PSI Center for Photon Science

Materials Science at Large Scale Facilities

Beamlines and detectors

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EPFL course MSE-435

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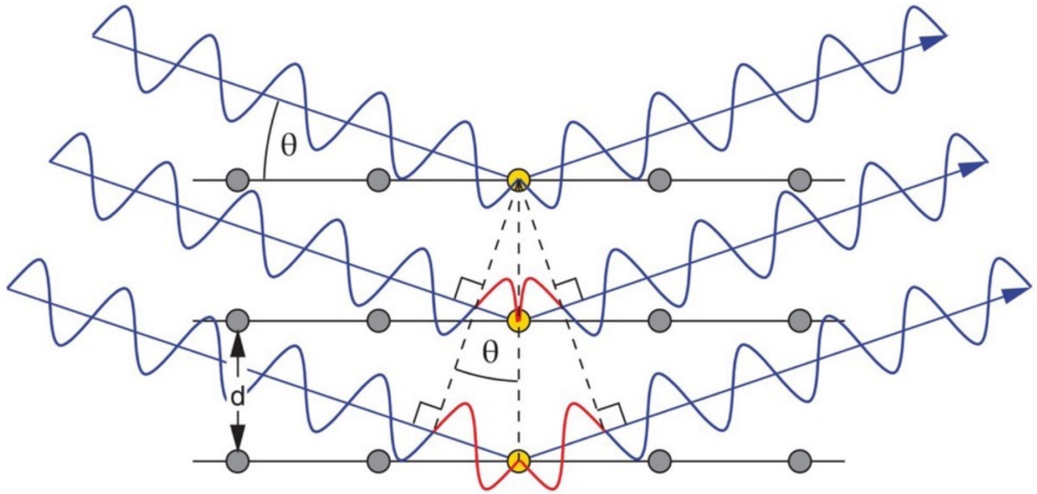
Bragg's law

The Bragg law describes interference between rays elastically scattered off successive atomic planes, separated from one another by a distance *d*.

When the optical path difference between adjacent rays is an integer multiple *m* of the x-ray wavelength, interference is constructive, and a diffraction peak will be seen at that angle.



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

Neutron beam lines

- Neutron sources are **large c**ompared to sample size *(100 cm^2 vs 1 cm^2)*
- Neutrons sources are **weak**
- Neutron beams are **divergent** *(beam divergence in the range of 0.2° to 1°)*
- Neutron beams are **”polychromatic”** *(wide energy range, e.g. thermal range: 5-80 meV)*
- Neutron beams are **not polarized**

Neutron beam lines

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- We need to transport neutrons to the experimental area (guides)

  → Mirror & Super-Mirror : Flux, divergence, neutrons with wavelengths in the right range

- We need to determine incident and scattered neutron directions

  → Collimator : Angular resolution

- We need to select out unwanted neutrons

  → Filters

- We need to determine incident and scattered wavelengths (Energies)

  → Crystal Monochromator : Wavelength resolution (Energy resolution)

- We need also to polarize neutron beams

  → Crystal/ Super-Mirror/ ^{3}He spin filter : Orientation of the neutron spin

Neutron optics

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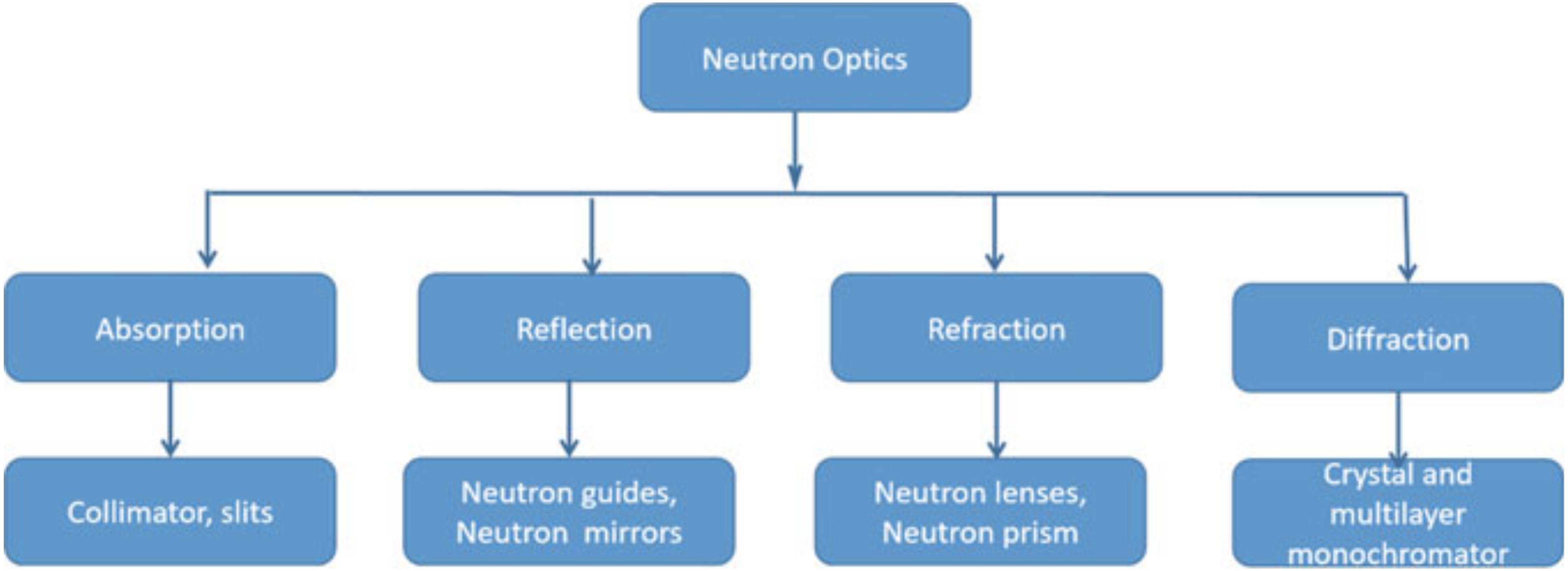
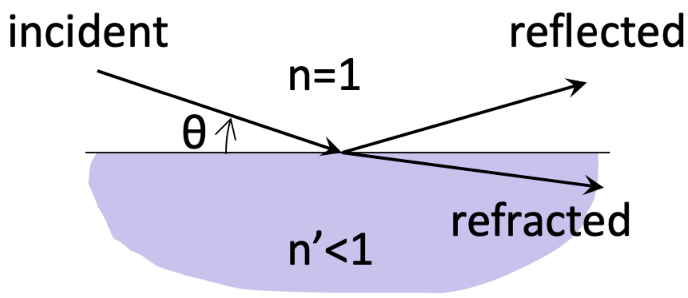


Neutron mirrors



critical angle of total reflection θ_c

$$\left.\begin{aligned} \cos\theta_c &= n'/n = n' \\ n' &= 1 - \frac{N\lambda^2 b}{2\pi} \\ \cos\theta_c &\approx 1 - \theta_c^2/2 \end{aligned}\right\} \Rightarrow \theta_c = \lambda\sqrt{Nb/\pi}$$

Definition:

$$Q = 4\pi \sin\theta / \lambda$$

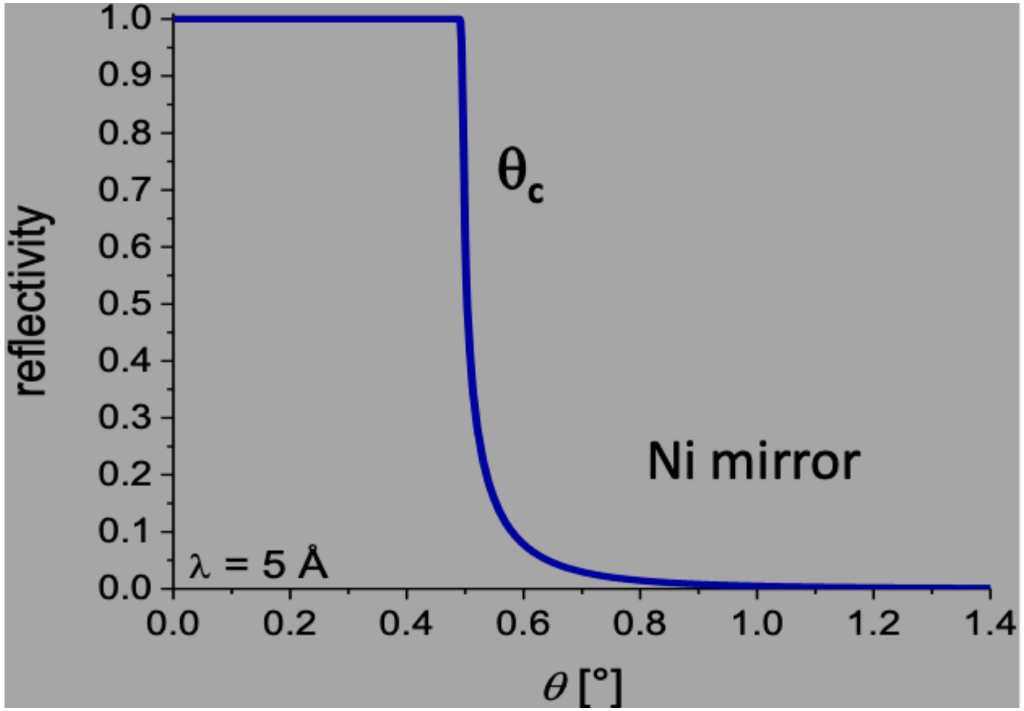
for natural Ni:

$\theta_c = \lambda[\text{Å}]\times 0.1°$

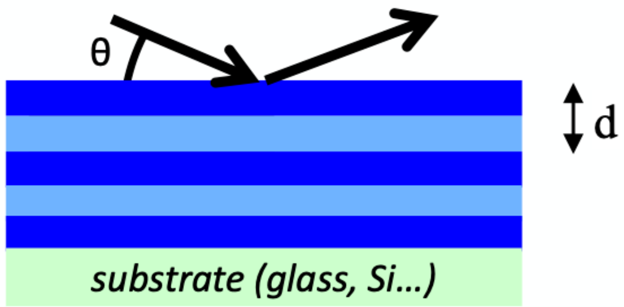
$Q_c = 0.0218$ Å^{-1}

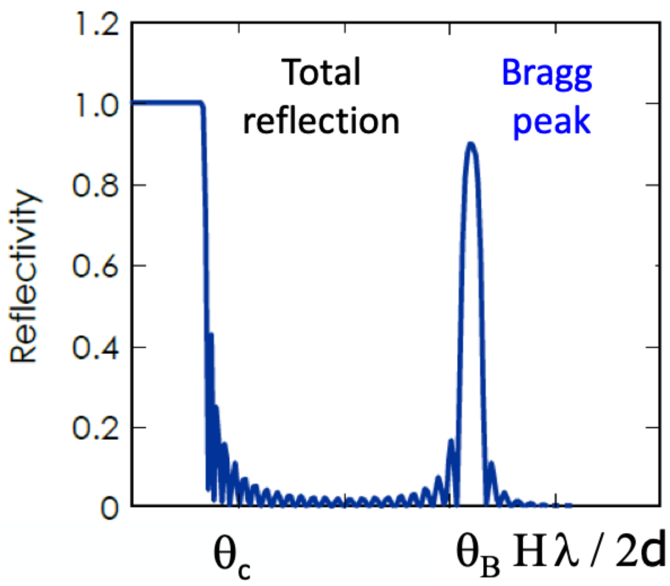
Ken Andersen, Neutron Guides, 2015

Neutron mirrors



Periodic multilayers

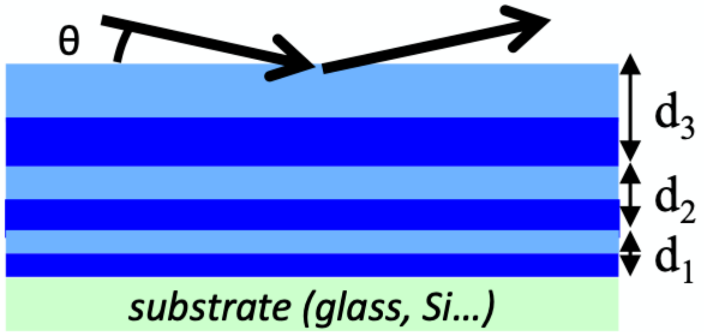




Total reflection for $\theta < \theta_c$

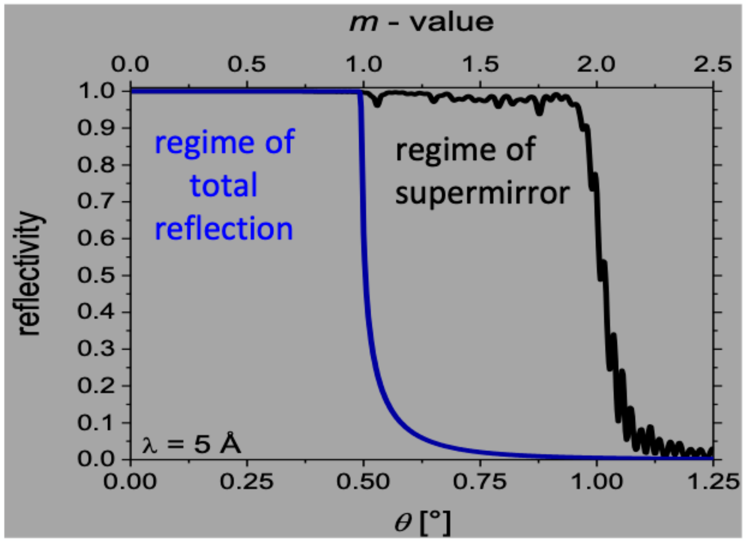
Additional Bragg peak at $\theta_B \approx \lambda / 2d$

