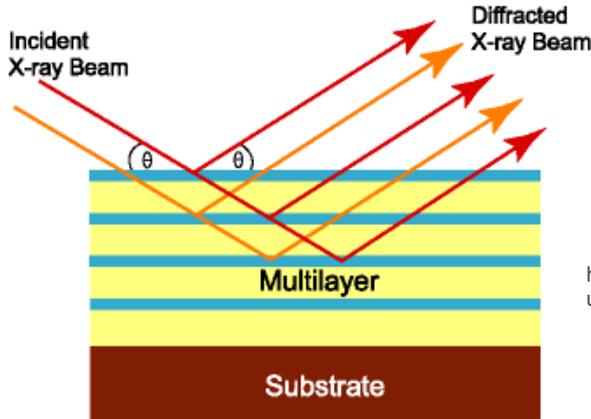


The challenge in making hard x-ray zone plate for is making Δr_n small while maintaining a high thickness to efficiently absorb the unwanted wave, i.e. you need to have a high aspect ratio – very challenging!!!!

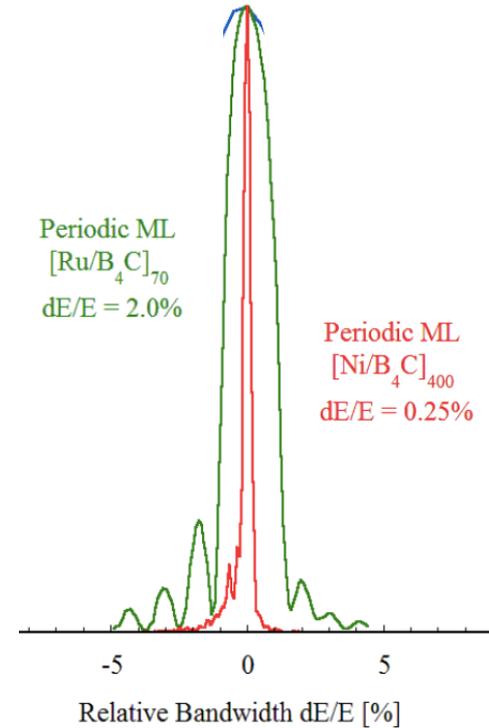
Zone plates are chromatic, i.e. the focal length is dependent on x-ray wavelength.

HARD X-RAY MULTILAYER OPTICS

- A “periodic multilayer” coating is a film stack comprising a number of identical repetitions of two or more optically dissimilar component layers.
 - Wide energy band-pass
 - Focusing : θ_B multilayer $\gg \theta_c$ mirror so multilayer length \ll mirror length
 - Increased numerical aperture
- The energy is selected using Bragg’s Law.
- The energy bandwidth is determined by the number of layers N ; $\Delta E/E \approx 1/N$.



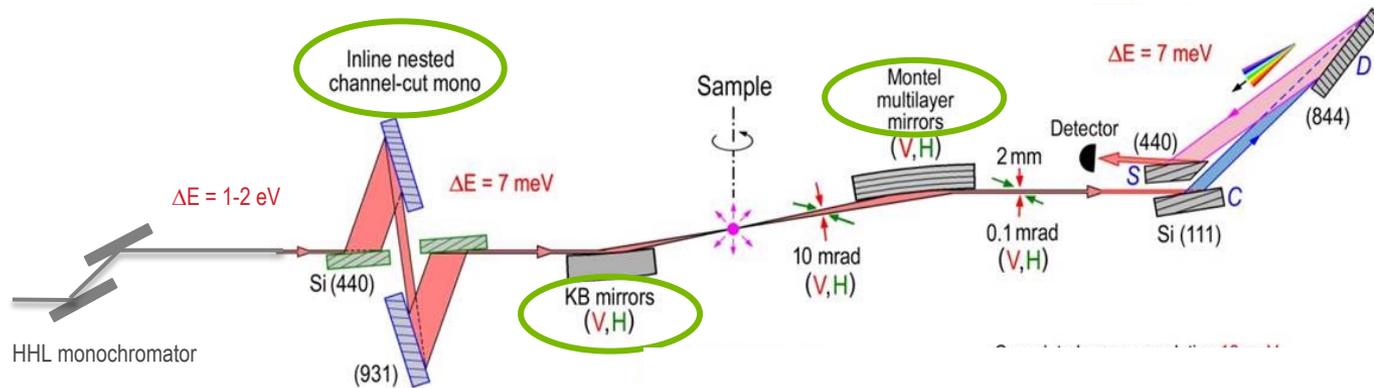
<http://xray0.princeton.edu/~phil/Facility/Guides/XrayDataCollection.html>



