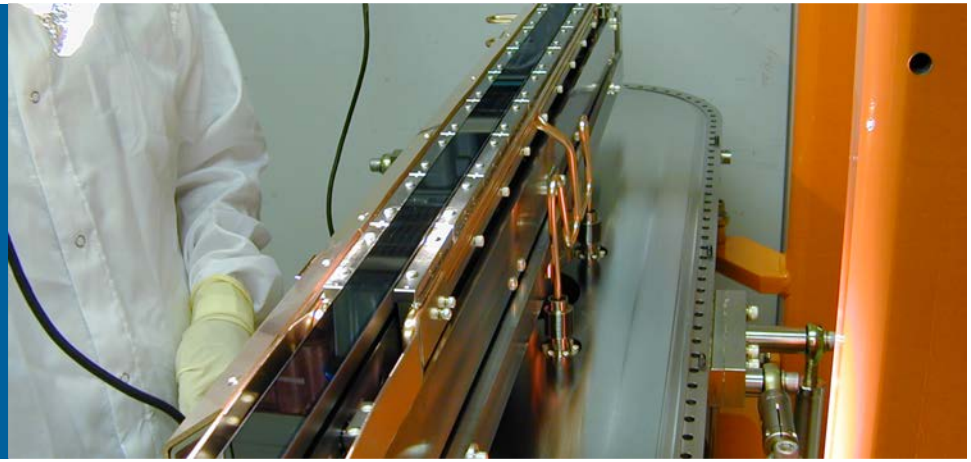
OPTICAL COMPONENTS & DETECTORS FOR HARD X-RAY SYNCHROTRON RADIATION SOURCES



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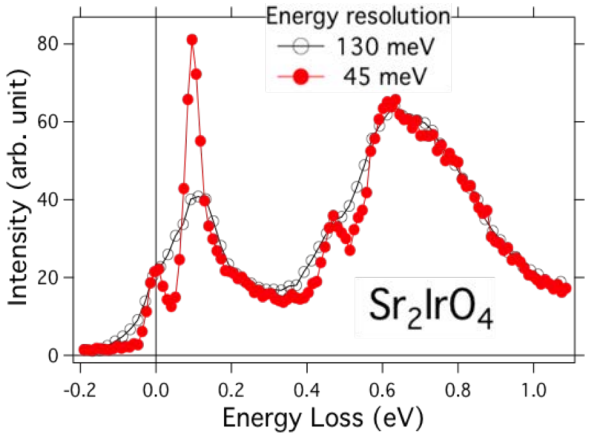
Outline of Presentation

1. Why Do We Need Optics?
2. X-ray Mirrors (Reflective Optics)
3. Perfect Crystal X-ray Optics (Diffractive Optics)
4. Multilayer Optics (Diffractive Optics)
5. Focusing Optics (Reflective, Diffractive and Refractive)

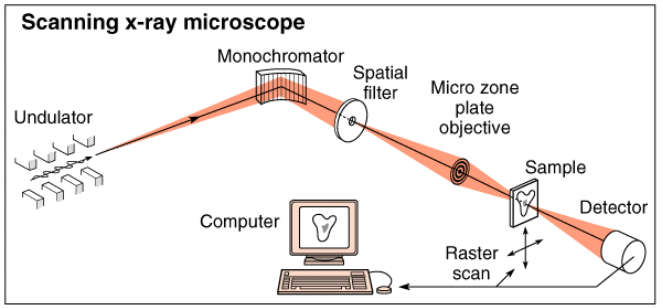
I will not be talking about gratings as they are used in the soft x-ray region of the spectrum and the focus of this talk will be hard x-ray optics.

WHY DO WE NEED OPTICS

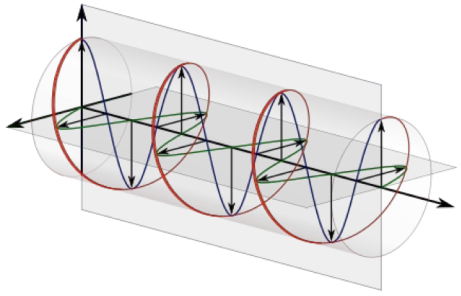
- Control the energy (E) and bandwidth (ΔE) of the beam.
  - **ΔE = 1-2 keV** @ 10 keV; ΔE/E = 10^{-1} (wide bandpass for increased flux - time-resolved studies)
  - **ΔE = 1-2 eV** @ 10 keV; ΔE/E = 10^{-4} (typical diffraction exp.)
  - **ΔE = a few milli-eV** @ 10 keV; ΔE/E = 10^{-7} (inelastic scattering – lectures later today)



- 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)



Now typically done by the ID and not the optics

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X-RAY MIRRORS

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INDEX OF REFRACTION FOR X-RAYS: N < 1

See Appendices 1 & 2 for more details

- This expression for the (real part) index of refraction:

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

is usually written as:

$$n = 1 - \delta, \quad \text{where} \quad \delta = (n_e r_e/2\pi)\lambda^2.$$

varies as the density and the square of the wavelength.

and $r_e = (e^2/mc^2)$ is the classical radius of the electron (2.82×10^{-13} cm), n_e is the electron density, and λ is the wavelength of the x-ray.

- When you plug in the numbers for the real part of the index of refraction you find:

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

- So you have:
  - **an index of refraction less than one**
  - **differing from unity by only a few ppm**

This simple treatment did not include any absorption. A more detailed calculation would result in an expression:

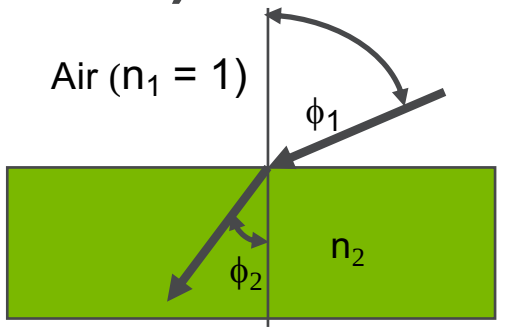
$$n = 1 - \delta - i\beta$$

Where $\beta = \lambda\mu/4\pi$, with μ the linear absorption coefficient ($I = I_o e^{-\mu t}$).

SNELL'S (OR THE SNELL-DECARTES) LAW

Willebrord Snellius

- The reflection and refraction of x-rays can be treated as any other electromagnetic wave traveling in a medium with index of refraction n_1 encountering a boundary with another material with index of refraction n_2.



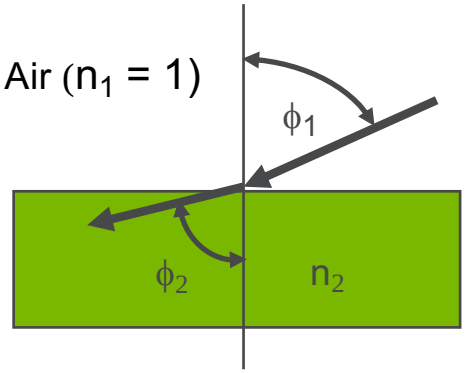
visible light:
ray bends toward the surface normal

Typical values for n_2 (at 5890Å) are:

water: $n_2 = 1.33$

glass: $n_2 = 1.52$

- The resultant kinematic properties (which follow from the wave nature of the radiation at boundaries) are:
  - The angle of incidence equals the angle of reflection
  - **$n_1 \sin(\phi_1) = n_2 \sin(\phi_2)$** (Snell's Law), where the ϕ's are measured with respect to the boundary normal



x-rays:
ray bends away from the surface normal

For x-rays, the direction of propagation bends away from surface normal.

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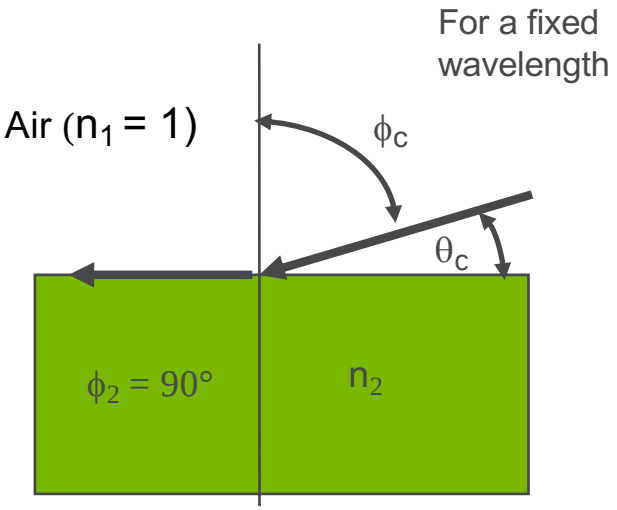
CRITICAL ANGLE FOR TOTAL EXTERNAL REFLECTION

- Let an x-ray (in vacuum, where $n_1 = 1$) impinge on a material with index of refraction n_2. From Snell's Law (when $\phi_2 = 90°$), we have:

$$n_1 \sin(\phi_c) = n_2 \sin(90°);$$

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

$$\cos(\theta_c) = n_2$$



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

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

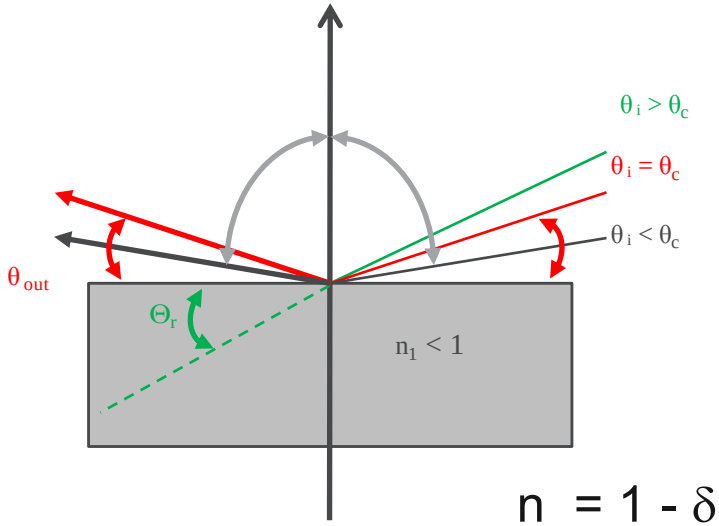
$$\mathbf{\theta_c = (2\delta)^{1/2}}$$

Recall that the typical values for δ at 1 Å is 10^{-5} to 10^{-6} and so the critical angle is going to be about 10^{-3} or a few milliradians

θ_c is the so-called **critical angle**, the angle at which there is **total external reflection** and the material behaves like a mirror.

X-RAY REFLECTIVITY

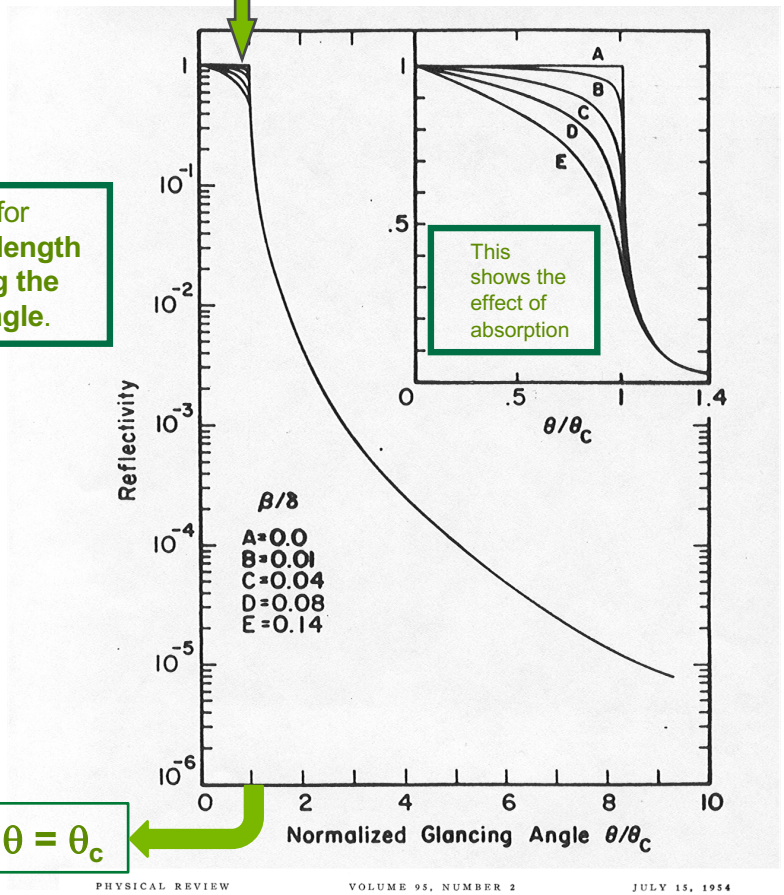
- The amplitude of the reflected wave can be determined through the Fresnel equations. Sparing you the details, it can be shown that:
  - **Below θ_c, there is unit reflectivity** (when β, the absorption. equals 0)
  - **Above θ_c, the reflectivity falls rapidly**



$$n = 1 - \delta - i\beta$$

Below θ_c, there is unit reflectivity (if β =0)

This plot is for **fixed wavelength** and **varying the incident angle**.