Super-mirror



Sequence of bi-layers of variable thicknesses d

Total reflection for $\theta < \theta_c$ + additional Bragg peaks

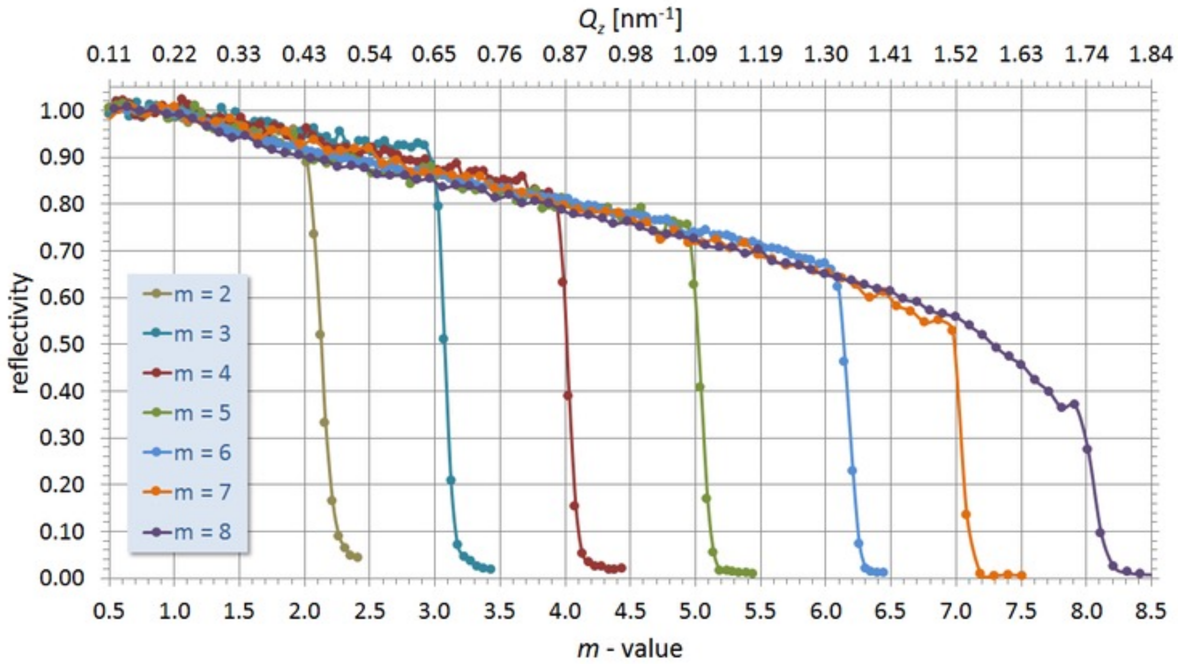


→ significant increase in critical angle

$$\theta_c = \lambda / 2d_{min}$$

Super-mirrors

- m value super mirror: $m = Q_c^{SM} / Q_c^{Ni}$
- Gain in Neutron flux ~m^2



Ni/Ti super-mirror

Ni/Ti super mirrors

Neutron guide can transport neutrons over long distances with limited loss

Monochromator

- Use of single crystals to select a given wavelength band according to the Bragg's Law

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

- The relative bandwidth $\Delta\lambda/\lambda$ is given for Gaussian distributions by:

$$\Delta\lambda/\lambda = (\alpha^2+\beta^2)^{1/2}\cot(\theta_B)$$

  - *α : divergence of the primary beam*
  - *β : full width at half maximum of the neutron "rocking curve" or neutron mosaic spread*

Monochromator

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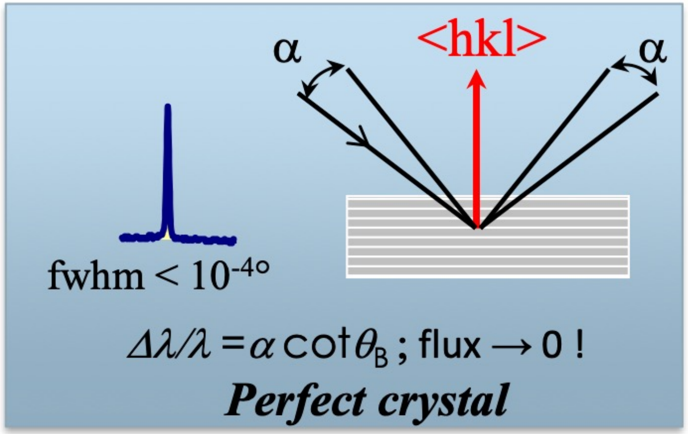
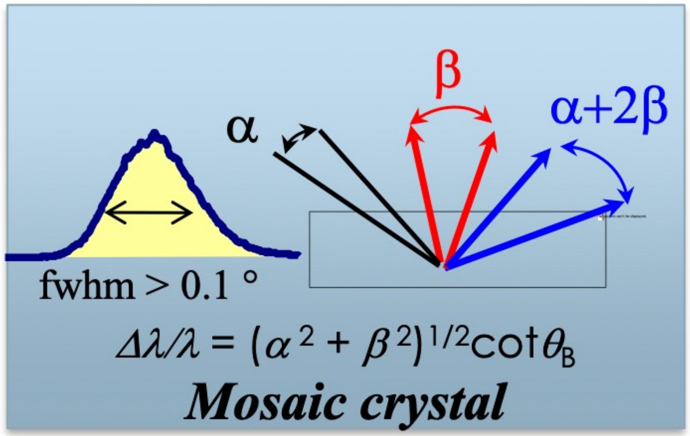




Perfect crystal

- The reflection range of perfect crystals is in the order of 0.001°.
- Perfect crystals do not reflect efficiently neutrons from the source of divergence α .
- Very high resolution but no intensity

Mosaic Crystal

- The reflection range of mosaic crystals is of the order of 0.3 ° even more....
- Mosaic crystals can reflect efficiently neutrons from the source of divergence α, if $\beta \sim \alpha$
- High intensity and reasonable resolution

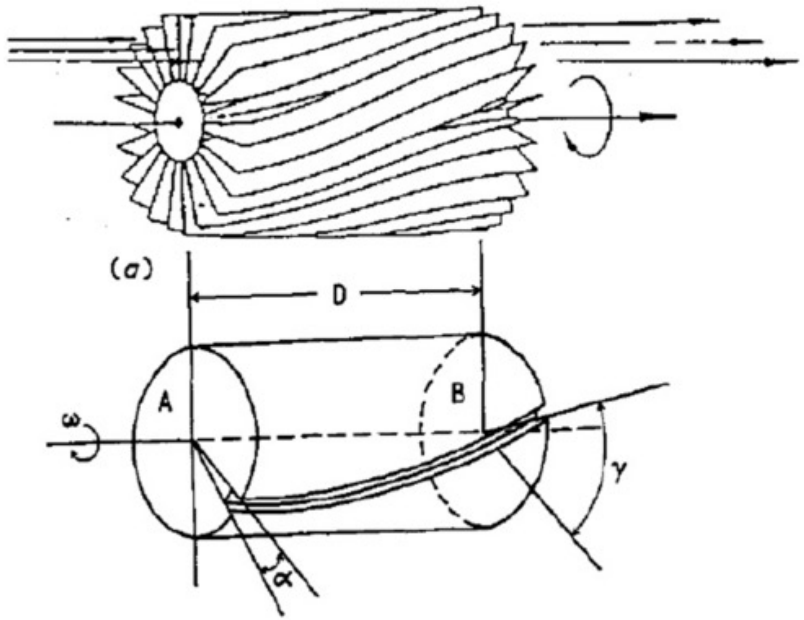
Neutron monochromator

Crystal	Orientation	Crystal mosaic	Neutron energy	Application
HOPG (C) d_{002} = 3.35Å	002	0.5°- 3°	Cold Thermal	High flux
Cu d_{111} = 2.08Å	111, 220, 200	0.01° - 3°	Hot Thermal	High flux or high resolution
Si d_{111} = 3.13Å	111, 113	Perfect	Cold Thermal	High resolution
Fe d_{111} = 3.26Å	111, 113	< 0.4°	Cold Thermal	High resolution
Heusler Cu_2MnAl	111	0.2 – 0.6 °	Hot Thermal	Polarized neutrons

HOPG = Highly Oriented Pyrolitic Graphite

Velocity selector

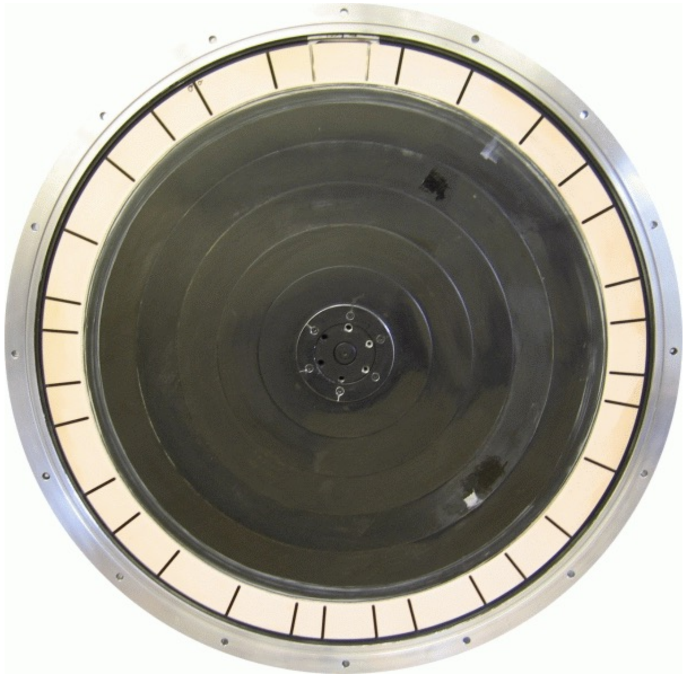
A velocity selector is a mechanical device that allows only neutrons of certain wave- length or velocity to get transmitted through.

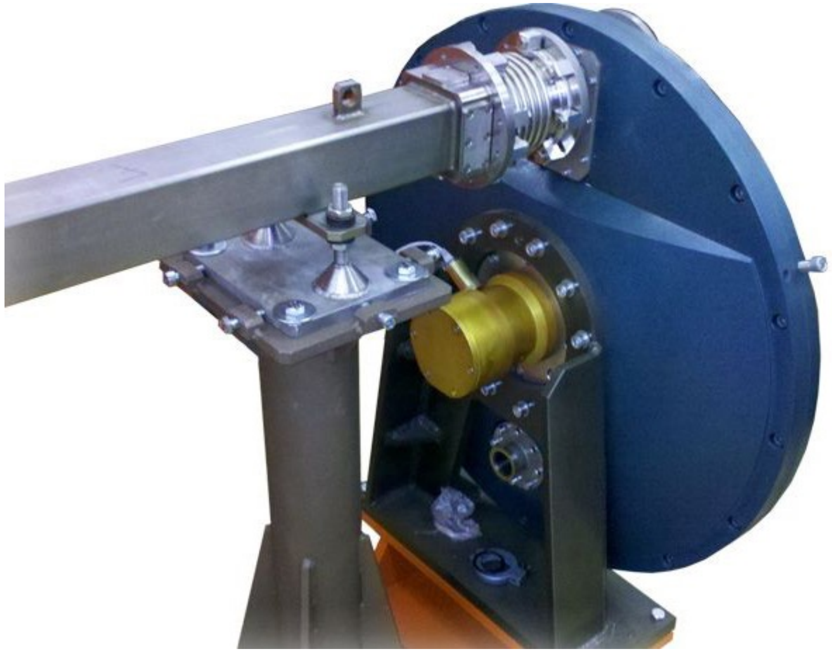


Neutron choppers

Interrupt the neutron beam for well-defined durations

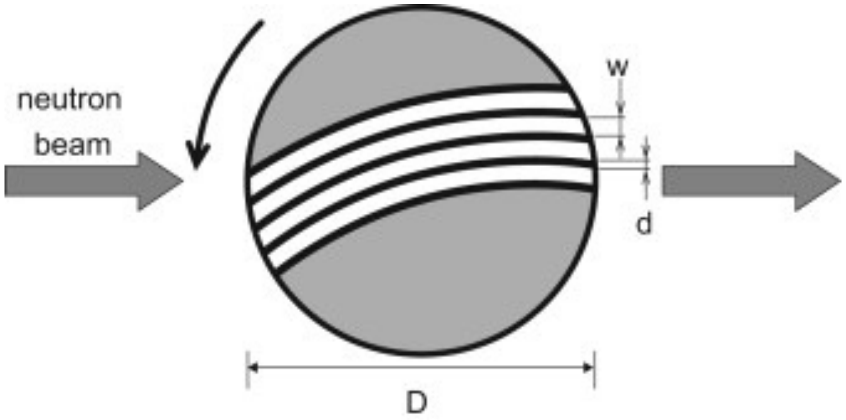
To convert continuous beam into a pulse one or to further shape an already pulsed beam

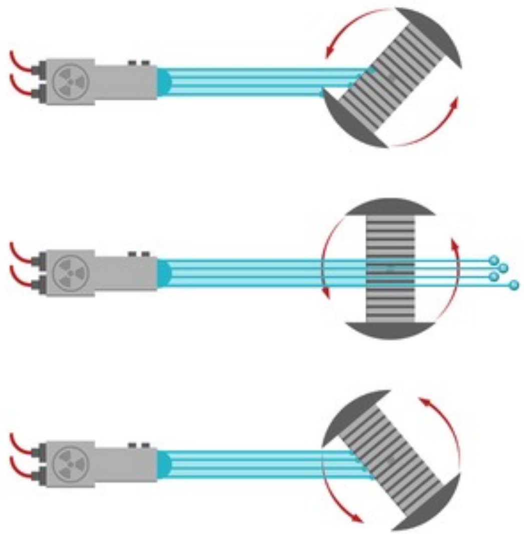




Fermi chopper

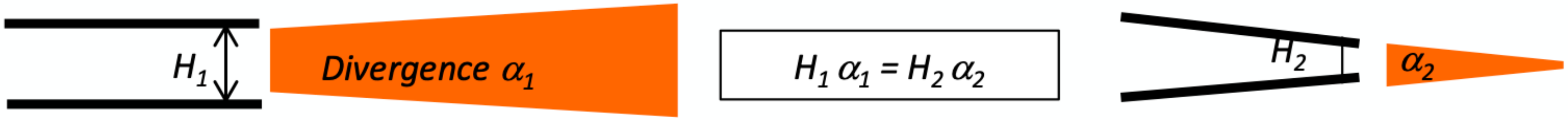
- At a pulsed neutron source, the Fermi chopper is used for monochromatization of neutrons over a wide energy range up to the eV region and consists of a slit package rotating in synchronization with the production timing of pulsed neutrons.
- The Fermi chopper opens when it rotates to a position with the slit direction parallel to the incident neutron beam, and neutrons pass through the slit
- The slit-open time determines the energy resolution of the chopper spectrometer.