Calculated bandwidth from 2 different multilayers:

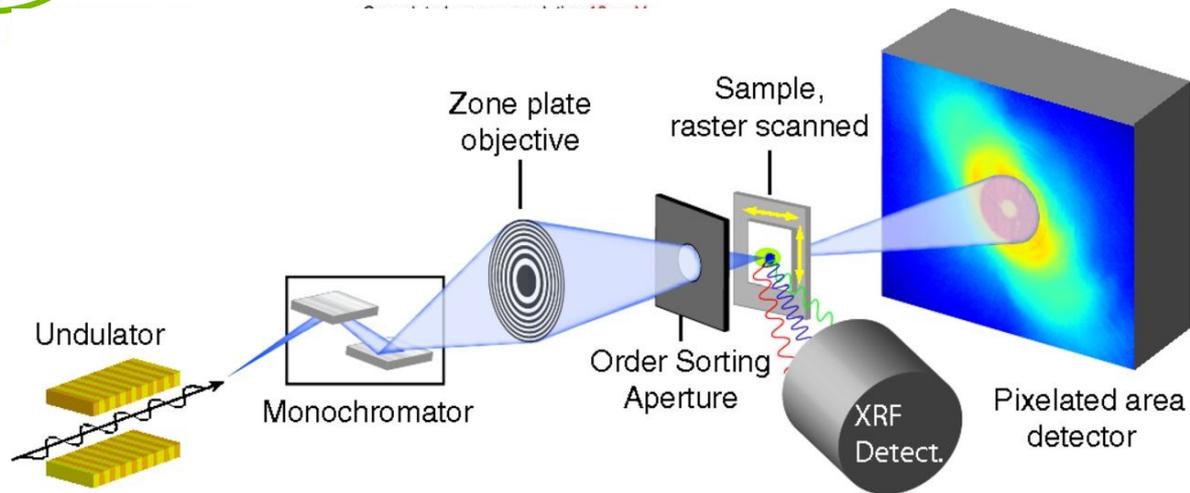
- Green: 70 layers of $\text{Ru/B}_4\text{C}$; $\Delta E/E = 2\%$
- Red: 400 layers of $\text{Ni/B}_4\text{C}$; $\Delta E/E = 0.25\%$

VARIOUS OPTICAL COMPONENTS ARE OFTEN FOUND IN A SINGLE BEAMLINE



Design of an ultrahigh-resolution crystal monochromator and analyzer (from sub-10 meV to sub-meV) for inelastic x-ray scattering.

Schematic layout of a scanning x-ray microscope (Bio-nanoprobe) at the APS.



HIGH HEAT LOAD X-RAY OPTICS

THERMAL LOADING ON OPTICAL COMPONENTS

- Along with the enormous increase in x-ray beam brilliance from insertion devices comes unprecedented powers and power densities that must be effectively handled so that thermal distortions in optical components are minimized and the full beam brilliance can be delivered to the sample.