PHYSICAL REVIEW VOLUME 95, NUMBER 2 JULY 15, 1954

Surface Studies of Solids by Total Reflection of X-Rays*

L. G. Parratt
Cornell University, Ithaca, New York
(Received March 22, 1954)

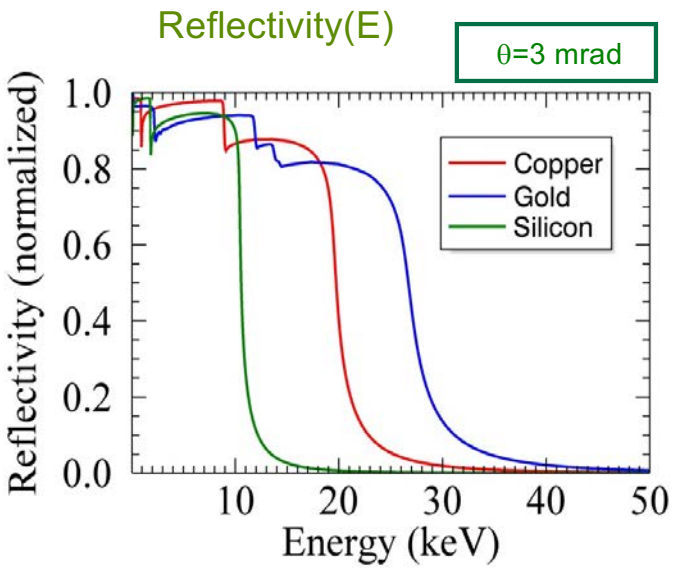
ENERGY CUTOFF FOR A FIXED ANGLE-OF-INCIDENCE MIRROR

- 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 critical wavelength, λ_c, where wavelengths above λ_c are reflected and those below λ_c are not. Since E = hc/λ, I can re-write this and get a relationship for a **fixed incident angle. θ, and determine the maximum energy, E_c, that will be totally reflected by the mirror.**

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

$$E_c = hc/\lambda_c = (hc/\theta)\,(n_e r_e/\pi)^{1/2}$$



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

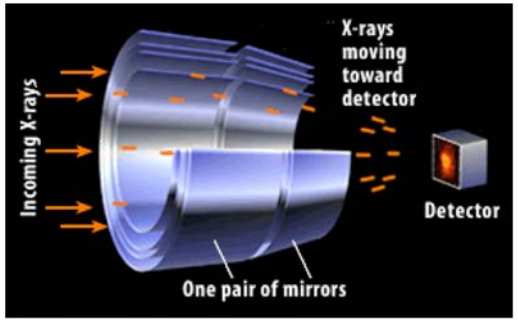
Critical energy, E_c for fixed angle θ

X-RAY MIRRORS

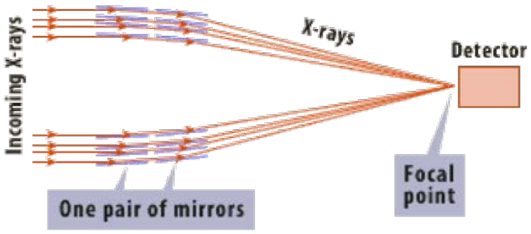
- Because the incidence angle are small (a few milliradians) to capture the full extent of the beam (say 1 mm or so), x-ray mirrors tend to be very long (sometimes over a meter).

- Low-pass filters
  - mirrors can be used to effectively suppress high energies
  - mirrors are designed so that the cutoff energy, E_c, can be varied by having several different coatings deposited on the mirror substrate

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



Courtesy Chandra mission website: http://chandra.harvard.edu

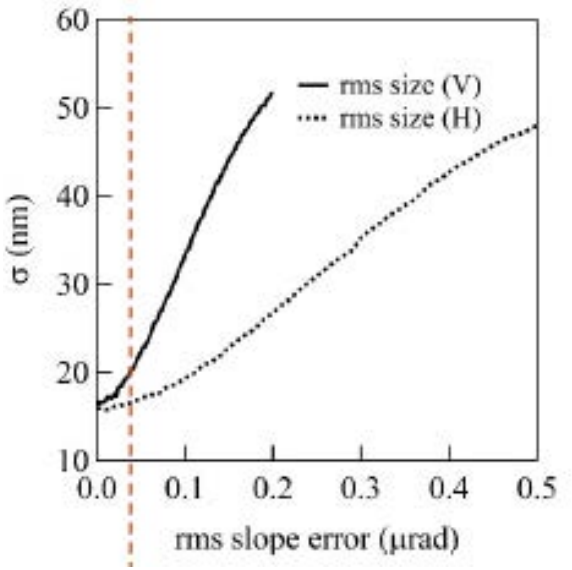


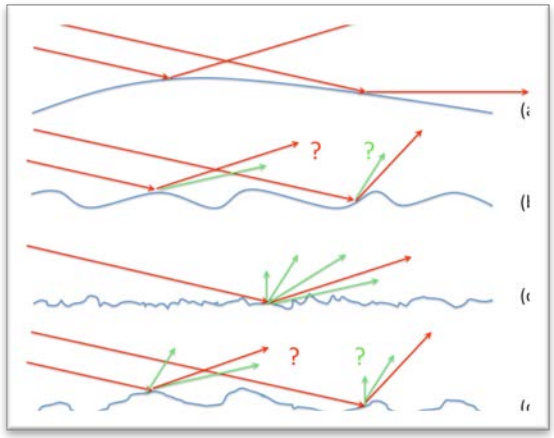
Chandra's mirrors are positioned so they're almost parallel to the entering X-rays. The mirrors look like open cylinders, or barrels. The X-rays skip across the mirrors much like stones skip across the surface of a pond.


Water cooled mirror in its vacuum tank.

HIGH-BRIGHTNESS SOURCES PUT STRINGENT DEMANDS ON MIRROR QUALITY

- The mirrors requirements are very stringent if you want to use them for focusing or to preserve the x-ray beam brightness.





Sources of errors in mirrors

(a) long range slope errors

(b) medium range slope errors

(c) surface roughness

(d) sum of all three errors

- If we hope to focus the x-ray beam to 20 nm, the specifications required are:

  - slope error must be < 0.03 microradians (rms)
  - surface roughness < 1 nm (rms)

  over the length of the mirror.

CRYSTAL OPTICS

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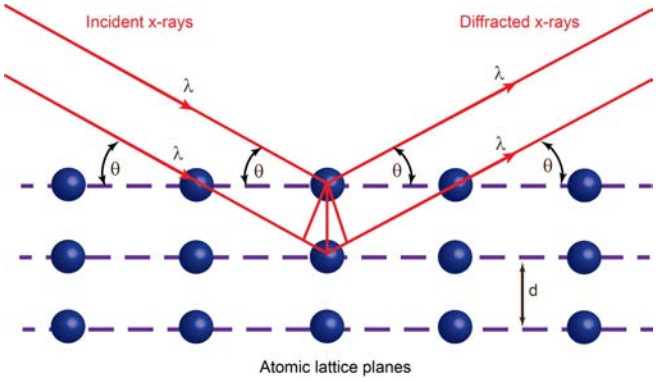
DIFFRACTIVE OPTICS

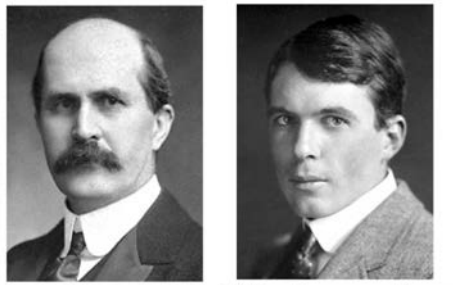
- By far, the most commonly used optical component for x-rays are crystals satisfying Bragg's law, i.e.,

Constructive interference when

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

Bragg's Law





William Henry Bragg William Lawrence Bragg

The Braggs shared the 1915 Nobel Prize in Physics.

- AT SR facilities, perfect (dislocation free) single crystals are used as the diffractive elements since:
  - they have **near-100% reflectivity** (more later)
  - the physics is well understood and components can be fabricated with **predicted characteristics**
  - If designed properly, they **preserve the beam brightness**

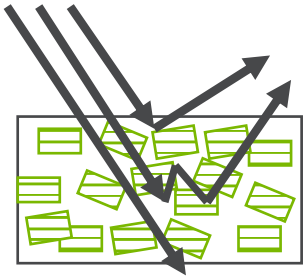
Aside: Up until their work on diffraction of x-rays from crystals. William Bragg thought x-rays were particles or corpuscular in nature. Barkla thought x-rays were an extension of the visible light spectrum.

Apparently there were heated discussions between them. In Compton's 1927 Nobel acceptance speech he states:

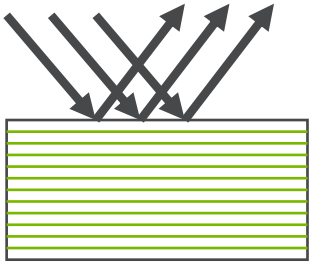
Many will recall also the heated debate between Barkla and Bragg, as late as 1910, one defending the idea that X-rays are waves like light, the other that they consist of streams of little bullets….".

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



Perfect crystal model

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

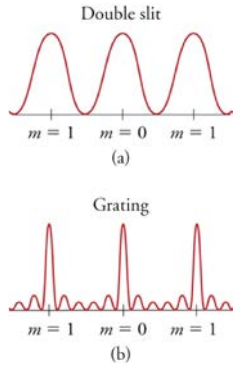
Aside: C. G. Darwin was the first to calculate the index of refractionfor x-rays. Charles G. Darwin was the grandson of the "more famous" Charles Darwin of evolution fame.

- In the case of a strong reflection from a perfect crystal of a monochromatic x-ray beam, the penetration of the x-rays in to 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".)

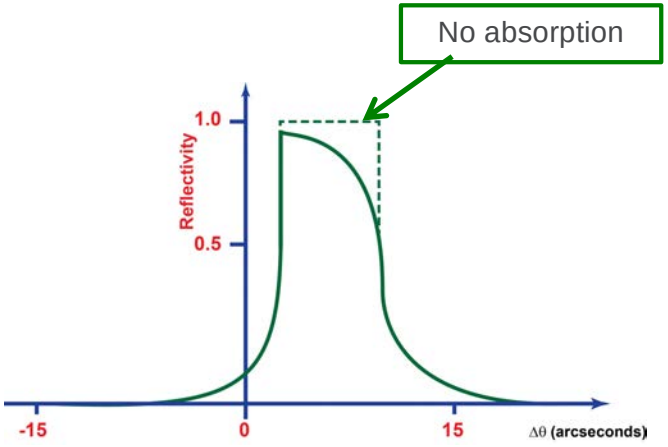
" if the crystal is perfect all the radiation that can be reflected is so, long before the depth at which the rays at a different angle are appreciably absorbed."