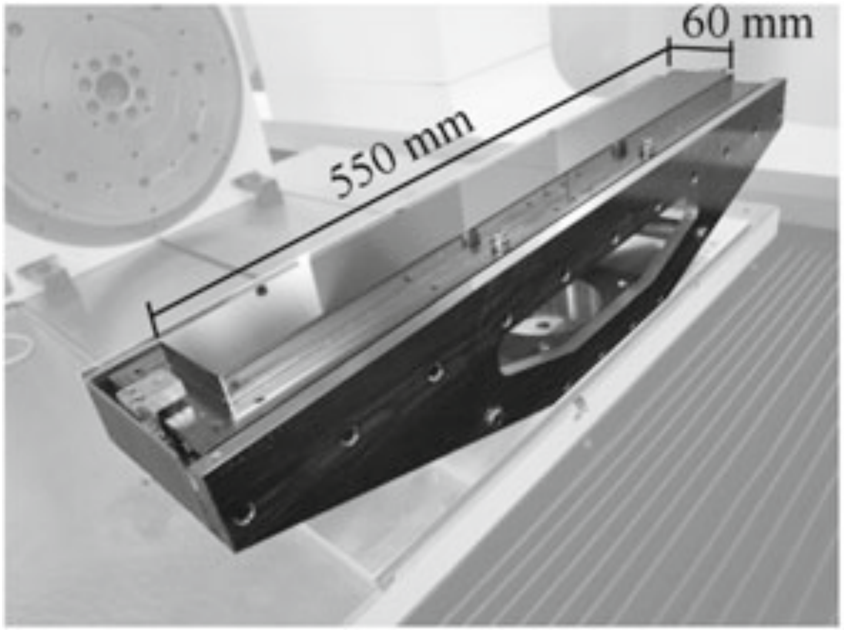


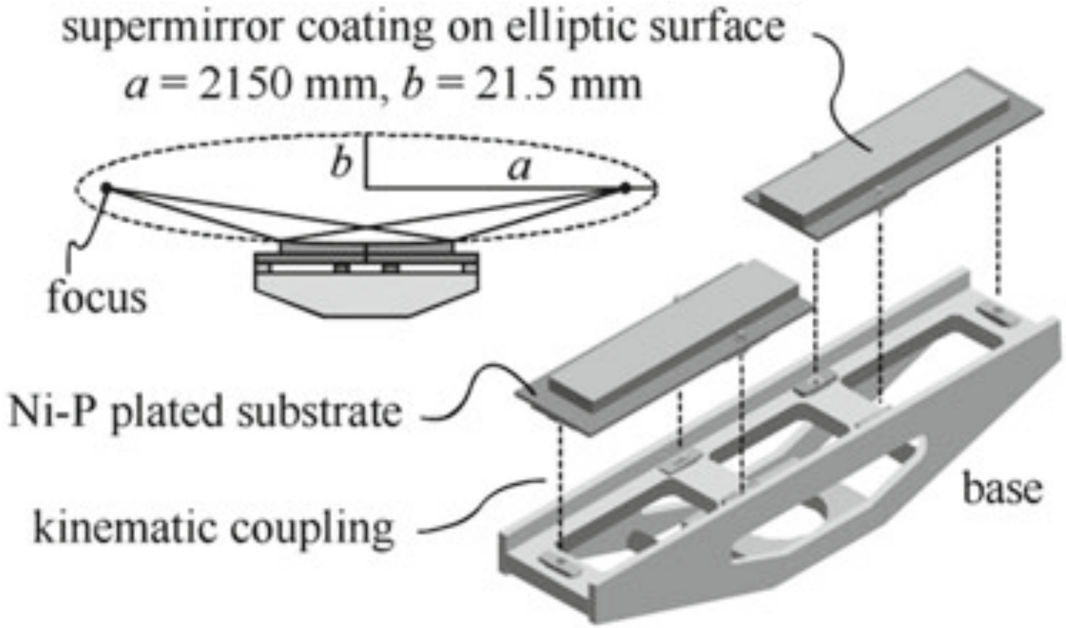
Focussing devices

Focusing devices are used to increase the neutron flux at the sample position. However, the increase of neutron flux implies a degradation of the angular resolution (Liouville's theorem)



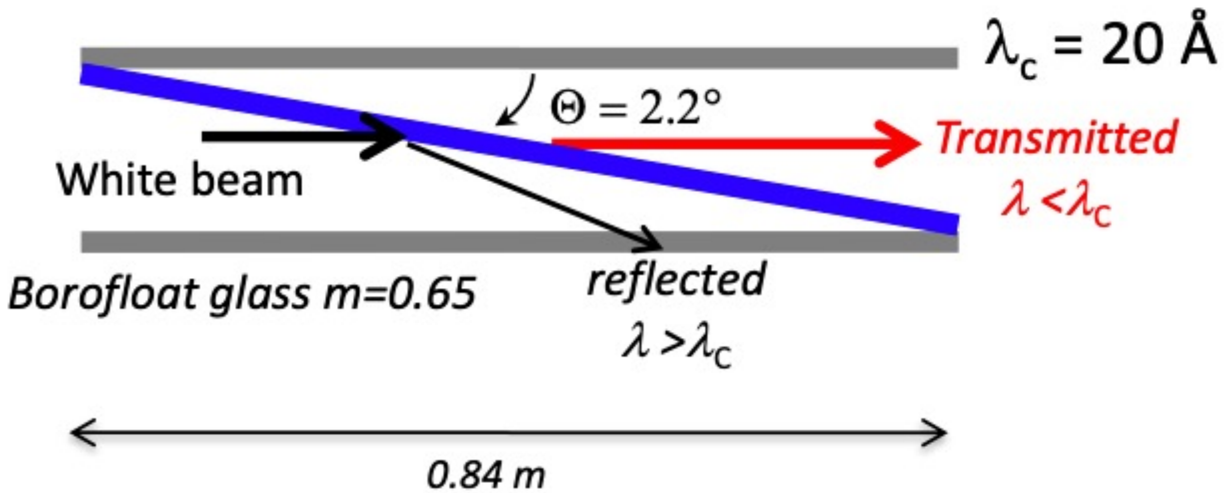
Elliptical focusing mirror





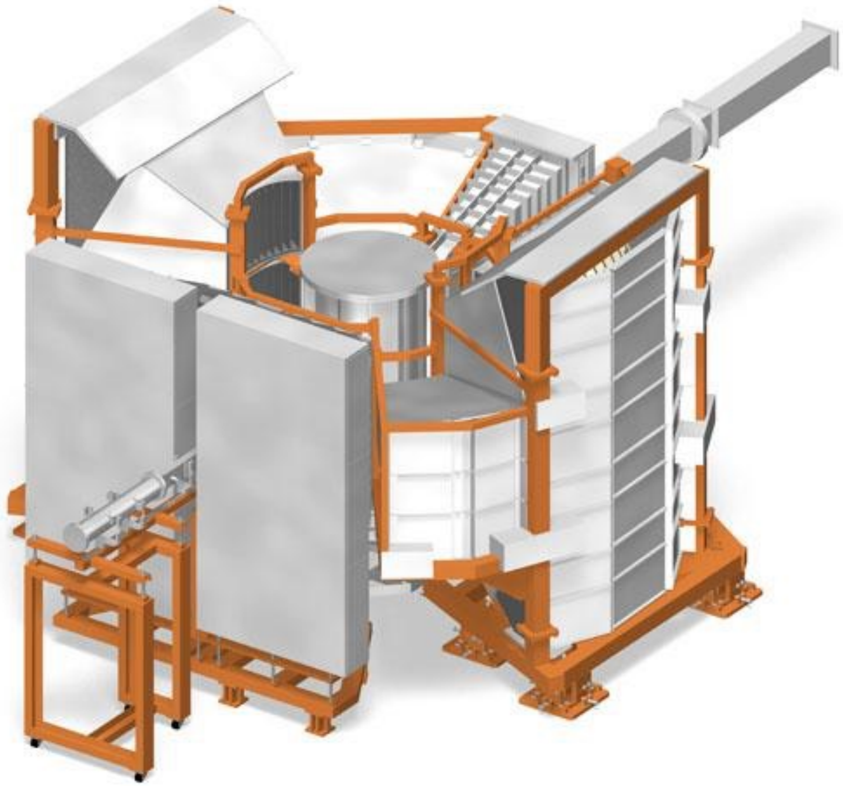
Neutron filters

Reflect out unwanted neutrons with wavelength $\lambda > \lambda_c = \theta / m\, \theta_c^{Ni}$



m=1.1 SM NiV/Ti on 0.5 cm thick Si substrate mounted at an angle θ in a neutron guide

Neutron detectors



Neutron detection

What does it mean to "detect" a neutron?

- Need to produce some sort of measurable quantitative (countable) electrical signal
- Can't directly "detect" slow neutrons

Need to use nuclear reactions to "convert" neutrons into charged particles

Then we can use one of the many types of charged particle detectors

- Gas proportional counters and ionization chambers
- Scintillation detectors
- Semiconductor detectors

Nuclear rections for neutron detectors

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$n + {}^{3}He \rightarrow {}^{3}H + {}^{1}H + 0.764$ MeV

$n + {}^{6}Li \rightarrow {}^{4}He + {}^{3}H + 4.79$ MeV

$n + {}^{10}B \rightarrow {}^{7}Li^{*} + {}^{4}He \rightarrow {}^{7}Li + {}^{4}He + 0.48$ MeV γ +2.3 MeV (93%)
$\rightarrow {}^{7}Li + {}^{4}He$ +2.8 MeV (7%)

$n + {}^{155}Gd \rightarrow Gd^{*} \rightarrow \gamma$-ray spectrum

$n + {}^{157}Gd \rightarrow Gd^{*} \rightarrow \gamma$-ray spectrum

$n + {}^{235}U \rightarrow$ fission fragments + ~160 MeV

$n + {}^{239}Pu \rightarrow$ fission fragments + ~160 MeV

^{3}He detectors

Natural abundance: 0.000137%

By-product of radioactive decay of tritium (nuclear weapons program)

$$^3H \rightarrow {}^3He + e + \nu_e \qquad T_{1/2} = 12.3\ y$$

U.S. mass production of tritium ceased in the mid 1990's (weapons reduction)

U.S. demand increased after Sept 11, 2001 (portal monitors, border security)

In 2008 a critical shortage of helium-3 in the U.S. was realized (supply << demand)

BF_3 commonly used as neutron gas detector prior to the widespread availability of He-3

- but BF_3 is a toxic, corrosive gas (He-3 is inert)
- nevertheless, some facilities have recently revisited BF_3 due to the He-3 shortage

^{3}He detectors

The reaction: n + ^{3}He $\rightarrow$ ^{3}H + ^{1}H + 0.764 MeV

Momentum Conservation – trajectories antiparallel
Energy Conservation – KE split inversely proportional to mass (3:1)

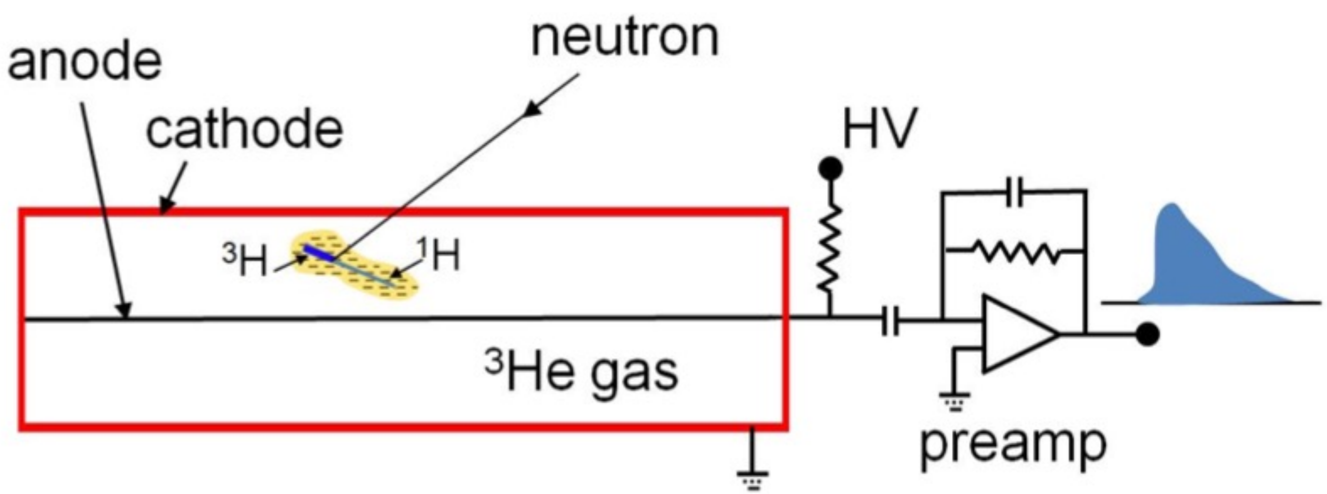
- proton kinetic energy = 573 keV
- triton kinetic energy = 191 keV