<u>Process</u>	<u>Approx. Heat Flux (W/mm²)</u>
Interior of rocket nozzle	10
Commercial plasma jet	20
Fusion reactor components	0.05 to 80
Meteor entry into atmosphere	100 to 500
APS Undulator @ 30m (on-axis 2.4 m 100 mA)	10 to 160



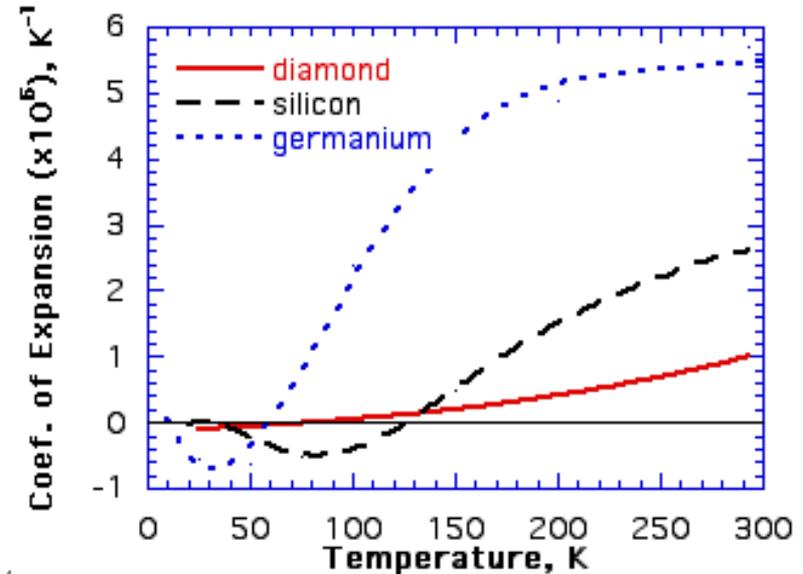
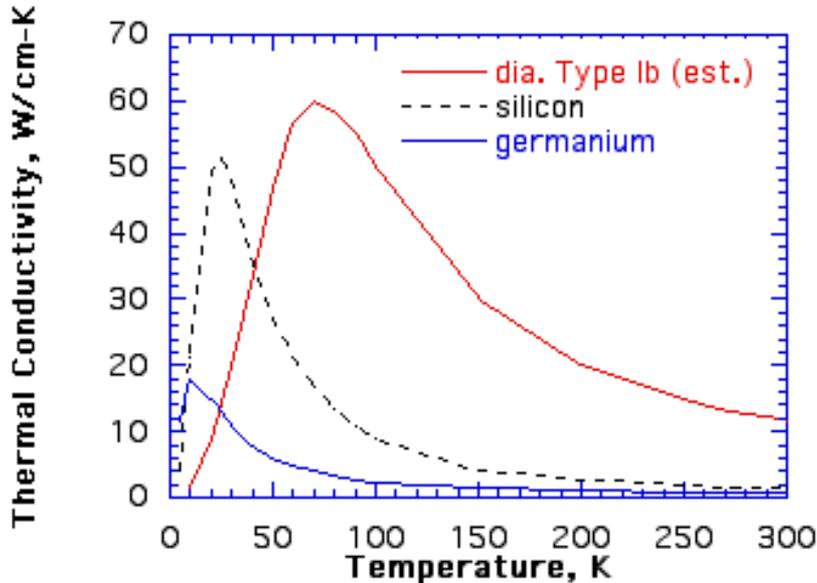
In order to maintain the beam intensity and collimation (i.e., brightness) through the optics, special attention must be paid to the issue of thermal management for those first optical components.

PROPERTIES OF SI, GE, AND C(DIAMOND)

Thermal gradients, ΔT , and coefficient of thermal expansion, α , contribute to crystal distortions:

$$\alpha \Delta T = \Delta d / d$$

We therefore need to look for materials that have a very low coefficient of thermal expansion, α , and/or have a very high thermal conductivity, k , so that the material cannot support large ΔT 's.