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 is often called the ***Darwin width***, ω_D (after Darwin)
  - Depends on the strength of the reflection (hkl) and wavelength.
- 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

V = volume of unit cell

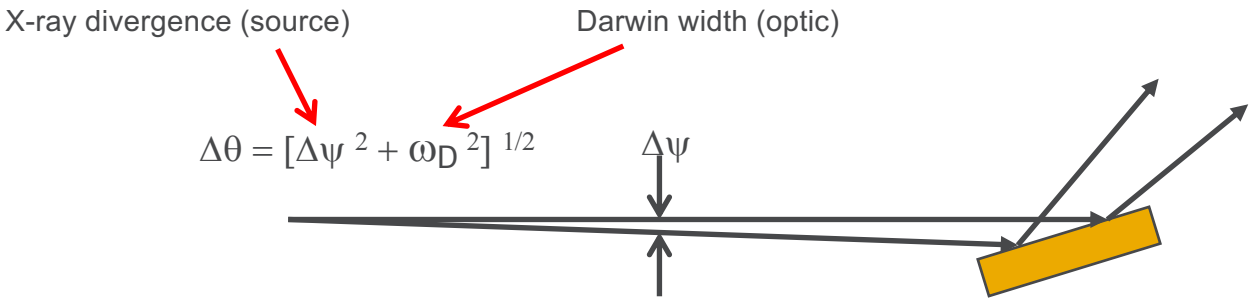
PERFECT CRYSTAL MONOCHROMATORS

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

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

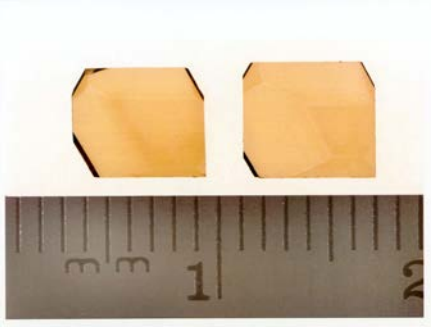
- If we differentiate Bragg's Law ($\Delta\lambda = \cos(\theta)\ \Delta\theta$), divide this by the original equation and recall that $\Delta\lambda / \lambda = \Delta E/E$ for small deltas, then we can determine the energy resolution of the monochromator.

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



- At 8 keV (1.5Å) for Si(111) $\omega_D \approx 40$ microradians. Recall that, for an APS undulator, the opening angle 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 monos as it can be easily obtained and has good mechanical properties for cooling.



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

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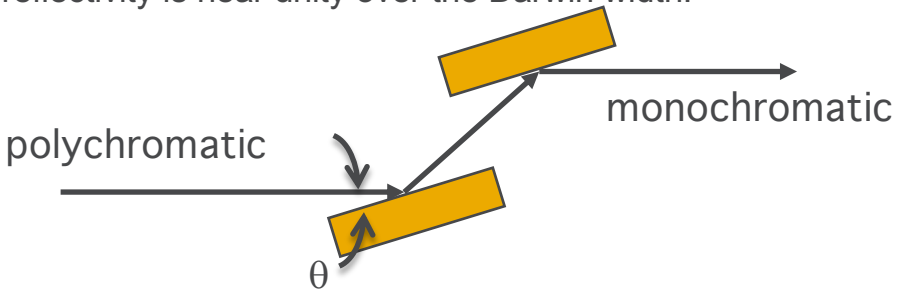
DOUBLE CRYSTAL MONOCHROMATORS

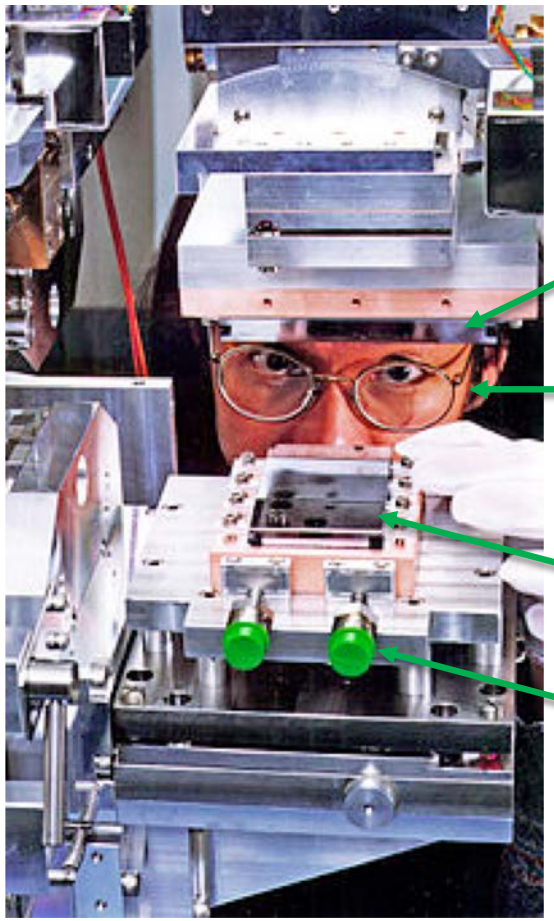
- The most common arrangement for a monochromator is the double-crystal monochromator (DCM). It:
  - is **non-dispersive**, that is all rays that diffract from the first crystal simultaneously diffract from the second crystal (if same crystals with same hkl's are used)
  - **keeps the beam parallel to 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.
  - Silicon monos are often liquid N_2 cooled to enhance thermal properties (higher conductivity and coefficient of thermal expansion goes through a zero at about 120° K).

See Appendix 3 for more information regarding thermal issues for monochromators.



polychromatic beam going into the slide

HIGH ENERGY RESOLUTION X-RAY OPTICS

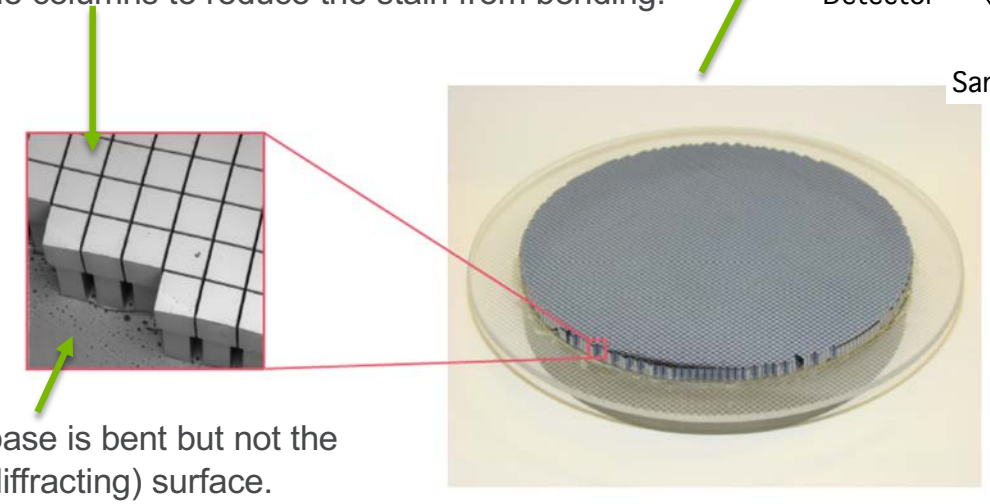
- A Si (111) crystal has an energy resolution $\Delta E/E \approx 10^{-4}$, however milli-electron volt resolution monochromators and analyzers are often required for inelastic scattering.

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

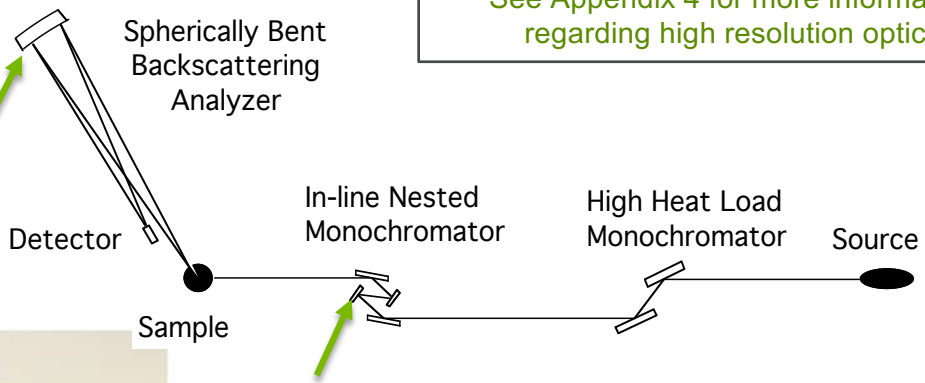
As θ approaches 90°, $\cot(\theta)$ goes to zero and $\Delta E/E$ gets very small and so a backscattering geometry is employed.

See Appendix 4 for more information regarding high resolution optics

The challenge here is to bend the optic into the surface of a sphere to focus the back-scattered x-rays on the detector but keep the crystal diffracting as if it was "perfect" (no strains). So we carefully dice the crystal into little columns to reduce the stain from bending.



The base is bent but not the top (diffracting) surface.



This "in-line nested monochromator" has the two inner crystals in backscattering geometry to provide a high resolution (small $\Delta E/E$) beam incident on the sample but does not need to be bent for focusing so is much easier to fabricate. The two outer crystals in the nested geometry condition the beam from the high heat load mono for better throughput.

MULTILAYER OPTICS

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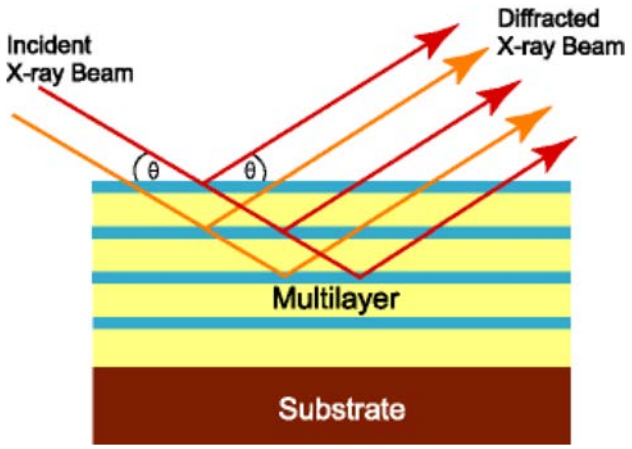
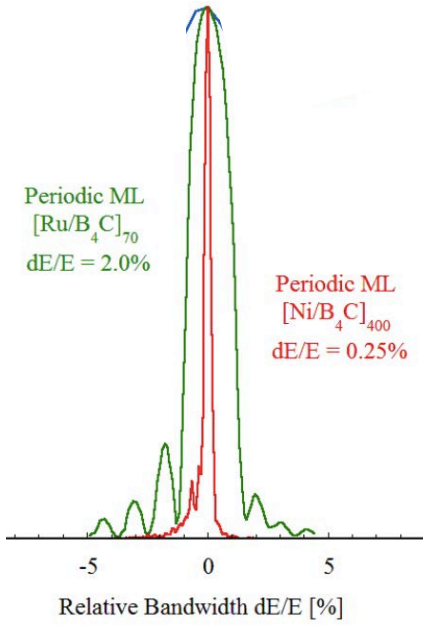
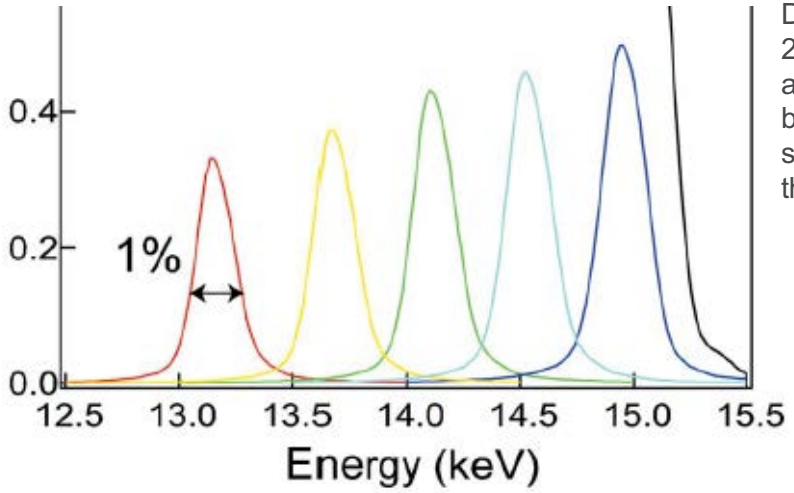
HARD X-RAY MULTILAYER OPTICS

- A “periodic multilayer” coating is a film stack comprising a number of identical repetitions of two or more optically dissimilar component layers.
- 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





Diffracted beam from a W/B_4C (d = 27.7 A) on a Si substrate provides an X-ray spectrum with 1% energy bandwidth. The energy of the X-ray spectrum can be tuned by changing the Bragg angle of the multilayer.