Reaction products lose energy through interactions with gas atoms (excitation and ionization)

Stopping gases are added to reduce size of charge cloud: Ar, C_3H_8, CF_4

^{3}He detectors

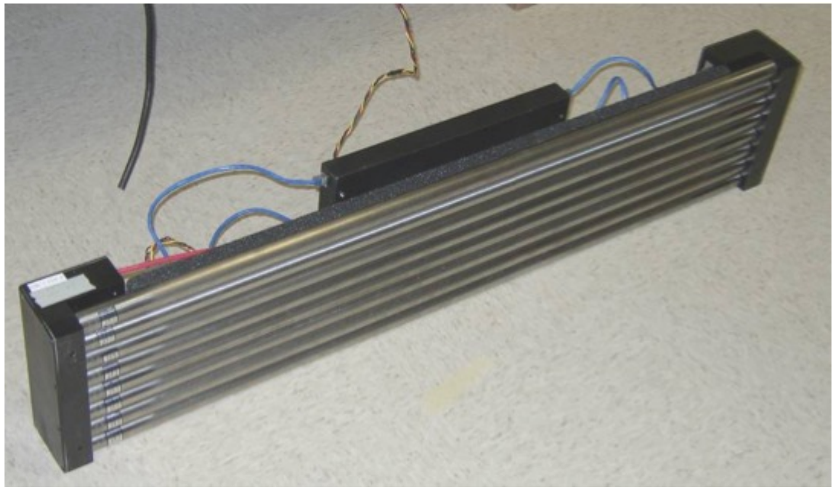
- Positive HV on wire,E field falls off as 1/r (strong near wire)
- Electrons drift in toward wire (anode), ions drift out toward grounded tube wall (cathode), but at velocities ~ 1000 times slower
- Current pulse is measured, proportional to charge collected on wire (Shockley-Ramo theorem).
- If voltage is high enough, electron collisions ionize gas atoms producing even more electrons

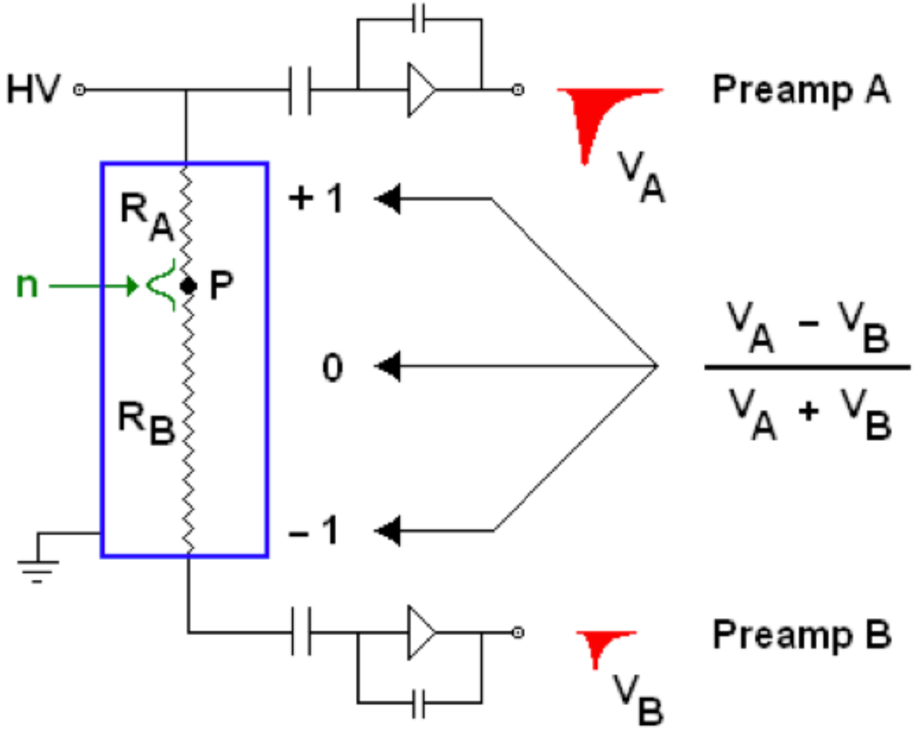


Linear position sensitive ^{3}He detector

- Modular Assembly "8 packs"
- Preamplifier at each end of tube
- Current divides according to resistance it sees to preamps
- Current from both preamps is measured, location is determined by:

  (A – B)/(A + B)

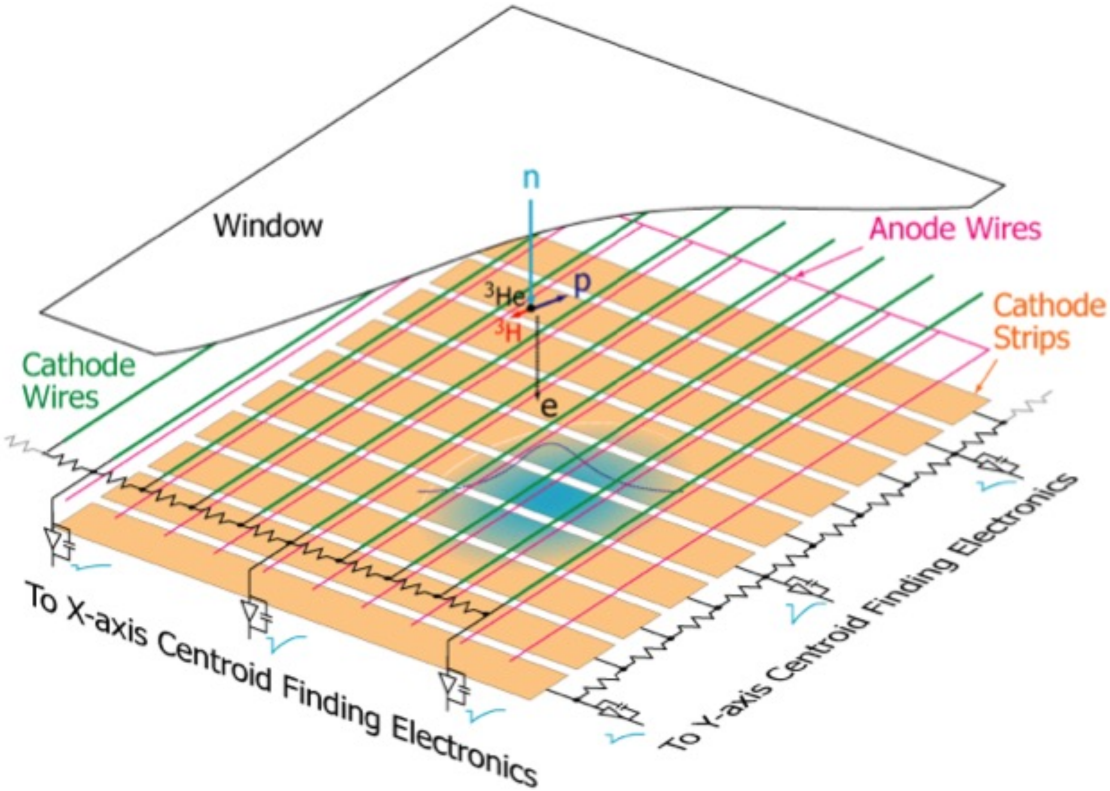




Multi-wire ^{3}He area detectors

- X and Y orthogonal cathodes with anode between at + high voltage
- Charge at anode induces signal on cathodes
- Preamps distributed evenly
- Resistive charge division between each pair of nodes (preamps)
- Interpolation based on signals from three wires:

$$(C - A) / (A + B + C)$$



Scintillator detectors

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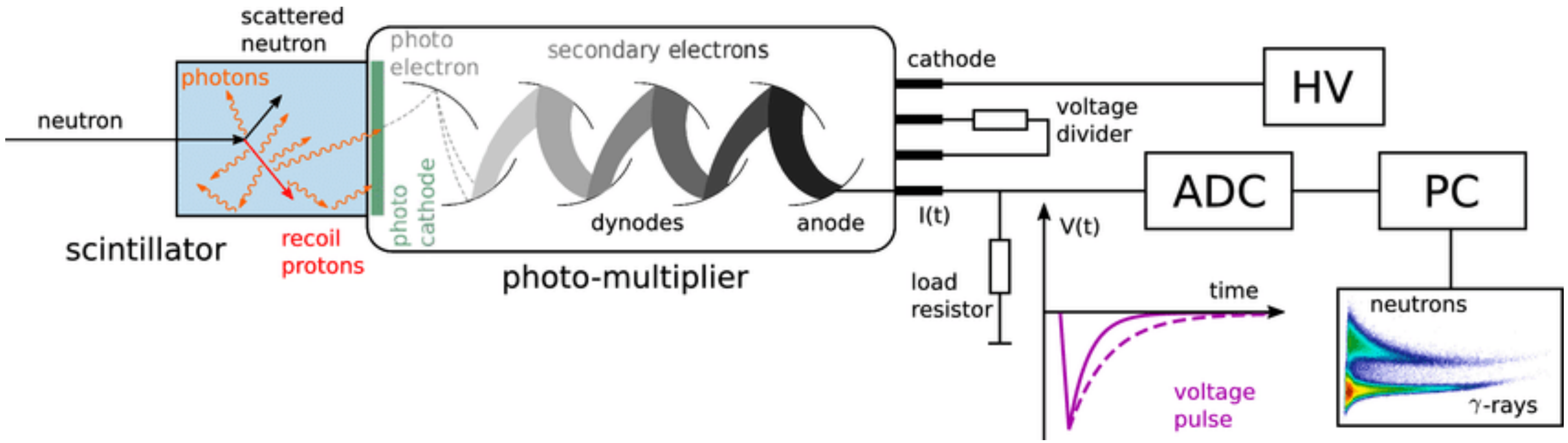
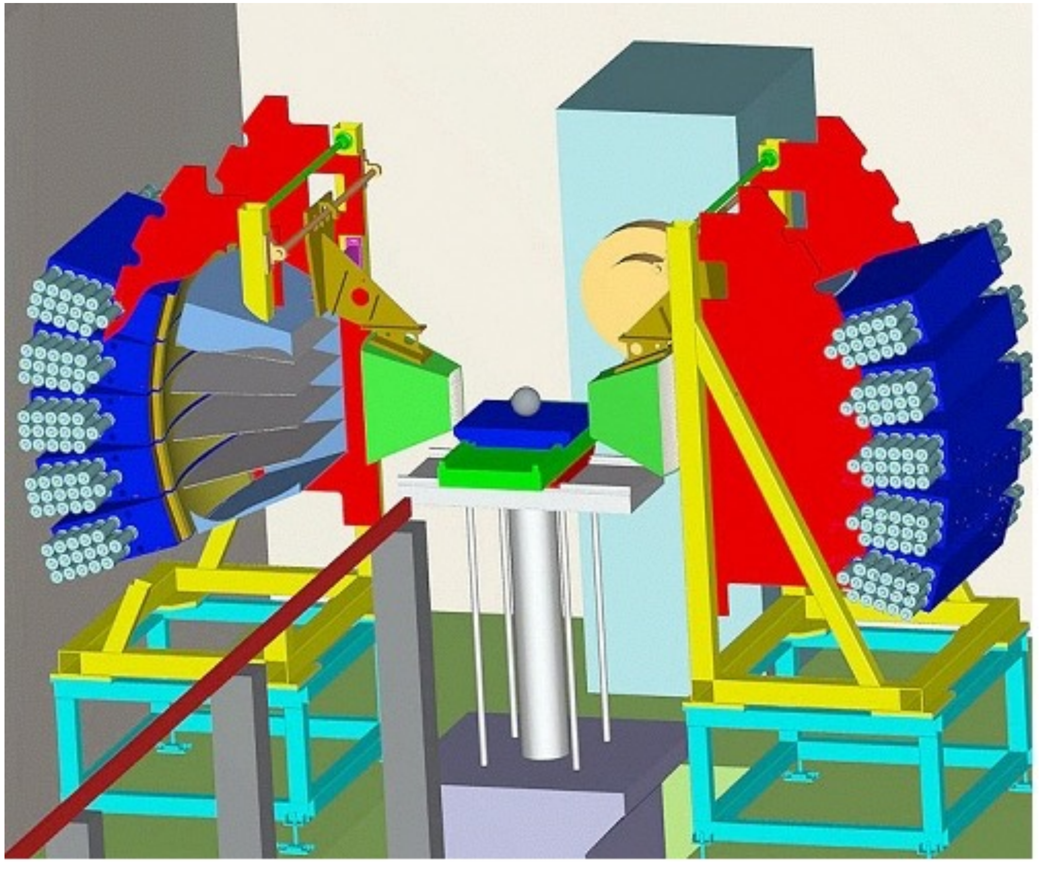
Reaction: n + ^{6}Li → ^{4}He + ^{3}H + 4.79 MeV