MINIMIZING DISTORTION OF OPTICAL COMPONENTS – THERMAL MANAGEMENT

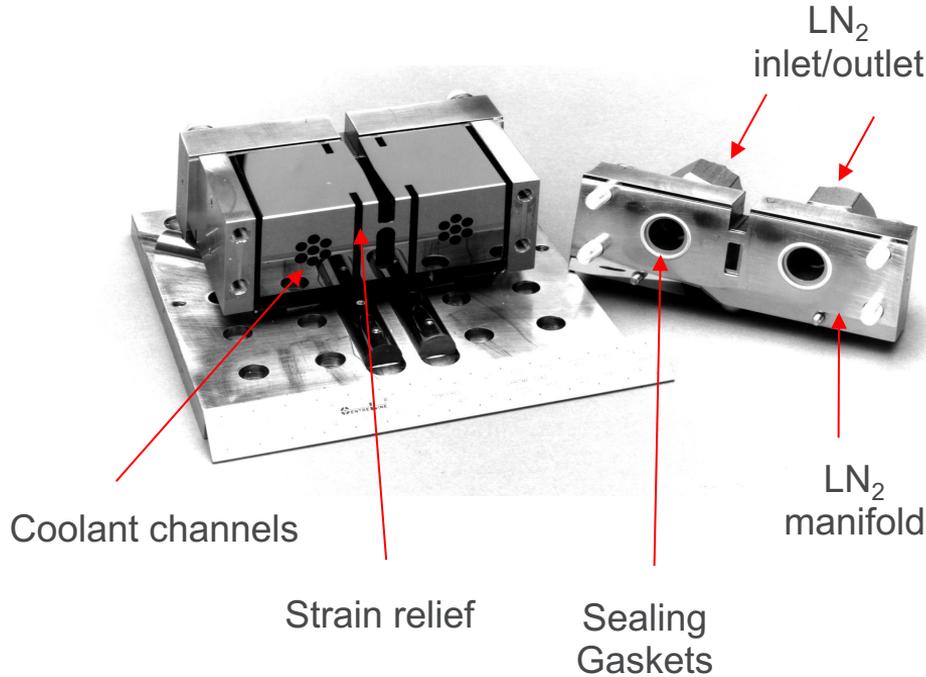
FOM of Typical Monochromator Materials

Material and Temperature	Thermal Conductivity (k)	Coef. Thermal Expansion (α)	Figure of Merit FOM (k/α)
Si (300K)	1.2 W/cm-K	2.3×10^{-6} /K	0.5
Si (78K)	14 W/cm-K	-0.5×10^{-6} /K	28
Dia. (300K)	20 W/cm-K	0.8×10^{-6} /K	25

These properties motivate us to use cryogenically cooled silicon or room temperature diamond as high heat load monochromators.

LN₂ COOLED Si MONOCHROMATORS

Cryogenically (LN₂) Cooled Si Mono



The historical development of cryogenically cooled monochromators for third-generation synchrotron radiation sources, Bilderback, Freund, Knapp, and Mills, J. Synch. Rad. 7, 2000.



Liquid nitrogen pump

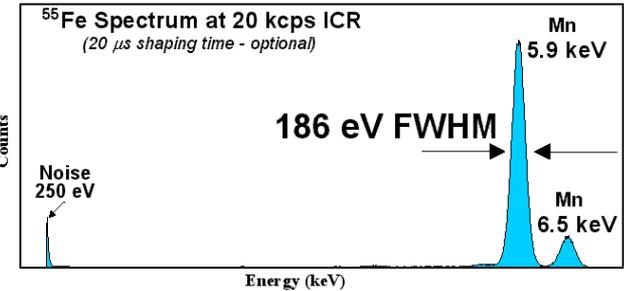
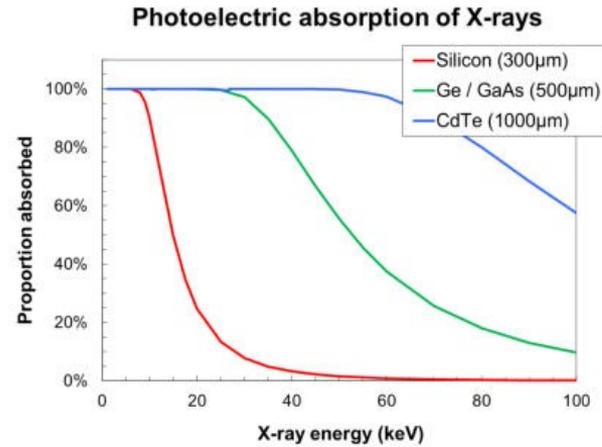
DETECTORS

(THANKS NINO MICELI FOR THE SLIDES!)