*“100 ps time-resolved solution scattering utilizing a wide-bandwidth X-ray beam from multilayer optics”, Ichiyanagi et al, JSR **16,** 2009.*

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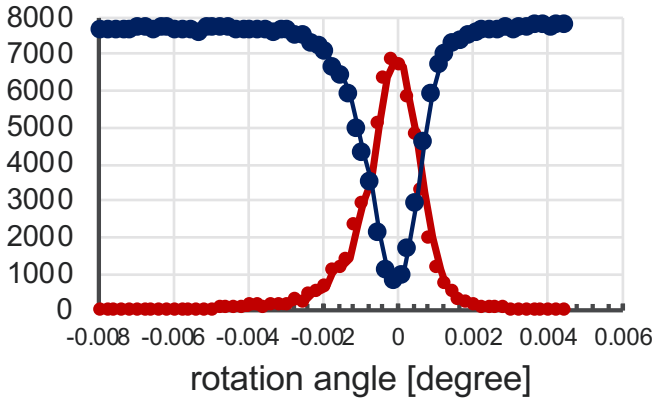
MULTILAYER FABRICATION

- The modular multilayer/mirror deposition (MDS) system at the APS is the world's first thin-film deposition system combining *in-situ* metrology with deposition and ion milling.
- New possibilities for increased performance in APS high energy beamlines.

Mo/B_4C x 250 bilayers
with a AlSi barrier layer on Mo



Reflectance and transmittance curves showing >90% reflectance at >100 keV x-rays.

Modular multilayer/mirror deposition system

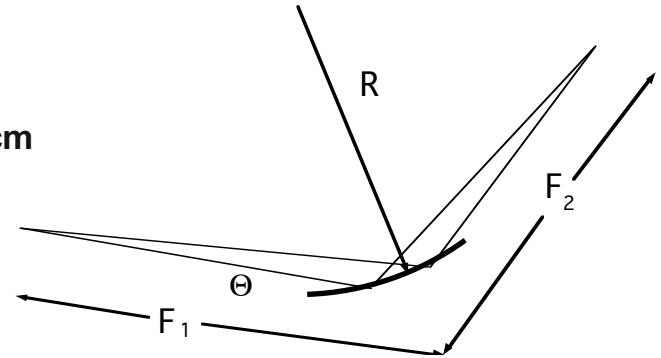
- Substrates as large as 1.5m long, 20 cm wide, and 14 cm thick can be coated.
- A variety of target materials are available.
- Thin film stripping and substrate cleaning capability is available.
- Short turn-around time for many types of coatings.
- Ellipsometry, profilometry, and x-ray reflectometry are used for thin-film characterization.

FOCUSING OPTICS

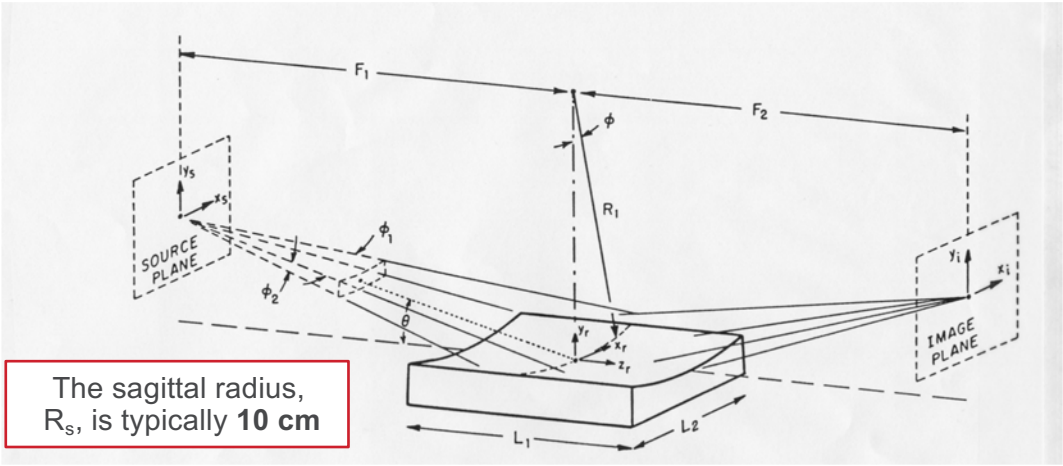
REFLECTIVE FOCUSING OPTICS: MIRRORS

- **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).

Typical meridional radius is around **10 kilometers.**



- **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



The sagittal radius, R_s, is typically **10 cm**

Manufacture of mirrors with so different radii is extremely difficult while maintaining the required slope-errors and surface roughness.

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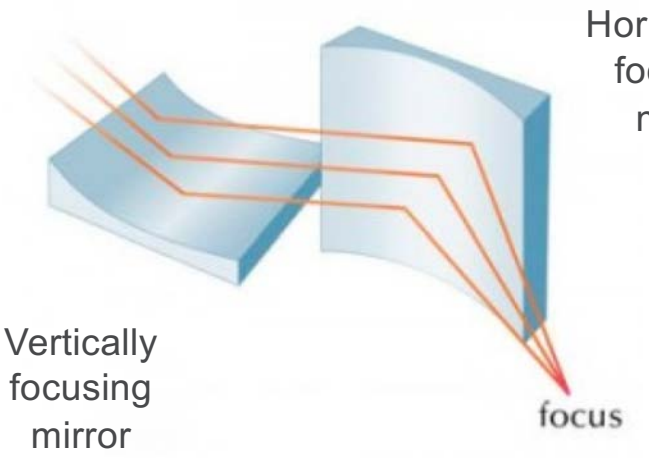
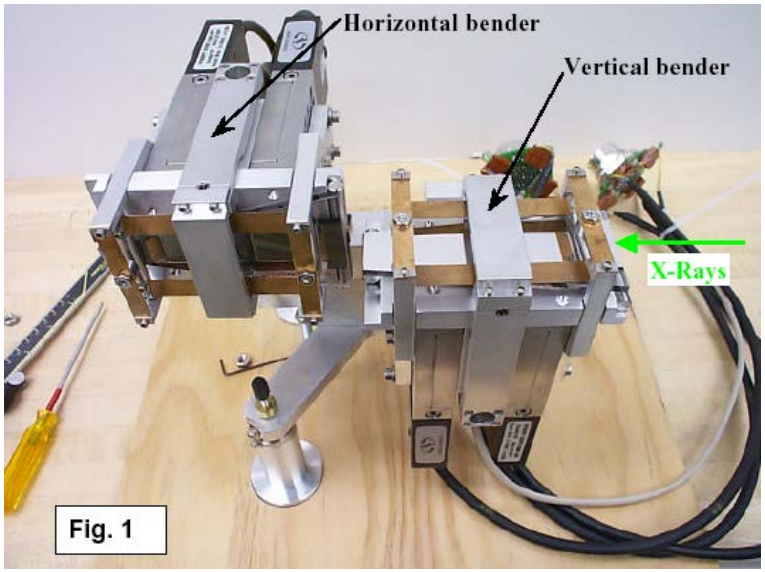
FOCUSING IN TWO DIMENSIONS - KB SYSTEMS

- 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.





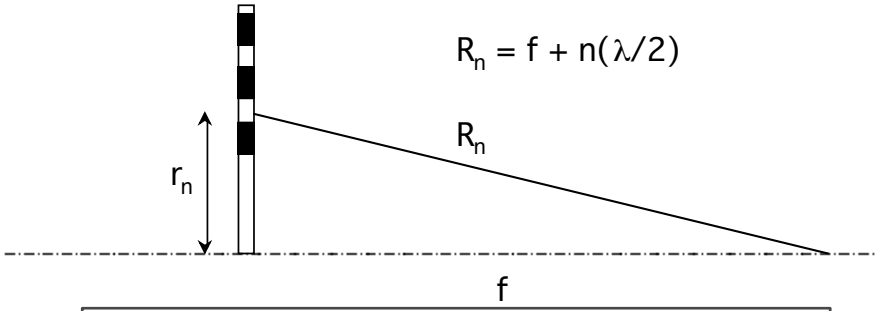
These are achromatic, i.e. the focal length is not dependent on x-ray wavelength.

DIFFRACTIVE FOCUSING OPTICS: X-RAY ZONE PLATES

- Zone plates are diffraction gratings, that is, structures composed of alternating concentric zones of two materials with different (complex) refractive indices.
- 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.

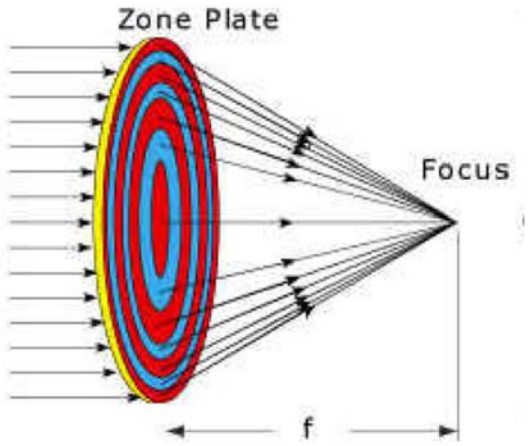
Condition so the pathlength varies by $\lambda/2$ for each ring.

$$R_n = f + n(\lambda/2)$$



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

Cape Meares Lighthouse (Oregon); first-order Fresnel lens



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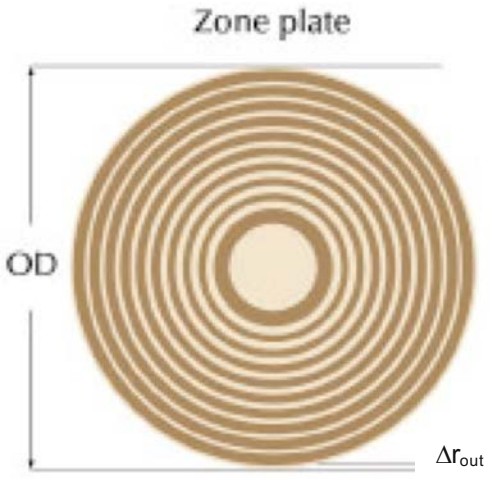
FOCAL SPOT SIZE FOR ZONE PLATES

- Zone plates are usually fabricated with a circular geometry for 2-D focusing.
- In general, the size of the **focal spot from the zone plate, Δx**, is determined by the **width of the outermost ring**, Δr_n , and is given by:

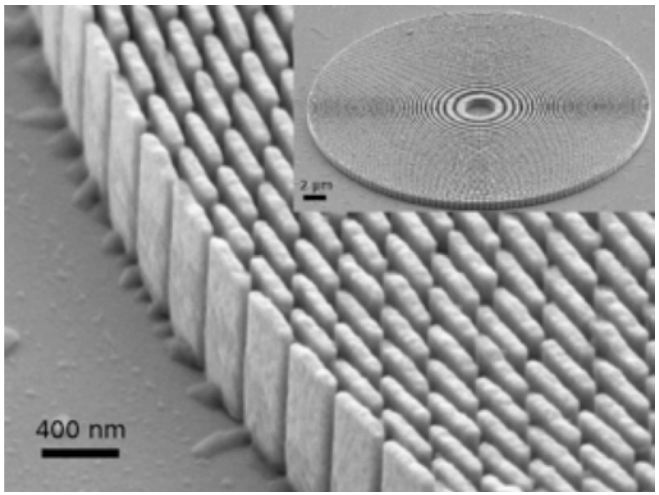
$$\Delta x = 1.22\, \Delta r_n$$

- The challenge in making zone plates for hard x-rays is making Δr_{out} small while maintaining a high thickness for efficiency) i.e. a high aspect ratio.

Zone plate fabrication is an active area of R&D at the APS



Zone plates with outermost ring widths of less than 20 nanometers can currently be fabricated.



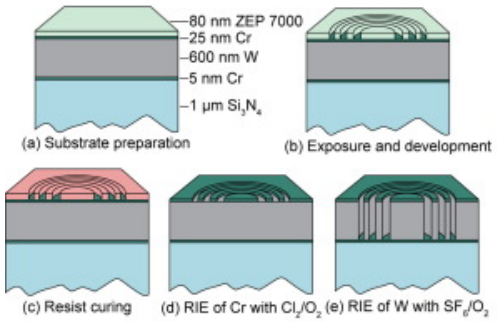
HARD X-RAY PHASE ZONE PLATES

- The difficulty with making zone plates at hard x-ray energies is one of fabrication. You need:
  - small width outermost zone for focusing (less than 50 nms)
  - but it has to be thick (high) to totally absorb the unwanted waves
  - i.e. the aspect ratio (height/width) is very large – 10^2 - and therefore difficult (i.e. impossible) to fabricate

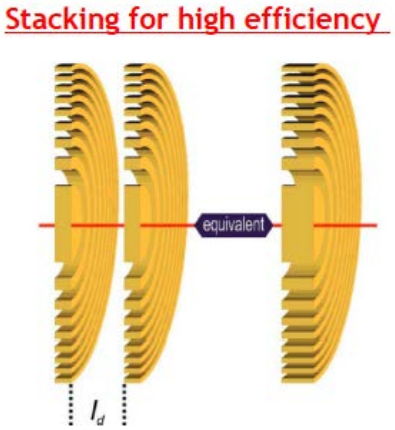
- An alternative to "blocking" out or absorbing those rays that are out of phase (as in an amplitude zone plate), the thickness of the material can be adjusted so that the wave experiences a phase shift of π - these are called **phase zone plates**.