Material	Density of ^{6}Li atoms (cm^{-3})	Scintillation efficiency	Photon wavelength (nm)	Photons per neutron
Li glass (Ce)	1.75×10^{22}	0.5%	395	~7000
LiI (Eu)	1.83×10^{22}	2.8%	470	~51'000
ZnS (Ag) – LiF	1.18×10^{22}	9.2%	450	~160'000



Scintillator detectors

Example: Engin-X at ISIS (Rutherford lab, UK)



Other detection systems

- X-ray film in connection with converter foils from Gd, Dy, oder In. The excitation and blackening of the film is caused by gamma and beta radiation as well as by conversion electrons.
- Highly light sensitive CCD camera detectors (cooled in most cases) looking onto the weak light emission from a neutron sensitive scintillator (Li-6 or Gd as neutron absorber).
- Imaging Plates contain Gd as neutron absorber and BaFBr:Eu 2+ as the agent which provides the photoluminescence.
- Flat panels based on amorphous silicon

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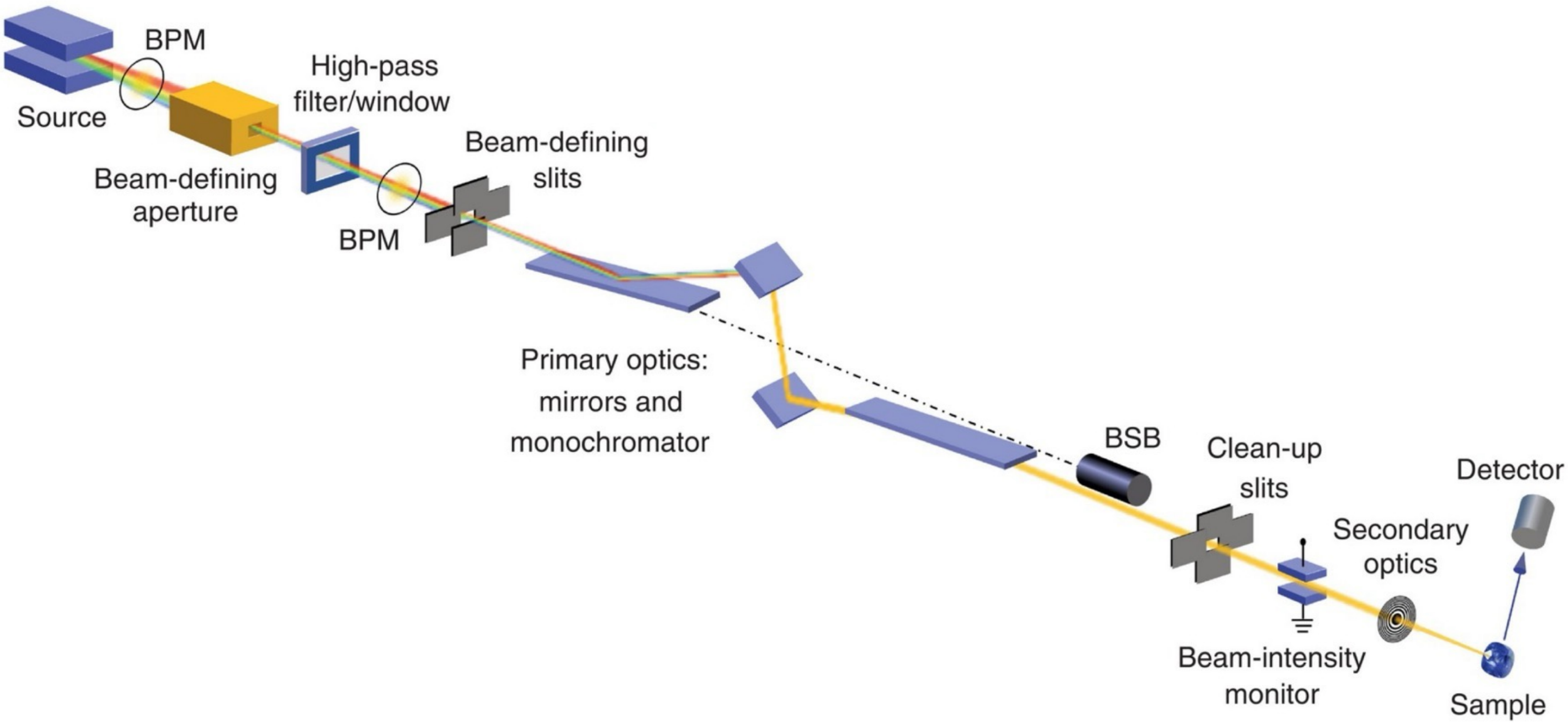
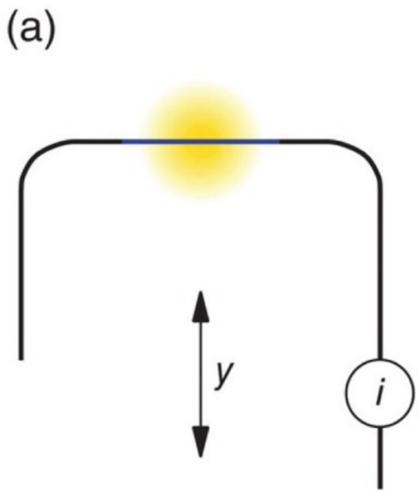
Synchrotron beam lines

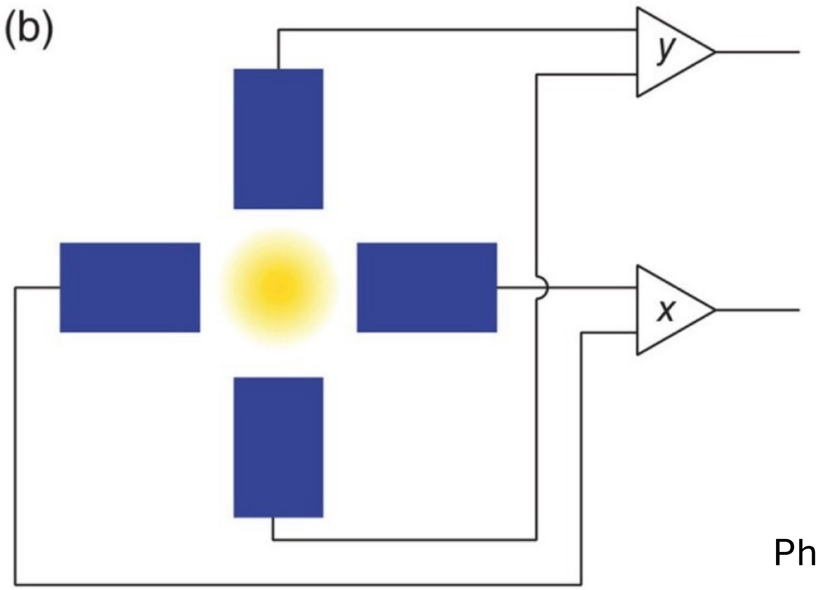


Philip Willmott, An Introduction to Synchrotron Radiation, 2019

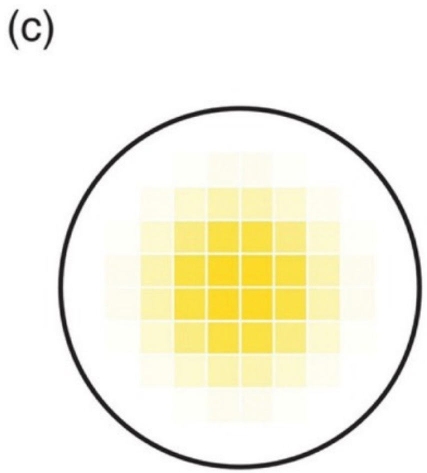
Beam position monitor



Thin wire scans across the beam to record its profile in one direction, by measuring the photocurrent induced by the x-rays



Four metallic blades intersect with the outer halo of the x-ray beam, thereby producing four independent photoelectron currents

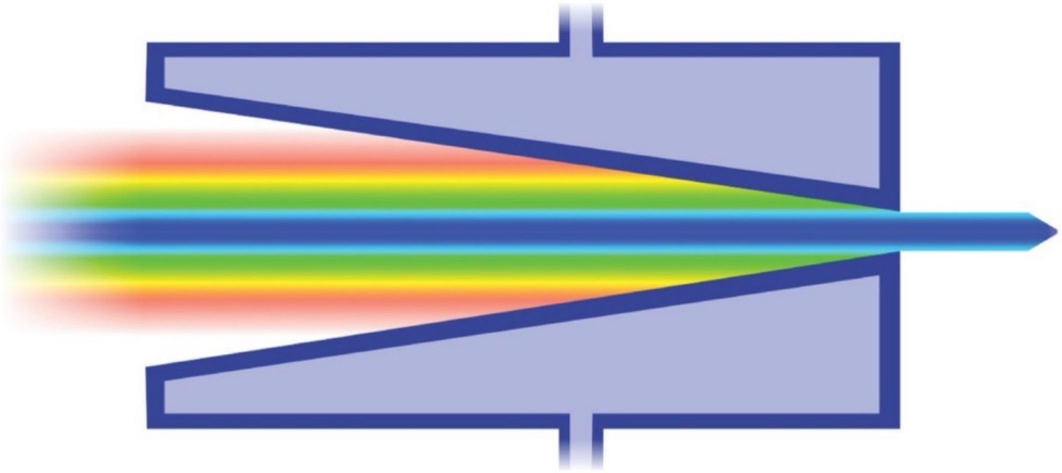


Photocurrents from an array of ultrathin metallic electrodes deposited on thin and transparent synthetic diamond discs

Philip Willmott, An Introduction to Synchrotron Radiation, 2019

Beam-defining primary apertures

The first component downstream of the source, these water– cooled apertures define the angular range to the beamline and often have a rectangular cone shape, in order to distribute the thermal load over a larger area. Because the outer parts of the synchrotron radiation contains lower-energy photons than the central cone, these apertures also act as high-pass filters.



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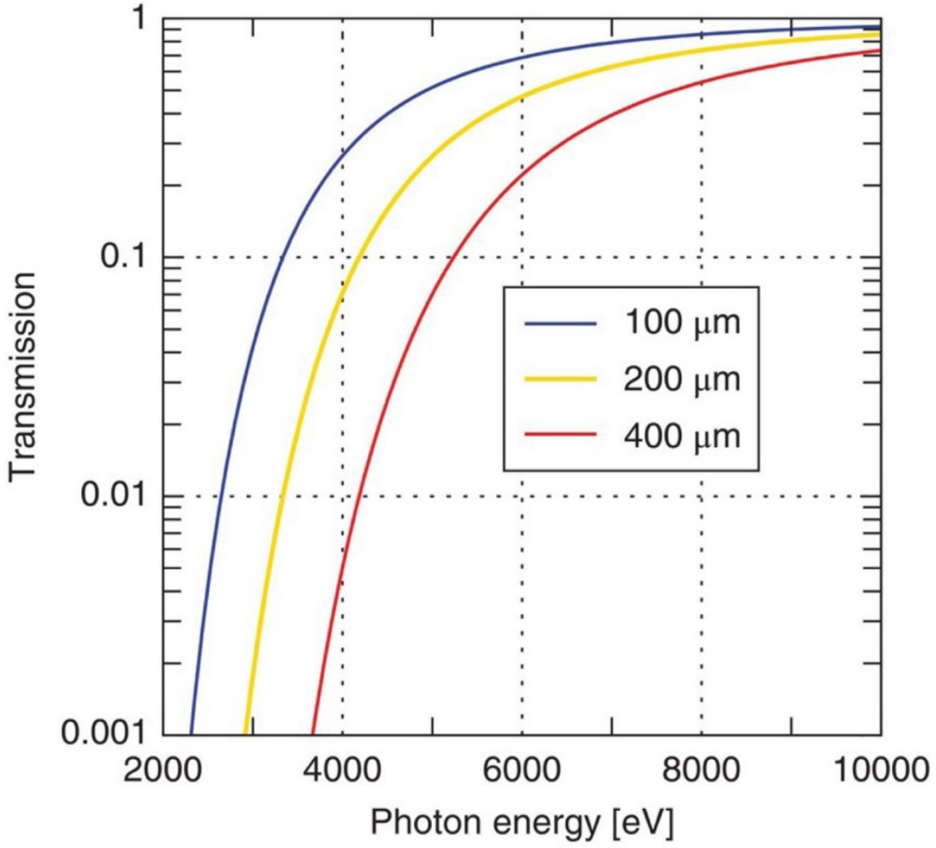
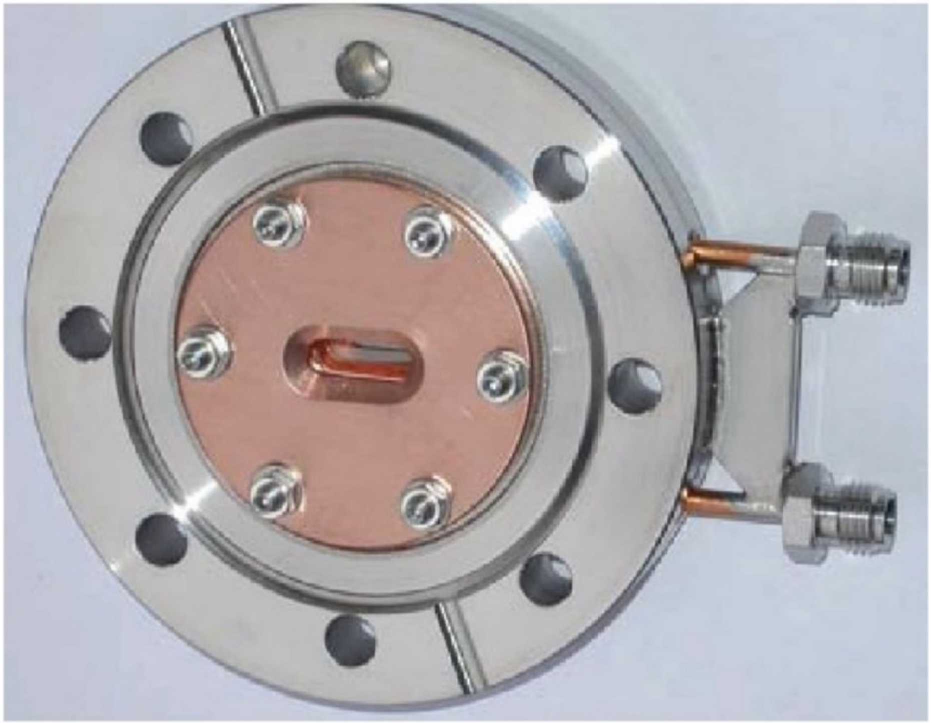
Low-energy filters





High-pass diamond front-end filters used to remove the low-energy component of the x-ray spectrum.