DETECTORS FOR X-RAYS

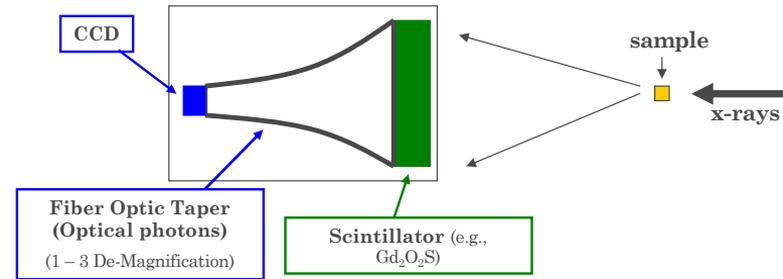
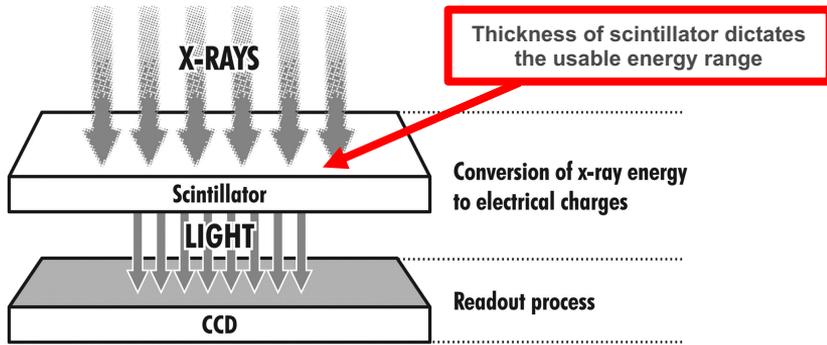
Things to think about when determining what characteristics you need in an x-ray detector:

- **Energy of x-rays** – what are the required absorption properties of the sensor for good efficiency?
- **Number of x-rays/sec** – can you count single (individual) x-rays or do you need to integrate the signal?
- **Energy resolution** – what $\Delta E/E$, if any, do you need?
- **Size over which you want to collect data** – can you use a point detector or do you need an area detector?



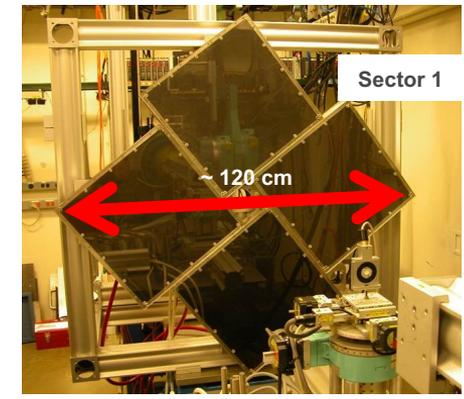
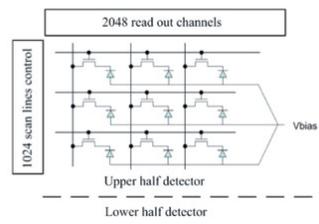
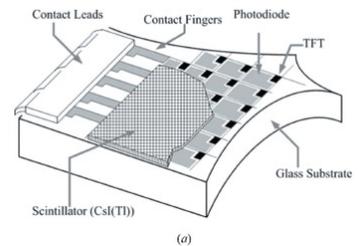
INDIRECTLY (X-RAYS → OPTICAL PHOTONS → ELECTRONS)