- Phase zone plates have a much better efficiency than amplitude zone plates (10% efficiency for amplitude zone plates vs 40% for phase zone plates).

- The phase zone plates ease the thickness requirement (as compared to the amplitude zone plates) but the aspect ratio is still an issue.



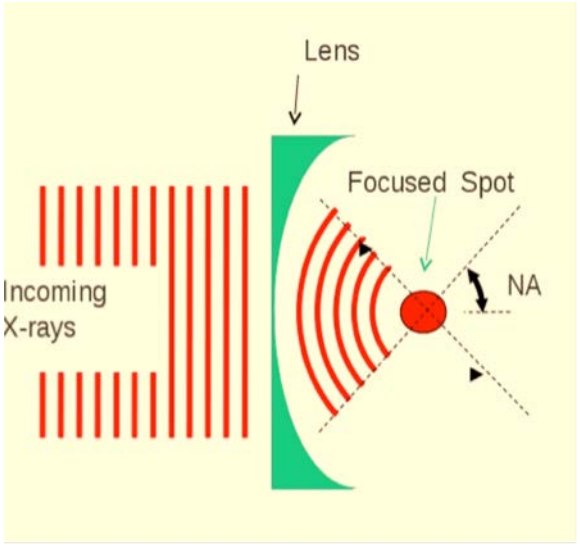
Improved tungsten nanofabrication for hard X-ray zone plates Parfenniukas et al, Microelectronic Engineering Volume 152, 20 February 2016.



At 8 keV, a tantalum ZP with outer most zone of 50 nm would need an aspect ration of 30:1 Multiple zone plates can be "stacked" to increase the effective thickness, but alignment is critical.

REFRACTIVE FOCUSING OPTICS: X-RAY LENSES

- Roentgen's first experiments convinced him that x-rays could not be concentrated by lenses; many years later his successors understood why (index of refraction is very close to 1).
- Refractive lenses were considered for focusing x-rays by Kirkpatrick and Baez in their 1948 paper but were abandoned for crossed mirrors.

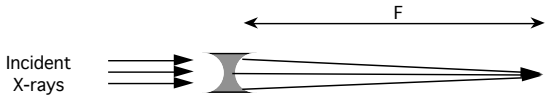


- Unfortunately materials of with large δ's (high electron density) are also strong absorbers, because the absorption coefficient increases much more rapidly than δ with increasing atomic number. Therefore, an element of low atomic number, such as beryllium, is typically used.
- For a single lens with one curved surface: $1/F = \delta(1/R)$ (R is the radius of the curved surface and F the focal length)
- Plugging in some numbers, suppose that:

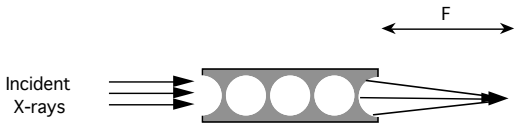
$$R = 1 \text{ mm} \quad \delta \approx 10^{-5}$$

- Then the focal length, F, would be at 100 m!

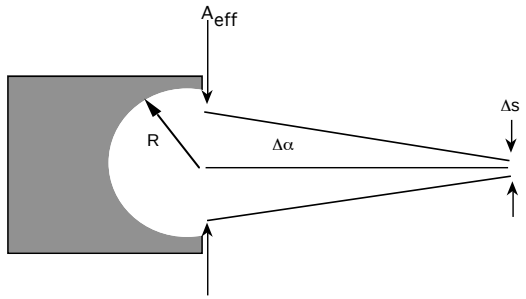
COMPOUND REFRACTIVE LENSES



Single Refractive Lens



Compound Refractive Lens



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

- The Lens Maker's Equation:

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

- For a single lens:

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

this is for a lens with 2 curved surfaces

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

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

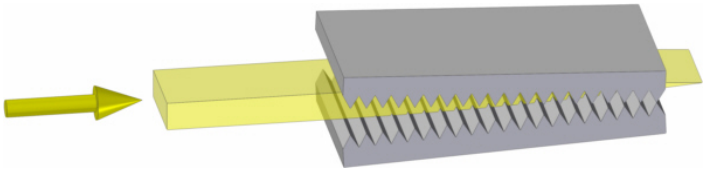
- Using the same numbers as before but with 50 lenses, i.e.:

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

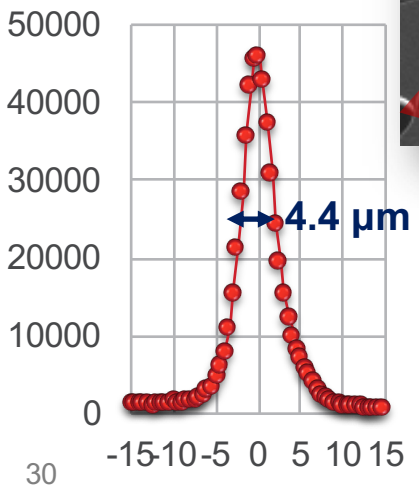
- Then the focal length, F, would be at 1 m.
- These lenses focus at rather larger distances and are well adapted to the scale of synchrotron radiation beamlines.

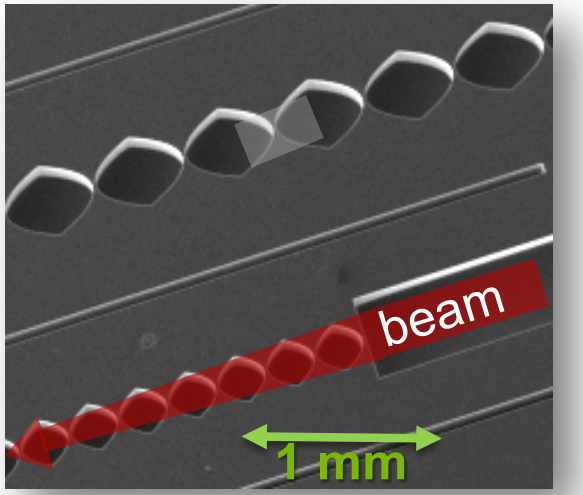
FOCUSING IN ONE DIMENSIONS WITH REFRACTIVE LENSES

- Planar technologies
  - Leverage planar technologies from micro-electronics industry
  - Fabricate compound lens systems in a small space
  - Small radius means moderate focal spots with a single lens or nano-focusing with a moderate number of lenses



Sawtooth lens - The amount of lens material projected on the lateral plane is a (nearly) parabolic profile
Vary opening angle to keep focal length fixed as energy is changed or to vary the focal length

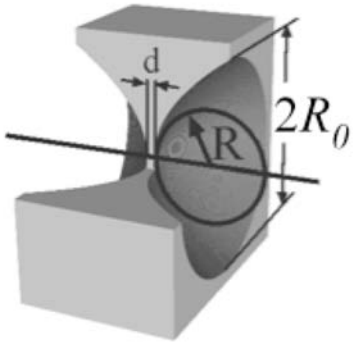


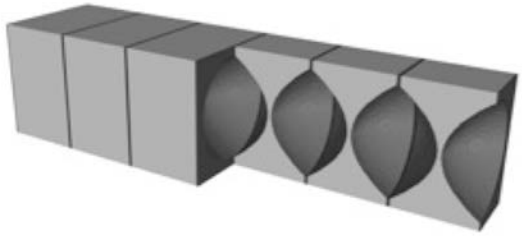


Parabolic lenses etched 400 µm deep into Si wafer made at CNM and tested at APS. The gray shaded area is one lens. At left is focusing performance at 87 keV.

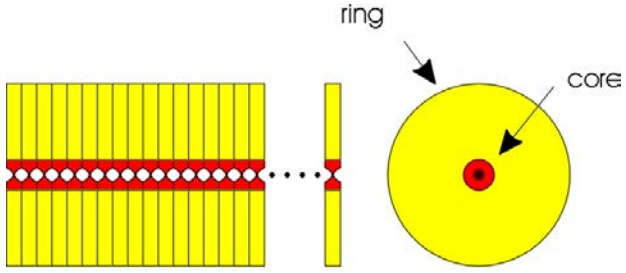
FOCUSING IN TWO DIMENSIONS WITH REFRACTIVE LENSES

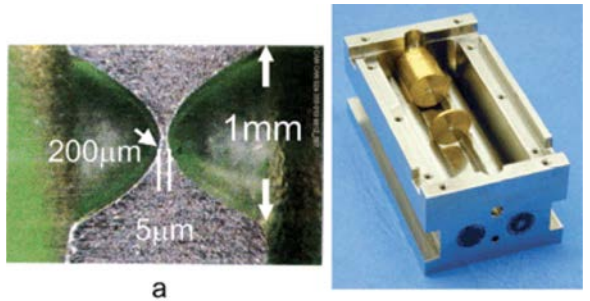
- 2-D lenses typically “embossed” and typically made from Be, Al or Ni
- Spherical lenses are easy to make but suffer from spherical aberrations.
- Paraboloids eliminate spherical aberrations.





Figures from:
http://2b.physik.rwth-aachen.de/xray/imaging/main.php?language=en&frames=&content=crl#Focusingmethods



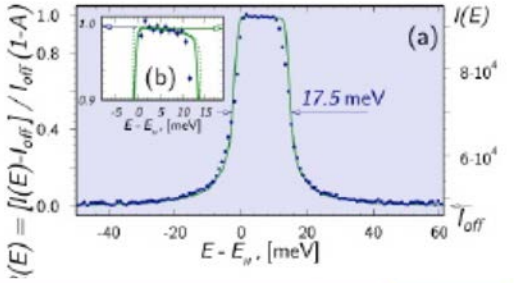


tu-dresden.de/.../isp/skm/research/xray_lenses

http://pubs.rsc.org/is/content/articlehtml/2006/em/b511446m

SUMMARY

- X-ray optics is an active area of research at both universities and national laboratories.
- **New nano-fabrication capabilities** are opening up new possibilities for focusing components such as the fabrication of zone plates, etc.
- High-brightness sources put 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 Stoupin[1], Alessandro Cunsolo[1,2], Ayman H. Said[1] and Xianrong Huang[2]

SCIENTIFIC REPORTS
OPEN

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

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Published: 08 March 2017

Istvan Mohacsi[1,2], Ismo Vartiainen[1,3], Benedikt Rösner[1], Manuel Guizar-Sicairos[1], Vitaliy A. Guzenko[1], Ian McNulty[4], Robert Winarski[4], Martin V. Holt[4] & Christian David[1]

LETTERS
PUBLISHED ONLINE: 22 NOVEMBER 2009 | DOI: 10.1038/NPHYS1457
nature physics

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

Hidekazu Mimura[1]*, Soichiro Handa[1], Takashi Kimura[1], Hirokatsu Yumoto[2], Daisuke Yamakawa[1], Hikaru Yokoyama[1], Satoshi Matsuyama[1], Kouji Inagaki[1], Kazuya Yamamura[3], Kenji Tamasaku[4], Yoshinori Nishino[4], Makina Yabashi[4], Tetsuya Ishikawa[4] ar

JSR JOURNAL OF SYNCHROTRON RADIATION
ISSN: 1600-5775

A middle energy-bandwidth X-ray monochromator for high-flux synchrotron diffraction: revisiting asymmetrically cut silicon crystals

Hiroo Tajiri,[a,c]* Hiroshi Yamazaki,[a,c] Haruhiko Ohashi,[a,c] Shunji Goto,[a,c] Osami Sakata[b,c] and Tetsuya Ishikawa[c]

Appl Cryst JOURNAL OF APPLIED CRYSTALLOGRAPHY
ISSN: 1600-5767

Correct interpretation of diffraction properties of quartz crystals for X-ray optics applications. Corrigendum