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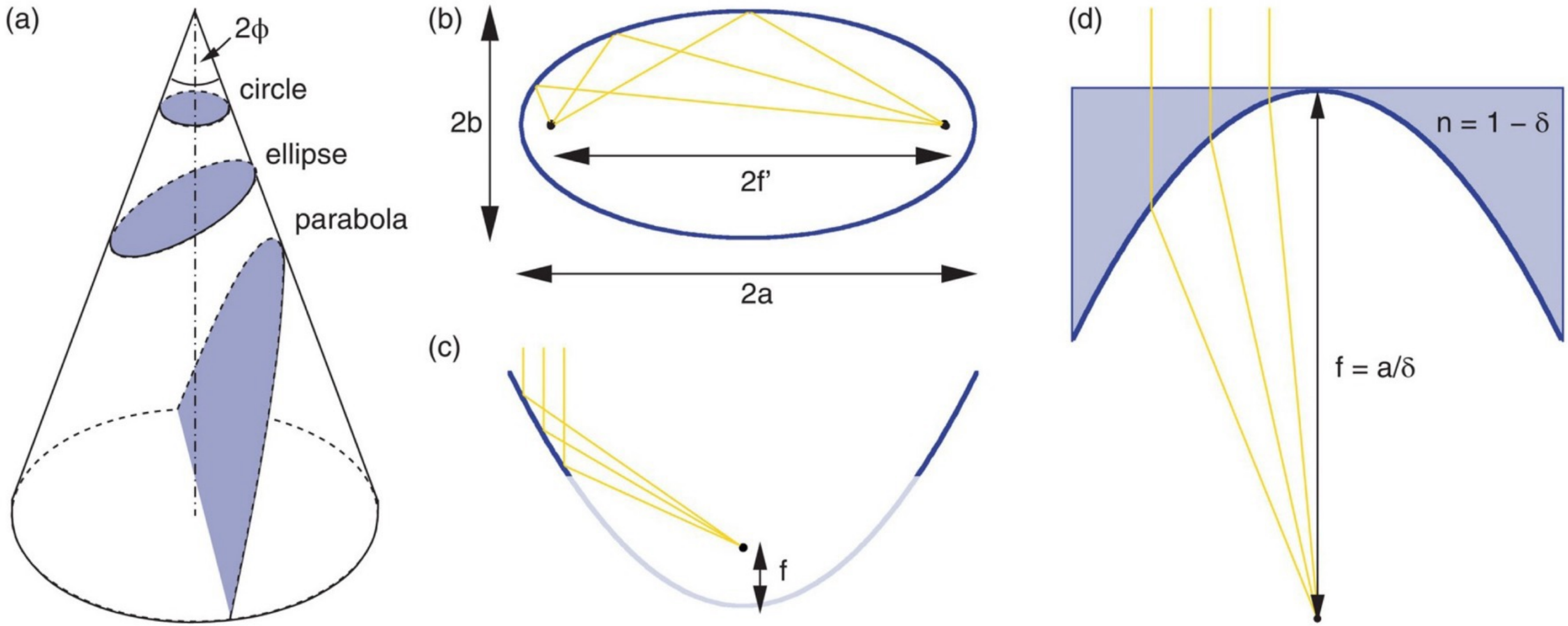
X-ray optics



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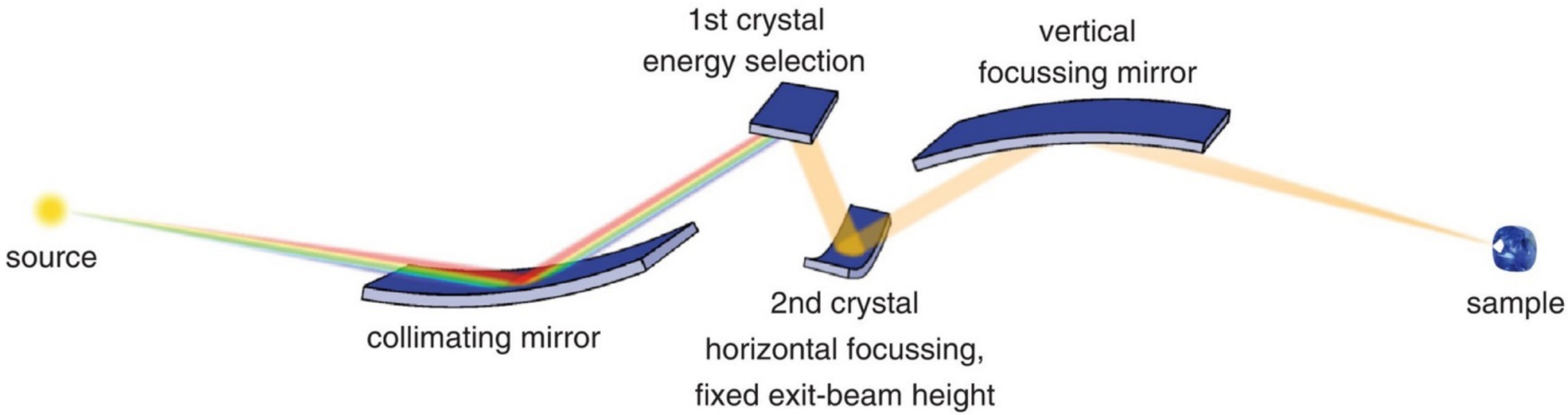
X-ray optics

Schematic of an x-ray optical system, consisting of a double-crystal monochromator and two mirrors. Note that the second crystal of the DCM can also be bent to provide horizontal focussing.



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Monochromators

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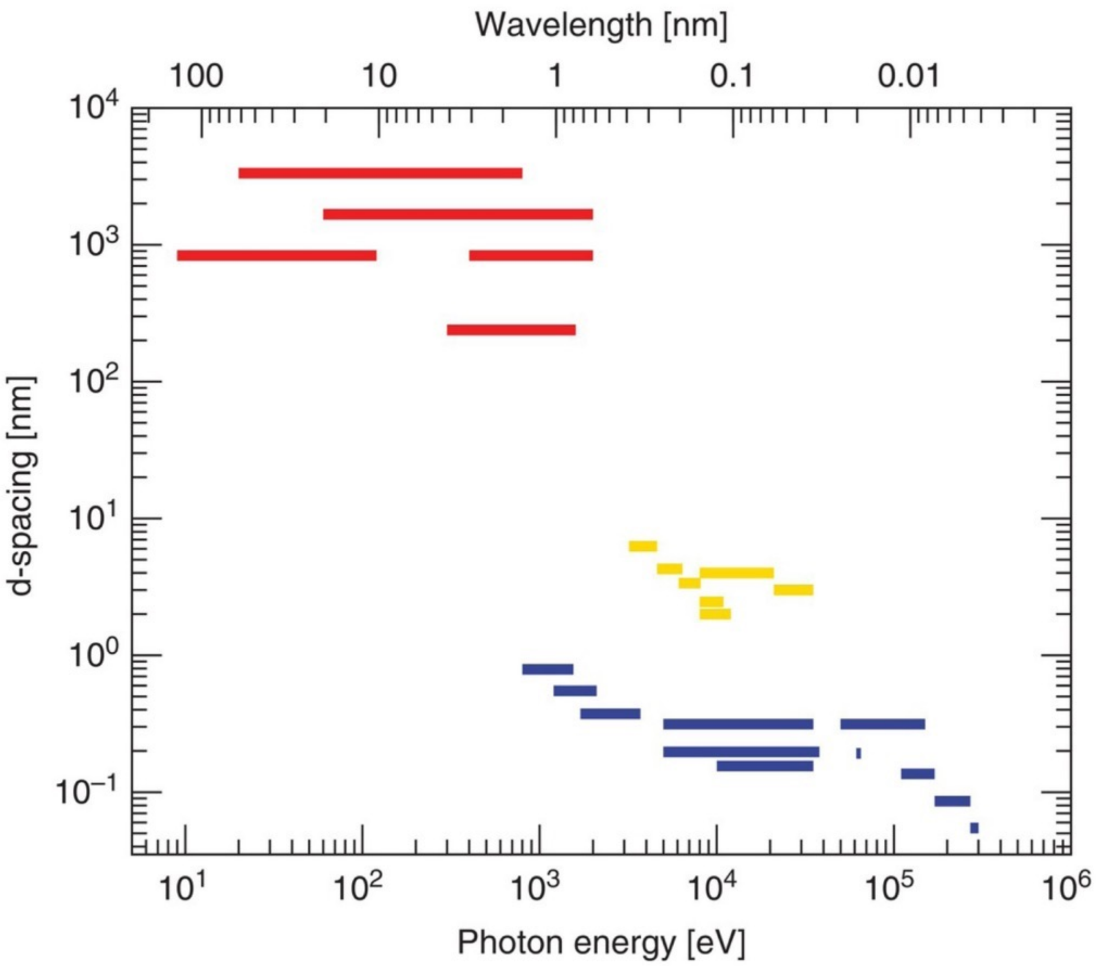
Grating monochromators (red)

Crystal monochromators (blue)

Multilayer monochromators (yellow)



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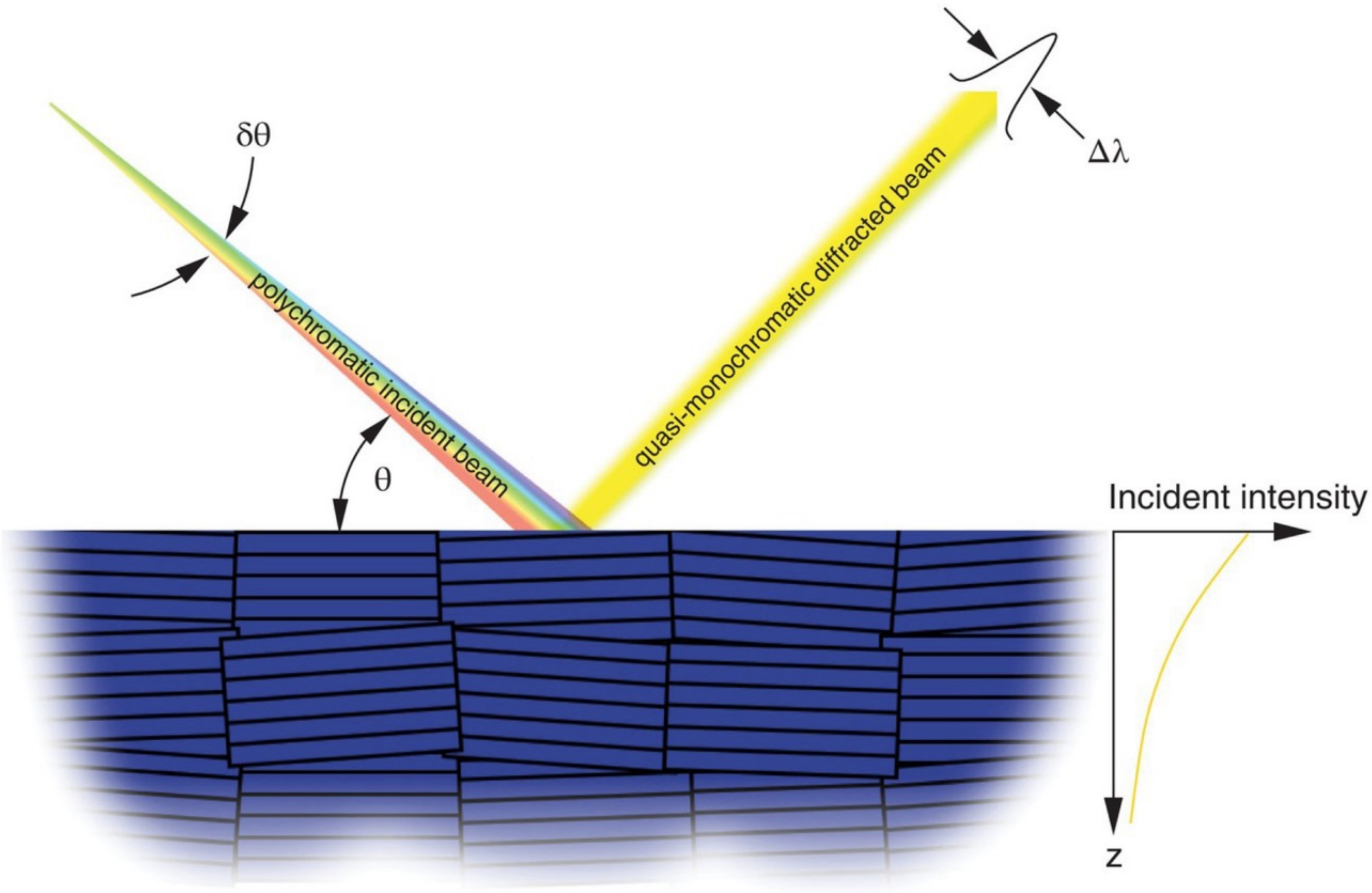
Crystal monochromator