Scintillators/Phosphors



a-Si and CMOS flat panels

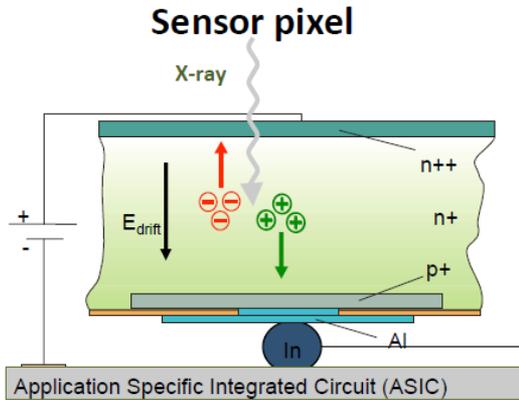
CCD-based detectors



DIRECT (X-RAYS → ELECTRONS)

Pixel array detectors (e.g., Pilatus, Eiger, Lambda, etc)

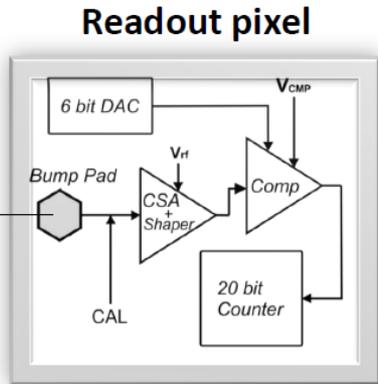
- Each pixel is a single photon counting detector
 - Thus count-rate limited (i.e., deadtime)
- Generally more sensitive/efficient/faster than indirect detection.



Direct Detection of X-rays in solid state sensor

→ Point Spread Function: < 1 pixel

3.6 eV to create 1 eh-pair @12keV: 3300 eh-pairs

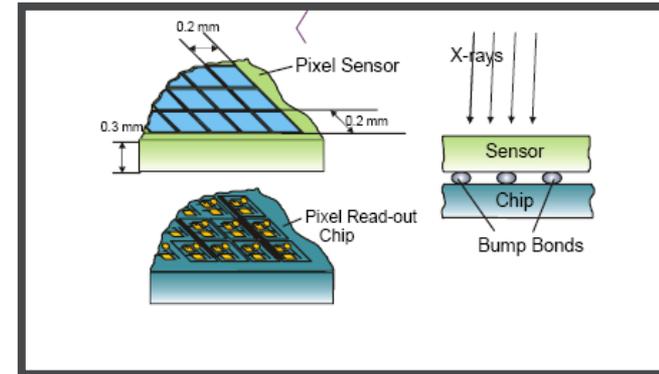


Single Photon-counting in CMOS

→ no readout noise & dark current

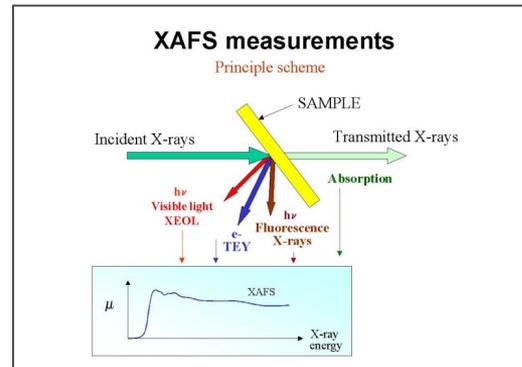
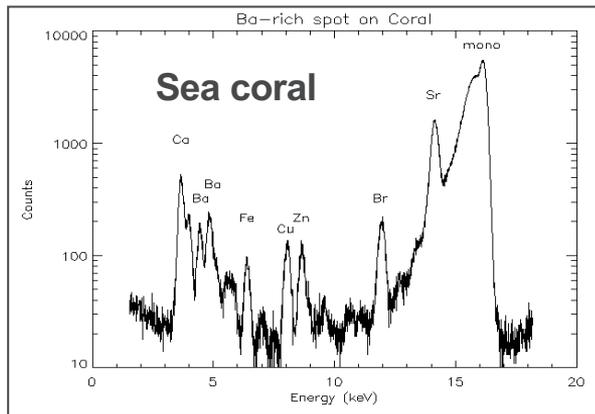
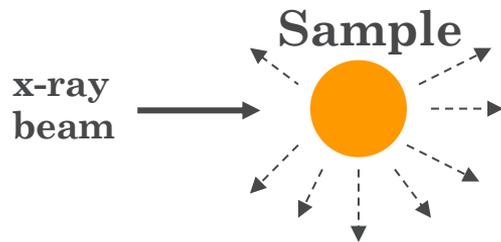
→ adjustable energy threshold