Xian-Rong Huang,[a]* Thomas Gog,[a] Jungho Kim,[a] Elina Kasman,[a] Ayman H. Said,[a] Diego M. Casa,[a] Michael Wieczorek,[a] Marcelo G. Hönnicke[b] and Lahsen Assoufid[a]

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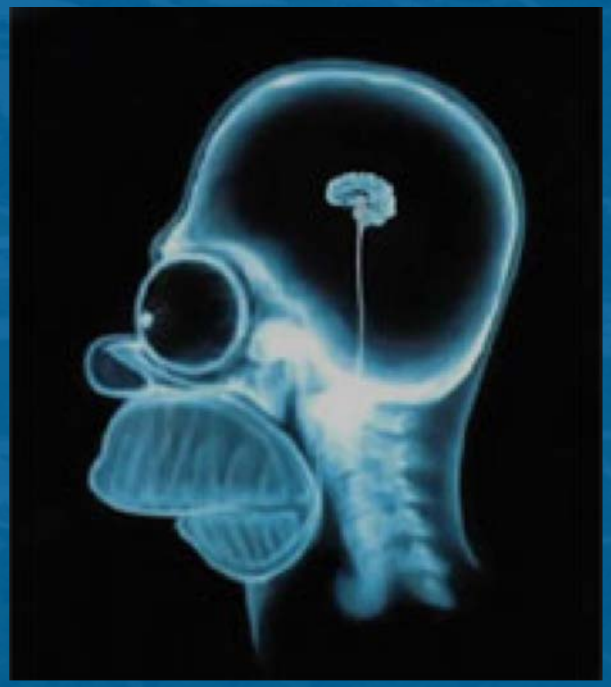
- *Report of the Basic Energy Sciences Workshop on X-ray Optics for BES Light Source Facilities*, Dennis Mills and Howard Padmore, Co-Chairs, March 27-29, 2013 (http://science.energy.gov/~/media/bes/pdf/reports/files/BES_XRay_Optics_rpt.pdf)

Soft X-ray Optics

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

DETECTORS



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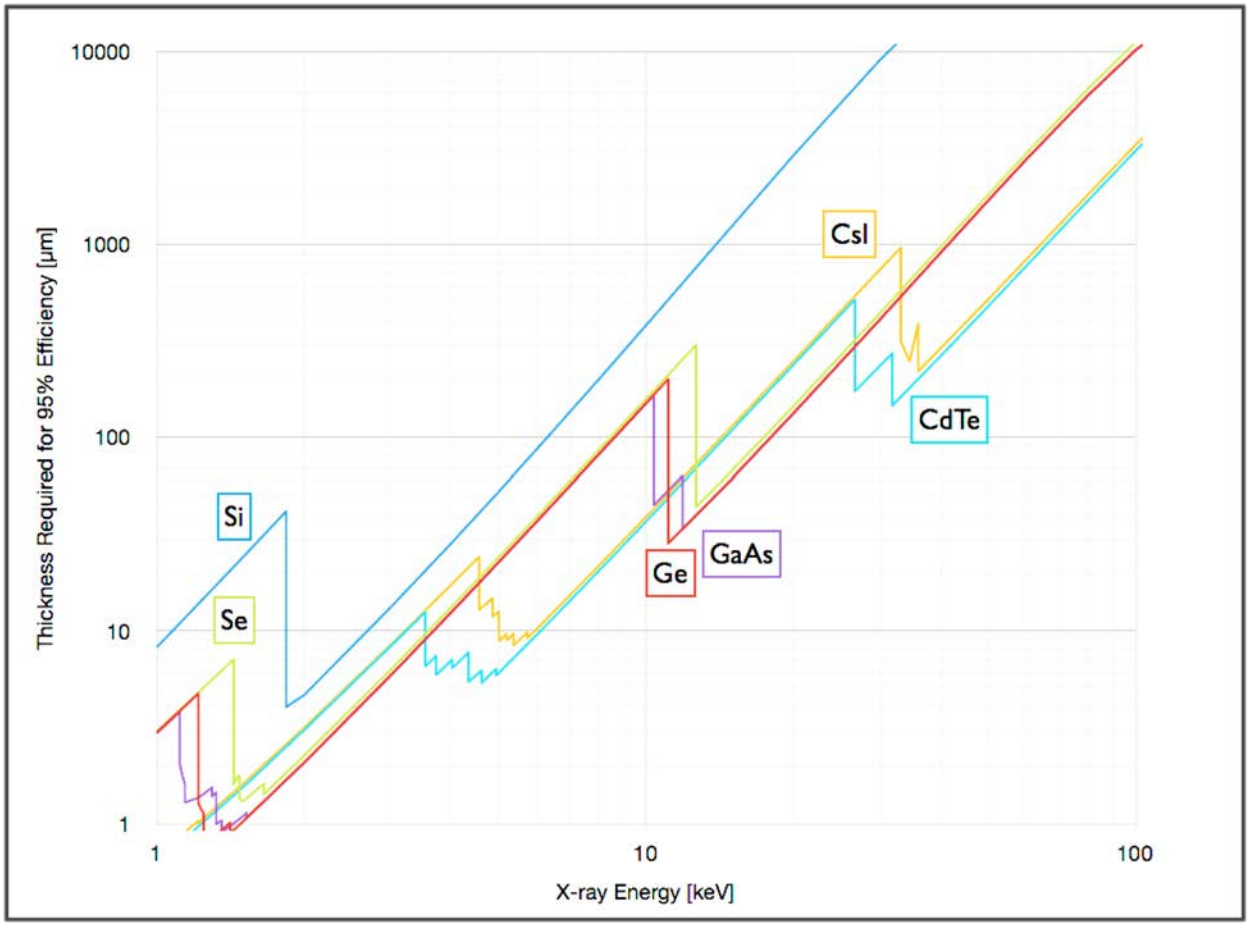
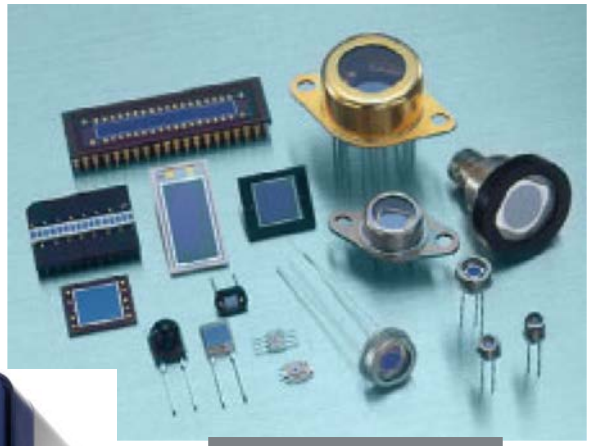
DETECTION OF PHOTONS

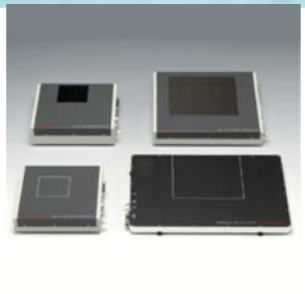
- Photons can only be “detected” by registering the deposition of energy in the detecting medium.
- Therefore, inelastic scattering processes (i.e., those that deposit energy) are relevant.
  - photoelectric effect
  - Compton scattering
  - pair (e-, e+) production

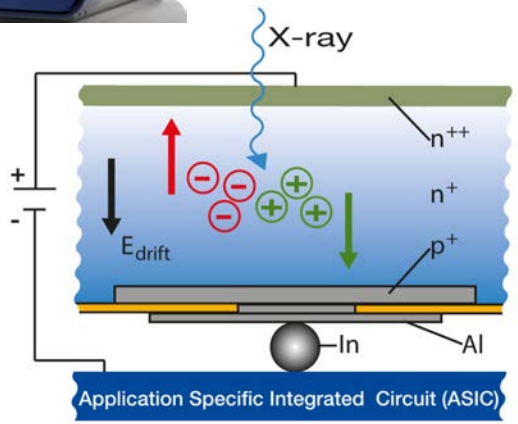


DETECTOR PROPERTIES

- We would like detectors to have ***all*** of the following characteristics (but they never do):
  - high efficiency (count every photon or make every photon count)
  - high count rate capabilities
    - single photon counting detectors that can "process" one x-ray at a time very fast or
    - integrating detectors that accumulate (integrate) over a fixed period of time, and the detector output is proportional to the total number of x-rays detected)
  - good spatial resolution for 1D (linear) or 2D(area) detectors - a few microns would be nice but that is very hard
  - energy resolution (signal proportional to energy of the incident x-ray)





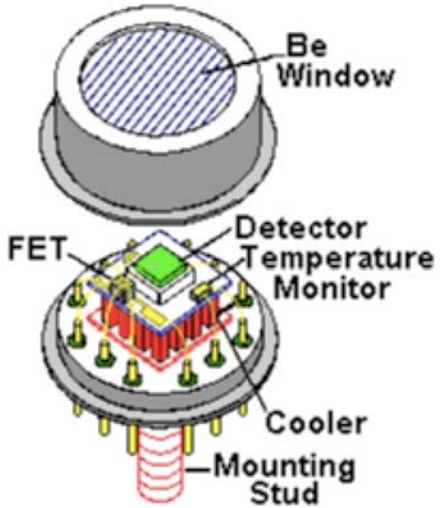


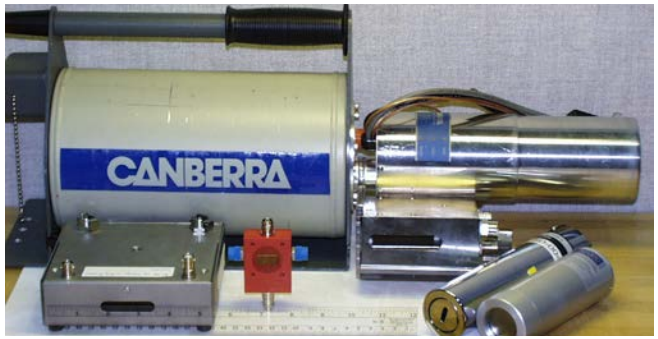
ENERGY RESOLVING DETECTORS - SSDs

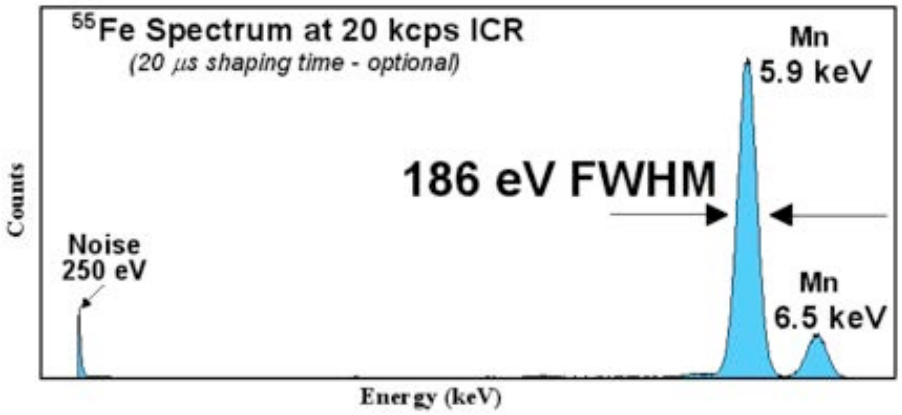
- Solid state detectors (SSDs) are basically large reverse-biased pin diodes that collect the electrons (holes) that are made as the x-ray is absorbed in a biased diode.

- They have quite good energy resolution ($\Delta E/E$ of 3-5%) since the energy required to generate and electron-hole pair is on the order of 3 eV in silicon and germanium.

- To keep the leakage currents as low as possible, SSDs are typically cooled to near liquid nitrogen temperature.