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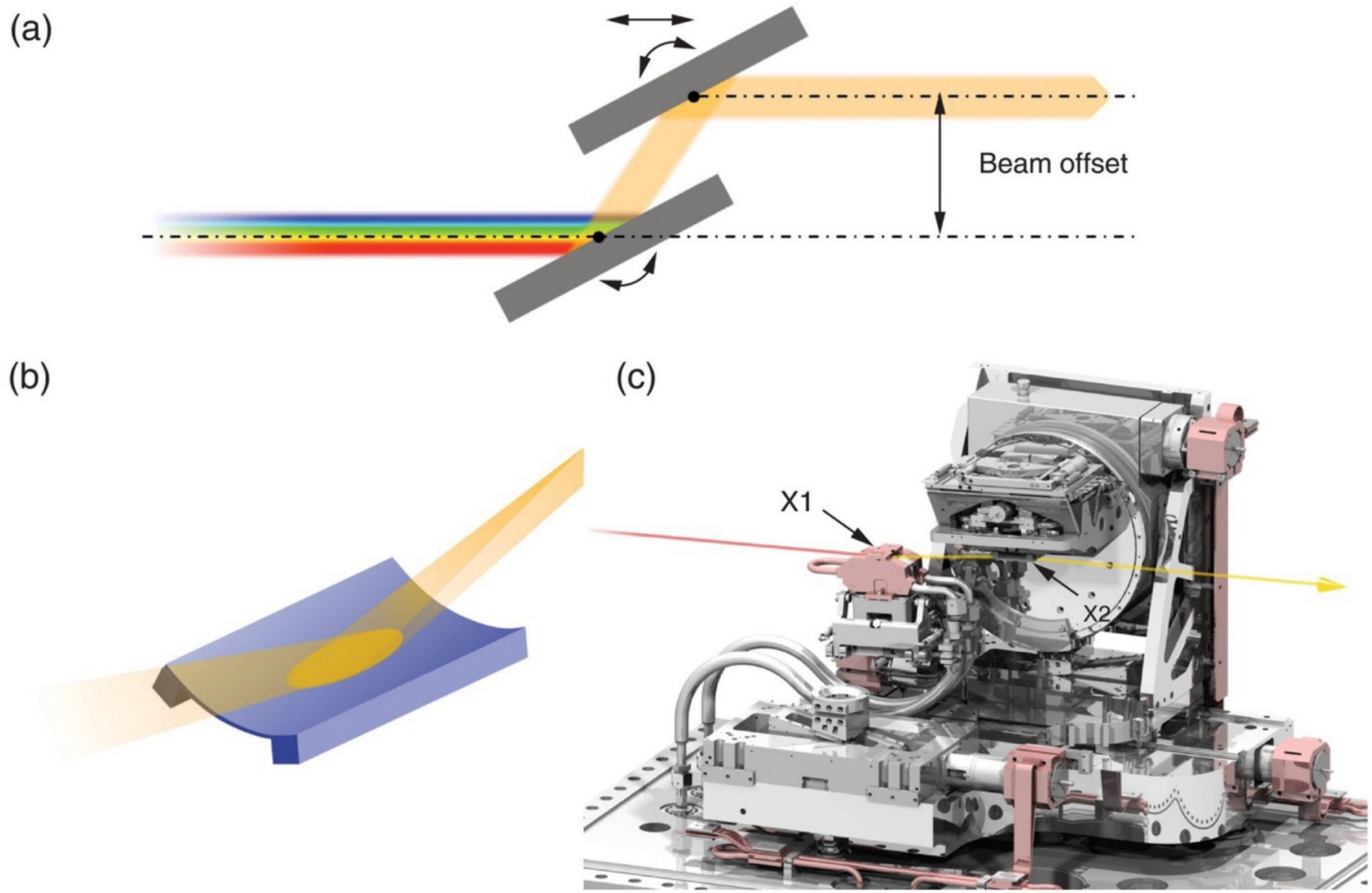
Double crystal monochromator



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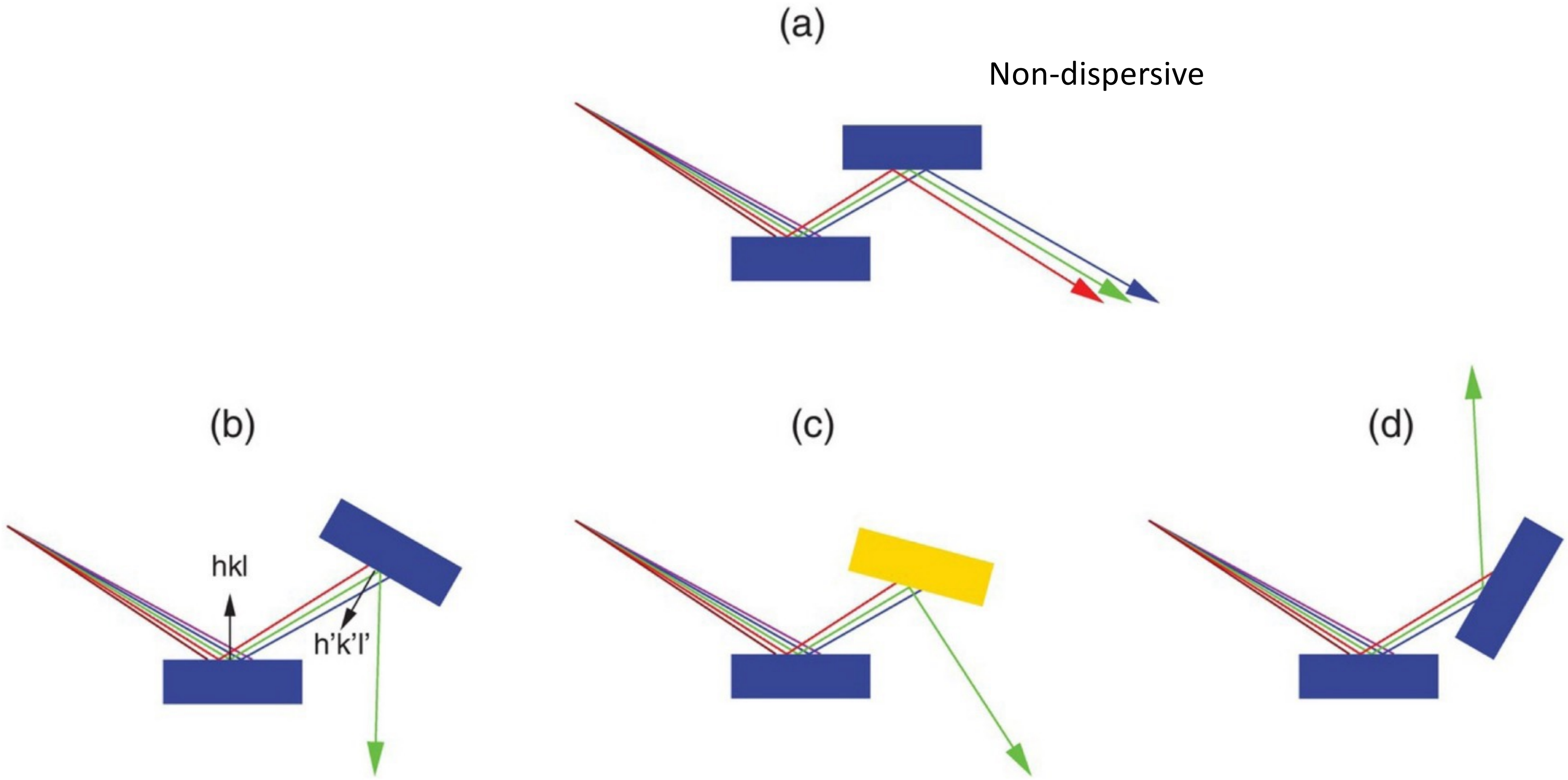
(non)-dispersive



Different crystal orientation

Different crystal type

Same sense of reflection

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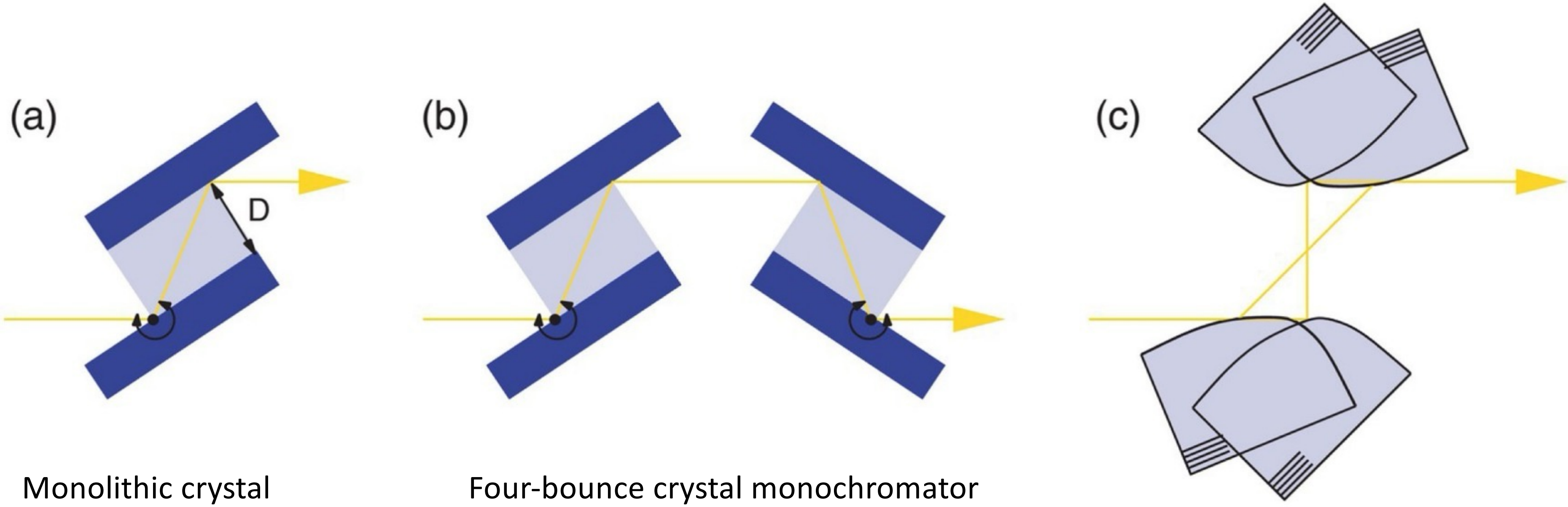
Channel-cut monochromators



Monolithic crystal

Four-bounce crystal monochromator

fixed-exit single channel-cut monochromator

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Double crystal deflectors

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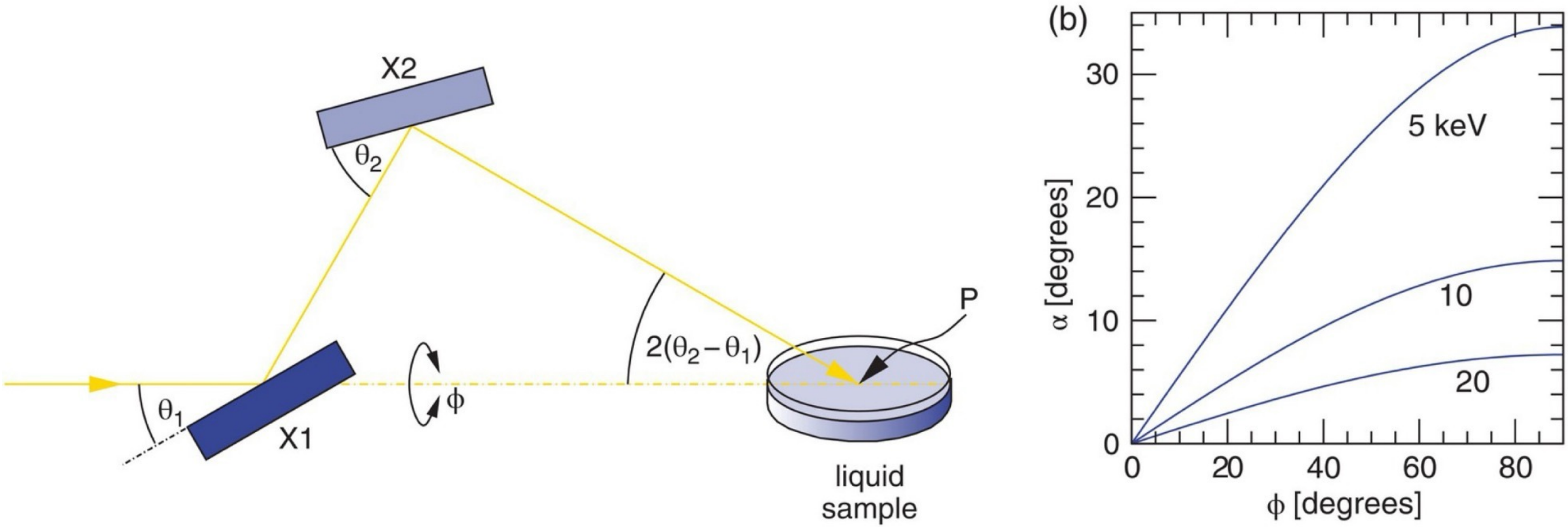