→ high dynamic range (20 bit)

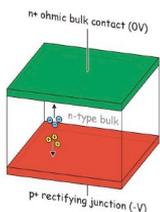


ENERGY RESOLVING DETECTORS – COUNTING

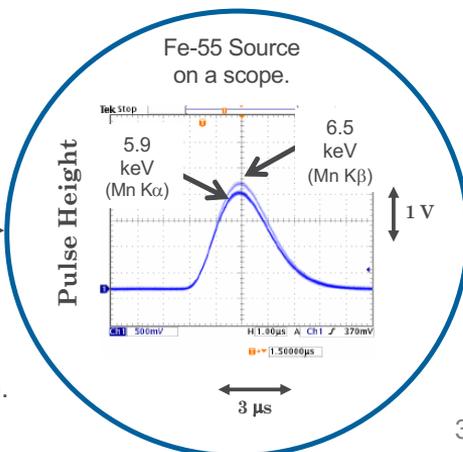
Energy Dispersive Detectors or Spectroscopic Detectors or XRF Detectors



Diode



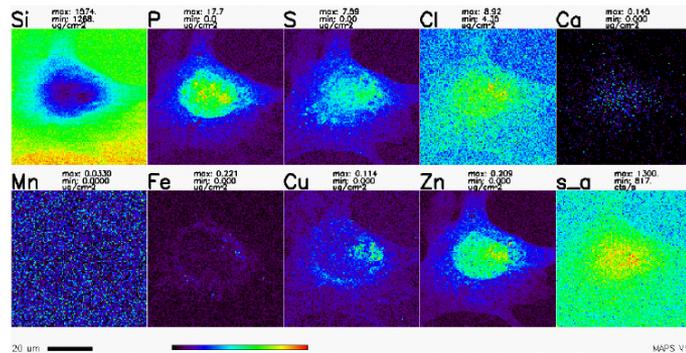
XRF detector



X-Ray Energy \sim # of e-h pair.

- 3.67 eV are need to produce 1 e-h pair for silicon.
- Energy resolution about 150 eV at 10 KeV

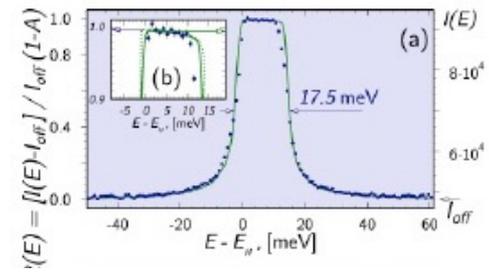
Nanoprobe XRF image of a cell



Need to collect >10 elemental ROIs \Rightarrow energy dispersive detector

SUMMARY

- X-ray optics is an active area of research at both universities and national laboratories.
- High-brightness sources provide new opportunities but ever higher demands on the quality of optics to ensure beam coherence is preserved through the optics.
- Metrology is key to making good optics – “You Can’t Improve What You Can’t Measure”



LETTERS

PUBLISHED ONLINE: 17 JANUARY 2010 | DOI: 10.1038/NPHYS1506

nature
physics

High-reflectivity high-resolution X-ray crystal optics with diamonds

Yuri V. Shvyd'ko^{1*}, Stanislav Stoupin¹, Alessandro Cunsolo^{1,2}, Ayman H. Said¹ and Xianrong Huang²