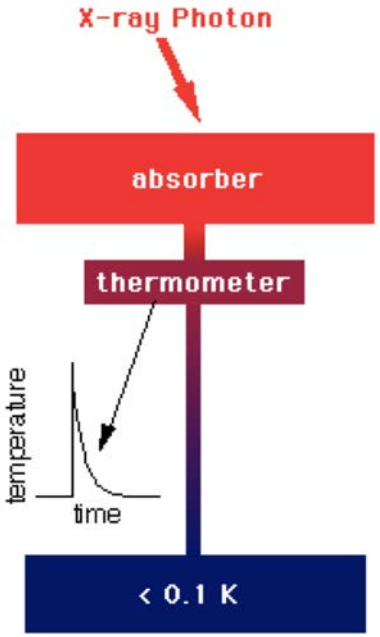


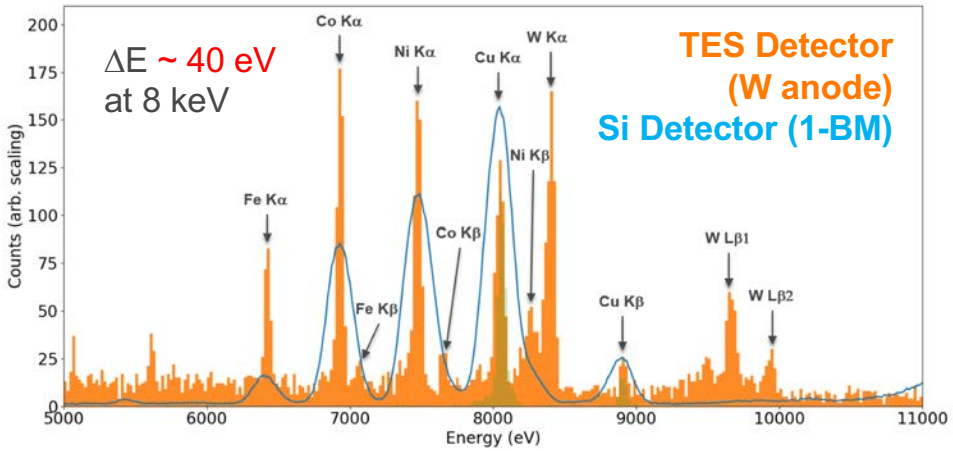




ENERGY RESOLVING DETECTORS – TES

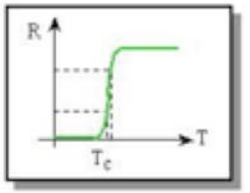
- Transition edge superconducting (TES) detectors
  - Currently under development, they work at less than a degree above absolute zero but have energy resolutions 3-15 times better than SSDs (but are very slow).
  - To get around the low count rates assemble arrays of individual detectors.
  - The sensor array shown at the bottom is a Mo/Cu bilayer on a SiN suspended membrane (developed at APS) that operates at T_C~100 mK.





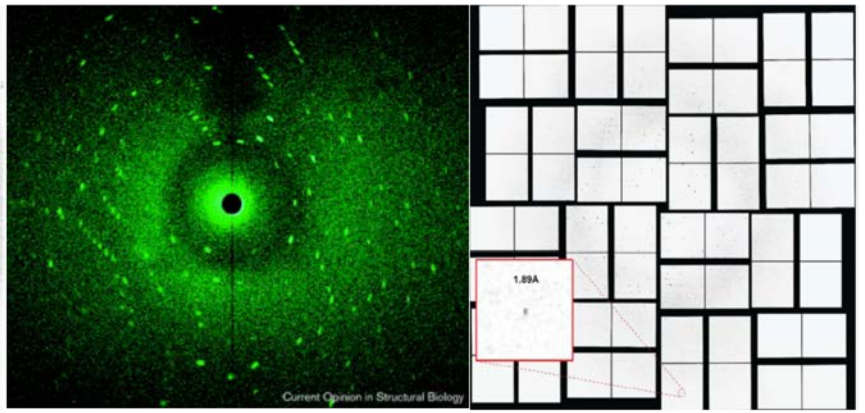
The TES is biased on the superconducting transition to maximize α, a measure of the sensitivity of the device.

$$\alpha = \frac{T}{R}\frac{\partial R}{\partial T}$$

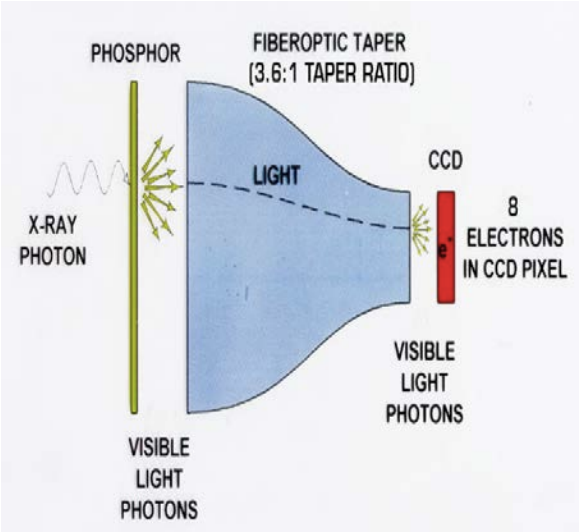


AREA DETECTORS - CCDs

- The original area detector was x-ray sensitive film.
- Now a days, all area detectors are electronic.
- Charge Coupled Detectors (CCDs)
  - These are basically the same devices that are in your phone:
    - a larger number of pixels (4096 x 4096)
    - smaller pixel size (10 microns)
    - higher resolution readout (16 bits)
  - To get a large area, can be tiled or coupled to a tapered fiber optic with a scintillator



(b) (c)



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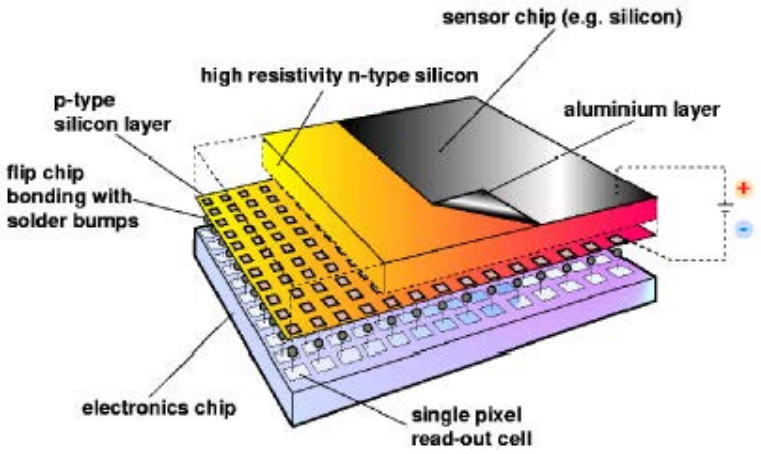
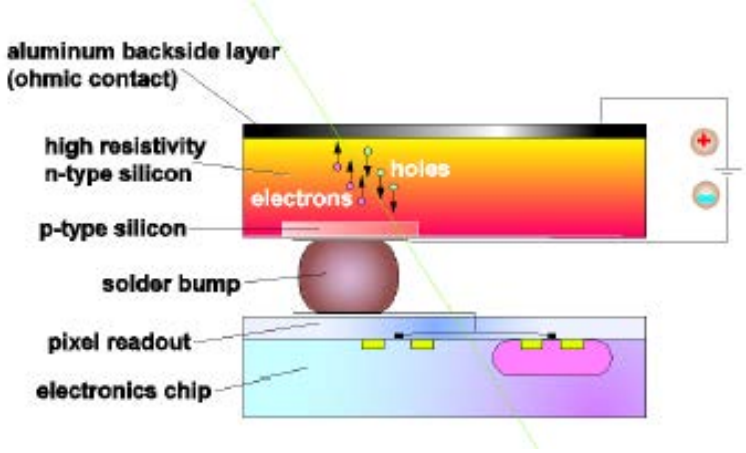
AREA DETECTORS - (PADS)

- In a pixel array detector (PAD) each pixel is a stand alone detector (usually a diode) that has its own electronics.

- Electronics can be tailored to the specific needs of the experiment via application specific integrated circuits (ASICs)
  - single channel analyzers
  - autocorrellators
  - local background subtraction
  - lock-in techniques

- High framing rates possible with built in memory, but total number of frames limited





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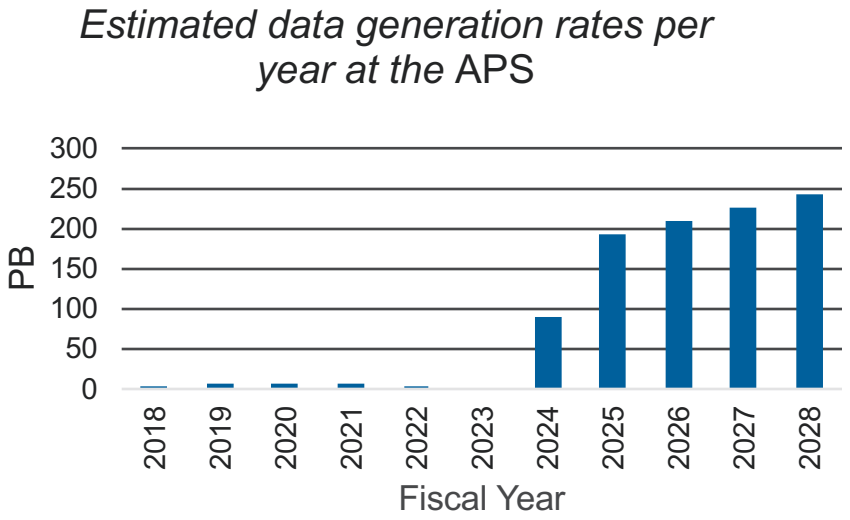
SUMMARY

- Sources have become immensely more powerful and are now brighter (more particles focused onto the sample per second) and more precise (higher spatial, spectral, and temporal resolution). Consequently, detectors must become faster, more efficient, and more discriminating.

- Unfortunately, in the U.S., a distinct lag in detector development is slowing progress toward interpreting what we're seeing. Urgently needed detector improvements would reveal chemical composition and bonding in 3-D and in real time, allow researchers to watch "movies" of essential life processes as they happen, and much more efficiently use every X-ray and neutron produced by the source.

- However, even with current high frame-rate, megapixel detectors, users are being swamped with huge data sets!

AND WHAT DO YOU DO WITH ALL THAT DATA?



That's the topic of another lecture...(or should be!).

QUESTIONS

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APPENDIX 1A: DIELECTRIC CONSTANT AND THE DRUDE MODEL

The dielectric constant, κ, is defined as follows:

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

For a single electron: $P = -ex$

and for multiple electrons: $P = -exn_e$ (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:

$$\omega_o = [4\pi n_e e^2/m]^{1/2} .$$

If we assume a simple harmonic approximation then:

$$F = ma = mx'' = -eE - kx$$

where k is the "spring constant" associated with ω_o (= $[k/m]^{1/2}$).

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APPENDIX 1B: X-RAY INDEX OF REFRACTION

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

$$x = (e/m)E/(\omega_o^2 - \omega^2) \quad \text{and} \quad P = -(e^2/m)n_e E/(\omega_o^2 - \omega^2)$$

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

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

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

$$\omega_o = \mathbf{5 \times 10^{16}/sec}$$

For a 1 Å x-ray, the angular frequency, ω (= $[2\pi c/\lambda]$), is **2×10^{19}/sec (>> ω_o)** and so we can write:

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

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

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APPENDIX 2A: INCLUSIONS OF ABSORPTION IN THE (COMPLEX) INDEX OF REFRACTION

This simple model did not include any absorption of the incident radiation. A more detailed calculation would result in an expression:

$$n = 1 - \delta - i\beta$$

where $\delta = (n_e r_e/2\pi)\lambda^2$ and $\beta = \lambda\mu/4\pi$, with μ the linear absorption coefficient ($I = I_o e^{-\mu t}$).

APPENDIX 2B: INDEX OF REFRACTION FOR X-RAYS IS <1

OK, isn't $V_{group} = (c/n)$? If n < 1, doesn't that mean the x-rays are traveling faster than the speed of light? **NO!**

$$V_{group} = \frac{d\omega}{dk} \quad \text{and} \quad \omega = \frac{ck}{n} \quad \text{so} \quad V_{group} = \frac{d}{dk}\left(\frac{ck}{n}\right); \quad k = \frac{2\pi}{\lambda} \quad n = 1 - \frac{2\pi n_e r_e}{k^2}$$

$$V_{group} = \frac{d}{dk}\left[\frac{ck}{1 - \frac{2\pi n_e r_e}{k^2}}\right] \approx \frac{d}{dk}\left[ck\left(1 + \frac{2\pi n_e r_e}{k^2}\right)\right] = c\frac{d}{dk}\left[\left(k + \frac{2\pi n_e r_e}{k}\right)\right] = c\left(1 - \frac{2\pi n_e r_e}{k^2}\right)$$

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APPENDIX 3A: THERMAL LOADING ON OPTICS

- 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.

Process	Approx. Heat Flux (W/mm^2)
Fission reactor cores	1 to 2
Interior of rocket nozzle	10
Commercial plasma jet	20
Sun's surface	60
Fusion reactor components	0.05 to 80
Meteor entry into atmosphere	100 to 500
APS insertion devices (2.4 m and 100 mA)	**10 to 160**

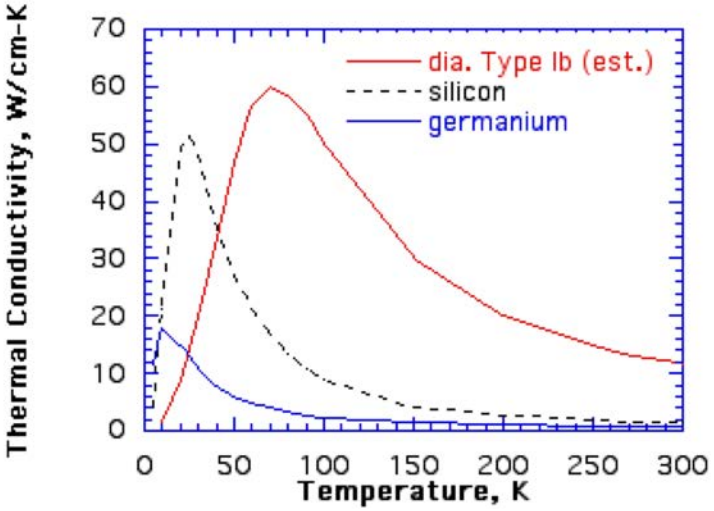
- In order to maintain the beam intensity and collimation (i.e., brilliance) through the optics, special attention must be paid to the issue of thermal management.

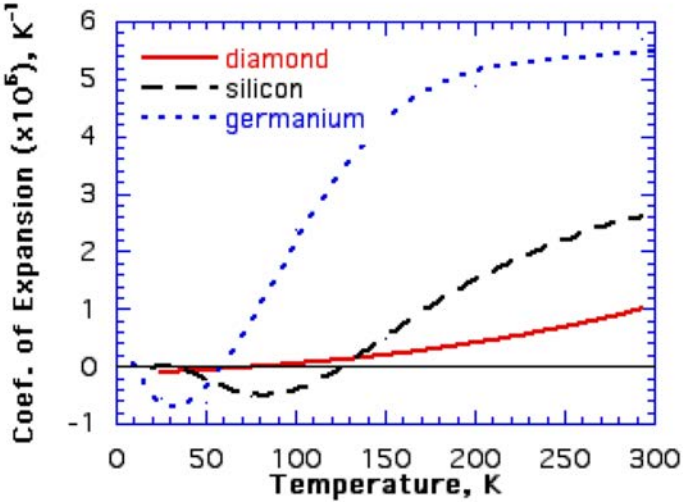
APPENDIX 3B: PROPERTIES OF SI, GE, AND C(DIAMOND)

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

$$\alpha \Delta T = \Delta d/d = \cot(\theta)\, \Delta\theta = \cot(\theta)\, \omega_D.$$

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.





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APPENDIX 3C: FIGURE OF MERIT (FOM) FOR VARIOUS MATERIALS AND TEMPERATURES

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

FOM of various materials

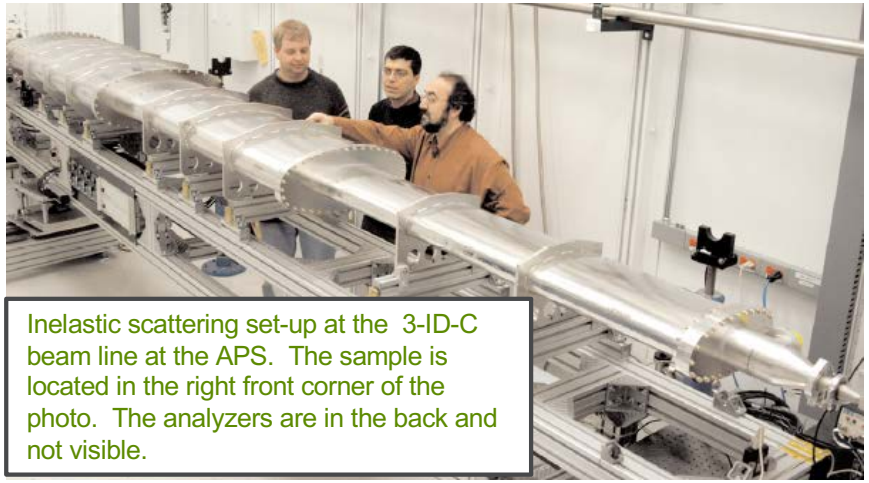
material	k - thermal conductivity	α - coef. of thermal expansion	k/α FOM
Si (300° K)	1.2 W/cm-° C	2.3×10^{-6} /° K	0.5
Si (78° K)	14 W/cm-° C	-0.5×10^{-6} /° K	28
Dia. (300° K)	20 W/cm-° C	0.8×10^{-6} /° K	25

APPENDIX 4: HIGH ENERGY-RESOLUTION OPTICS

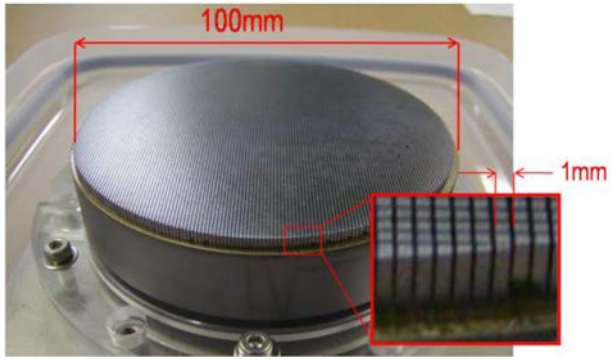
At $\theta = 89°$, $\cot(\theta) = 1.7 \times 10^{-2}$. For E = 20 keV (0.64Å), then:

$$\Delta E = E \cot(\theta)\Delta\theta = (2 \times 10^4 \text{ keV})(1.7 \times 10^{-2})(10^{-5}\text{rad})$$
$$= 3 \times 10^{-3} \text{ eV}.$$

Note: For Si (111) at a Bragg angle of $\theta = 89°$, the wavelength is 6.2Å (2 keV) and so to get near 20 keV at $\theta = 89°$, we need to use a very high d-spacing such as Si (11 11 11).

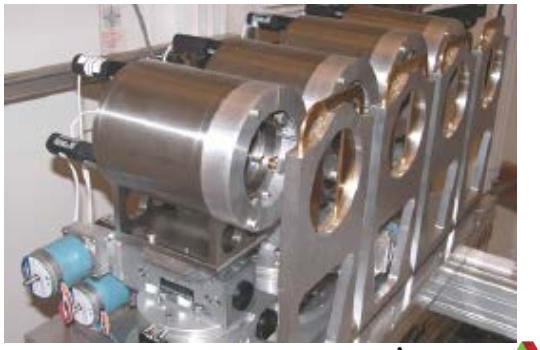


Inelastic scattering set-up at the 3-ID-C beam line at the APS. The sample is located in the right front corner of the photo. The analyzers are in the back and not visible.



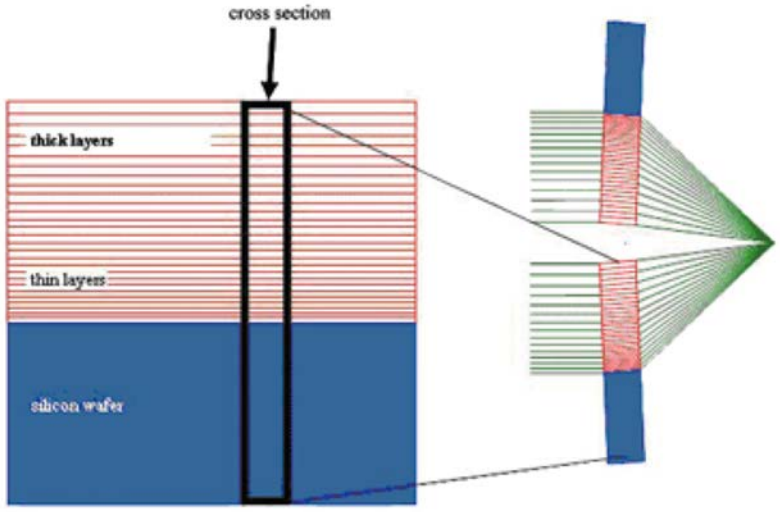
A diced, high energy resolution inelastic x-ray spherical analyzer.

The high energy resolution inelastic x-ray (HERIX) beamline at the APS with an array of analyzers.



A NEW APPROACH TO FABRICATING ZONE PLATES – MULTILAYER LAUE LENSES (MLLS)

Start with a linear zone plate geometry and then use a Kirkpatrick-Baez configuration to get focusing in both direct

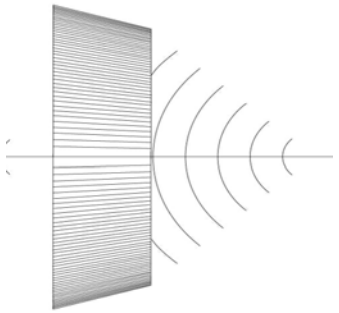


Using state-of-the-art deposition techniques, start with the thinnest layer first and fabricate a multilayer structure with the layer spacing following the Fresnel zone plate rule.

Slice and polish the multilayer structure to get a linear zone plate.

Each MLL comprises 1,588 layers (lines)
The thinnest layer (line) is 5 nanometers thick

The MLL has a current focus of 11 nanometers at 12 keV and 16 nanometers @ 19 keV!



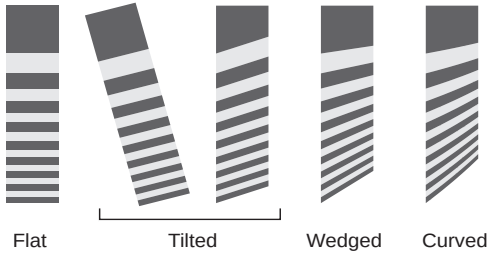
Wedged MLL

H. C. Kang, J. Maser, G. B. Stephenson, C. Liu, R. Conley, A. T. Macrander, and S. Vogt, "Nanometer Linear Focusing of Hard X Rays by a Multilayer Laue Lens," Phys. Rev. Lett. 96, 127401 (2006).

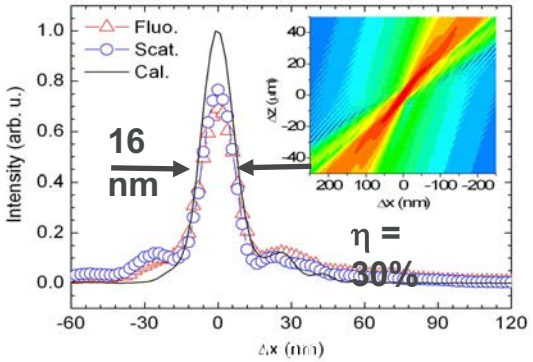
MULTILAYER LAUE LENSES

Technical approach:

Crossed multilayer-based linear zone plate structure

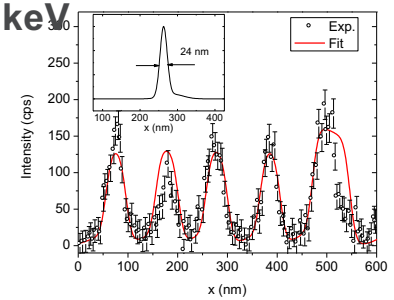


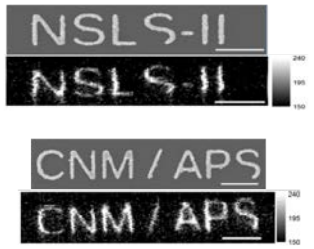
1-D focus: 16 nm (half MLL) @ **19.5 keV**



Kang et al, APL 92, 221114 (2008)

2-D focus: 25 x 40 nm^2 (2D, crossed MLL) @ **20 keV**





2-D imaging with crossed MLL (ANL/BNL collaboration)

Yan et al, H. Yan et al, Opt. Exp. 2011