SCIENTIFIC REPORTS

OPEN

Interlaced zone plate optics for hard X-ray imaging in the 10 nm range

Received: 18 August 2016

Iman Mekkouh^{1,2}, Ivan Vucelja^{1,3}, Benedek Pápai¹, Manuel Güenzel^{1,4}

Physics Reports 974 (2022) 1–40

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journal homepage: www.elsevier.com/locate/physrep



ELSEVIER



Wavefront preserving X-ray optics for Synchrotron and Free Electron Laser photon beam transport systems

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LETTERS

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nature
physics

Breaking the 10 nm barrier in hard-X-ray focusing

Hidekazu Mimura^{1*}, Soichiro Handa¹, Takashi Kimura¹, Hirokatsu Yumoto², Daisuke Yamakawa¹, Hikaru Yokoyama¹, Satoshi Matsuyama¹, Kouji Inagaki³, Kazuya Yamamura³, Yasuhisa Sano¹, Kenji Tamasaku⁴, Yoshinori Nishino⁴, Makina Yabashi⁴, Tetsuya Ishikawa⁴ and Kazuto Yamauchi^{1,3}

Journal of
Applied Physics

Proposal for entangled x-ray beams

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Published Online: 13 June 2022

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(http://science.energy.gov/~/media/bes/pdf/reports/files/BES_XRay_Optics_rpt.pdf)

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- *Soft X-ray Optics*, Eberhard Spiller, SPIE Optical Engineering Press (1994)

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- *Advanced X-ray Detector Technologies: Design and Applications*, K. Iniewski, Springer (2022).

QUESTIONS

APPENDIX 1A: DIELECTRIC CONSTANT AND THE DRUDE MODEL

The dielectric constant, κ , is defined as follows:

$$\kappa = \mathbf{D}/\mathbf{E} = (\mathbf{E} + 4\pi\mathbf{P})/\mathbf{E} = 1 + 4\pi(\mathbf{P}/\mathbf{E})$$

For a single electron $\mathbf{P} = -e\mathbf{x}$ and for multiple electrons $\mathbf{P} = -en_e\mathbf{x}$ (where n_e is the number of electrons/unit volume)

In the Drude model, the frequency of the collective oscillations of the electron gas around the positive ion background is the so-called plasma frequency and equal to:

$$\omega_o = [4\pi n_e e^2 / m]^{1/2} \quad (m = \text{mass of the electron})$$

If we assume a simple harmonic approximation then:

$$F = ma = m\ddot{x} = -eE - kx$$

where k is the “spring constant” associated with ($k = m \omega_o^2$).

APPENDIX 1B: X-RAY INDEX OF REFRACTION

If x has the form $x = Ae^{i\omega t}$, solving for x we get:

$$x = (e/m)E/(\omega_0^2 - \omega^2) \text{ and}$$

$$P = -(e^2/m)n_e E /(\omega_0^2 - \omega^2)$$

Using this simple model, one can then calculate the polarizability of the material:

$$\kappa = 1 + 4\pi(P/E) = 1 + 4\pi (e^2/m)n_e [1/(\omega_0^2 - \omega^2)]$$

For Si, $n_e = 7 \times 10^{23} \text{ e/cm}^3$ and so the plasma frequency is:

$$\omega_0 = 5 \times 10^{16}/\text{sec}$$

For a 1 Å x-ray, the angular frequency, $\omega (= [2\pi c/\lambda])$, is $2 \times 10^{19}/\text{sec} (\gg \omega_0)$ and so we can write:

$$\kappa = 1 + 4\pi (e^2/m)n_e [1/(\omega_0^2 - \omega^2)] \approx 1 - 4\pi (e^2/m)n_e [1/(\omega^2)]$$

$$n = \kappa^{1/2} = [1 - (n_e(e^2/mc^2) \lambda^2/\pi)]^{1/2} \approx 1 - (n_e r_e/2\pi)\lambda^2$$