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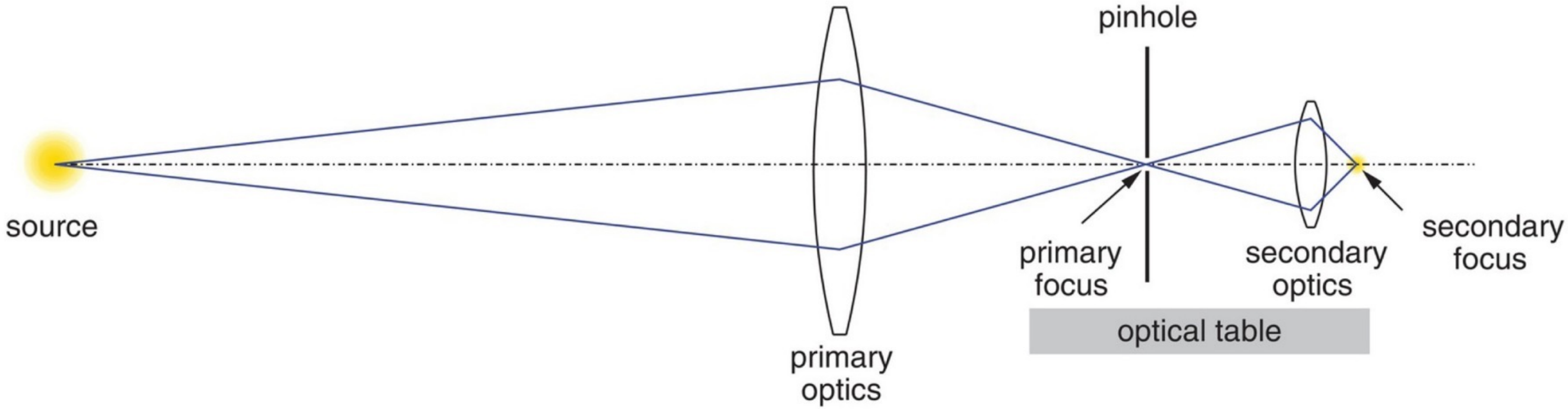
Microfocus and nanofocus optics

- Micro- and nanofocus beams are formed by selecting a fraction of the primary focus using a pinhole, and demagnifying this ‘virtual source’ using secondary optics.
- Compound refractive lenses, tapered glass capillaries, Fresnel zone plates, multilayer Laue lenses, Kirkpatrick–Baez mirrors



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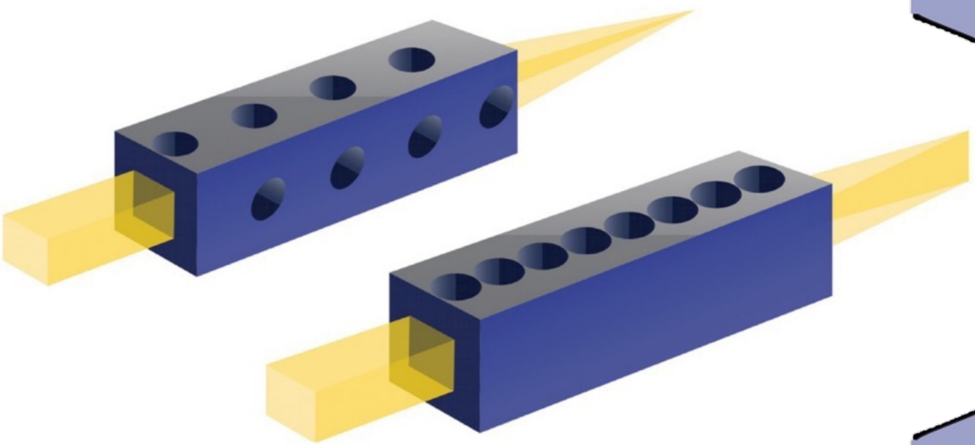
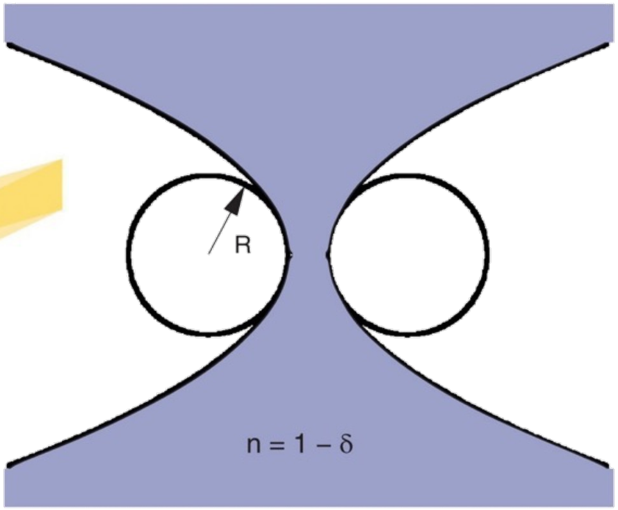
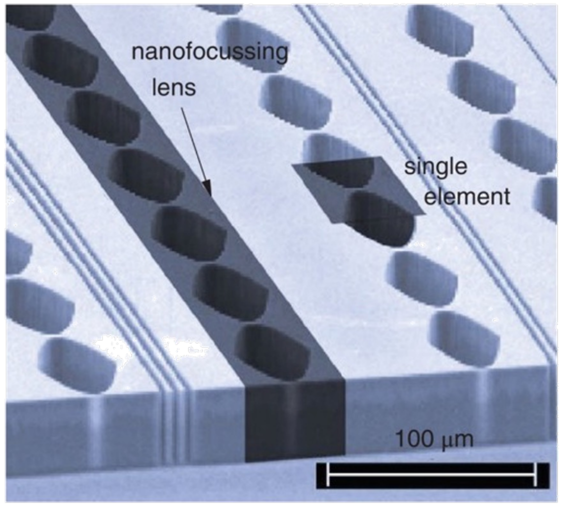
Compound refractive lenses

- Compound refractive lenses. X-ray focussing elements can be constructed from high-transmission material drilled with arrays of holes, which act in much the same way as (very weak) cylindrical convex lenses do for visible light.
- Example: a Be lens consisting of an array of thirty 200 μm-diameter holes has a focal length of approximately 73 cm at 12 keV.

$$f = R/2n_h\delta$$







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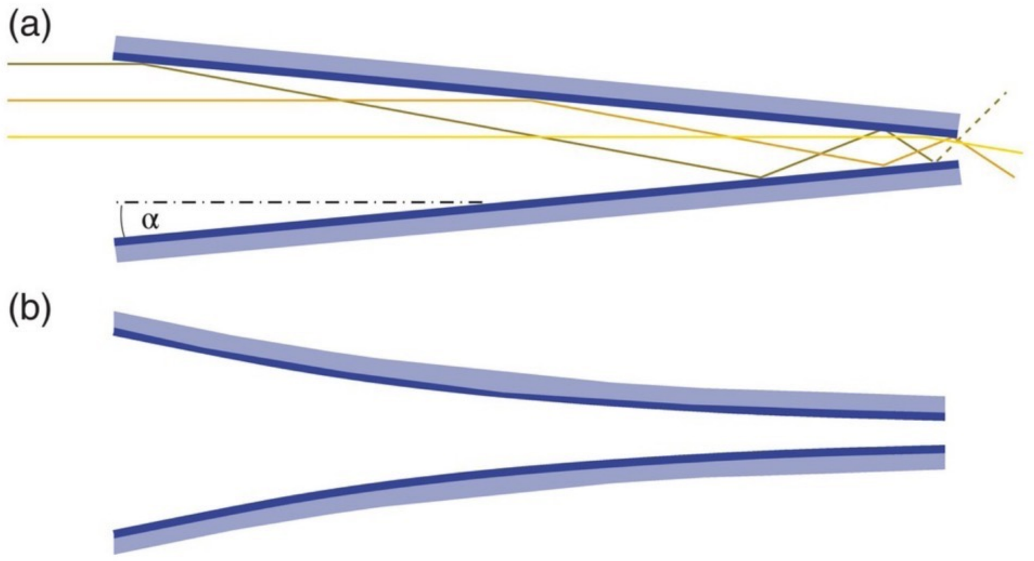
Tapered glass capillaries

- Parallel rays entering the capillary will impinge on the surface at an angle α and be deflected by an angle 2α
- The nth reflection impinges on the surface at an angle $(2n - 1)\alpha < \alpha_c$
- the focal length is independent of the photon energy,
- Needs to be placed very close to the sample



Schematics of (a) a conically tapered glass capillary lens and (b) a curved capillary lens. The inner surface of the capillary can be coated with a high-Z material to increase the critical angle for total external reflection.

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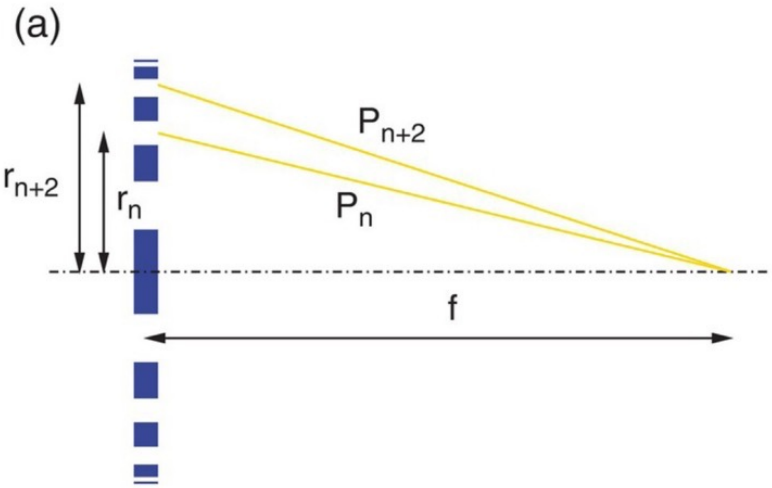
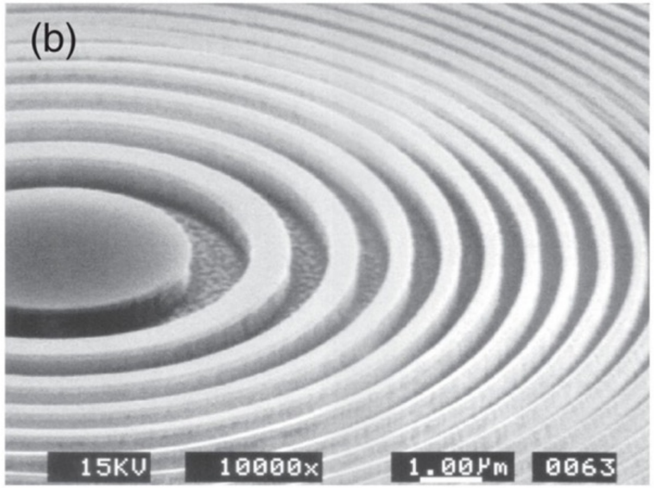
Fresnel Zone Plates

- Fresnel zone plates (FZPs) focus x-rays using diffraction
- electron-beam microlithography allows FZPs to be produced with outer rings well under 100 nm
- hard x-rays in the angstrom range can now be focussed down to just a few tens of nm

$$\sqrt{f^2 + r_{n+2}^2} - \sqrt{f^2 + r_n^2} = m\lambda$$

$$f = \frac{2r_N \Delta r_N}{\lambda} \qquad \Delta r_N = \sqrt{\frac{\lambda f}{4N}}$$





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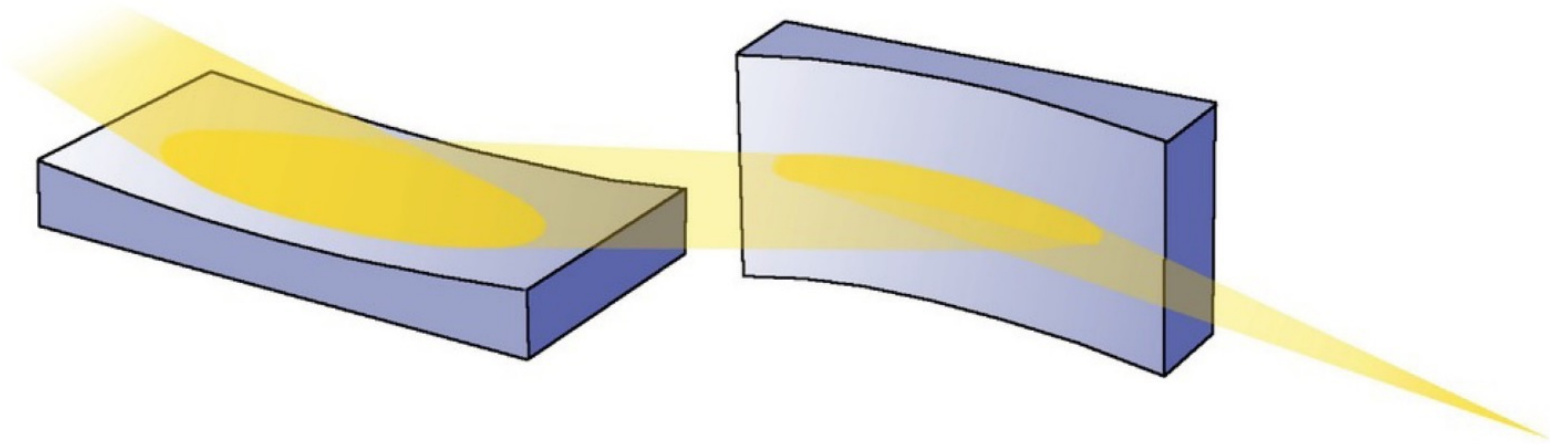
Kirkpatrick–Baez mirrors

- By using two cylindrical mirrors in series, vertical and horizontal focussing can be achieved independently in the so-called 'Kirkpatrick–Baez' (KB) mirror configuration
- The focal lengths are independent of the photon energy
- Time-consuming to optimize spot size



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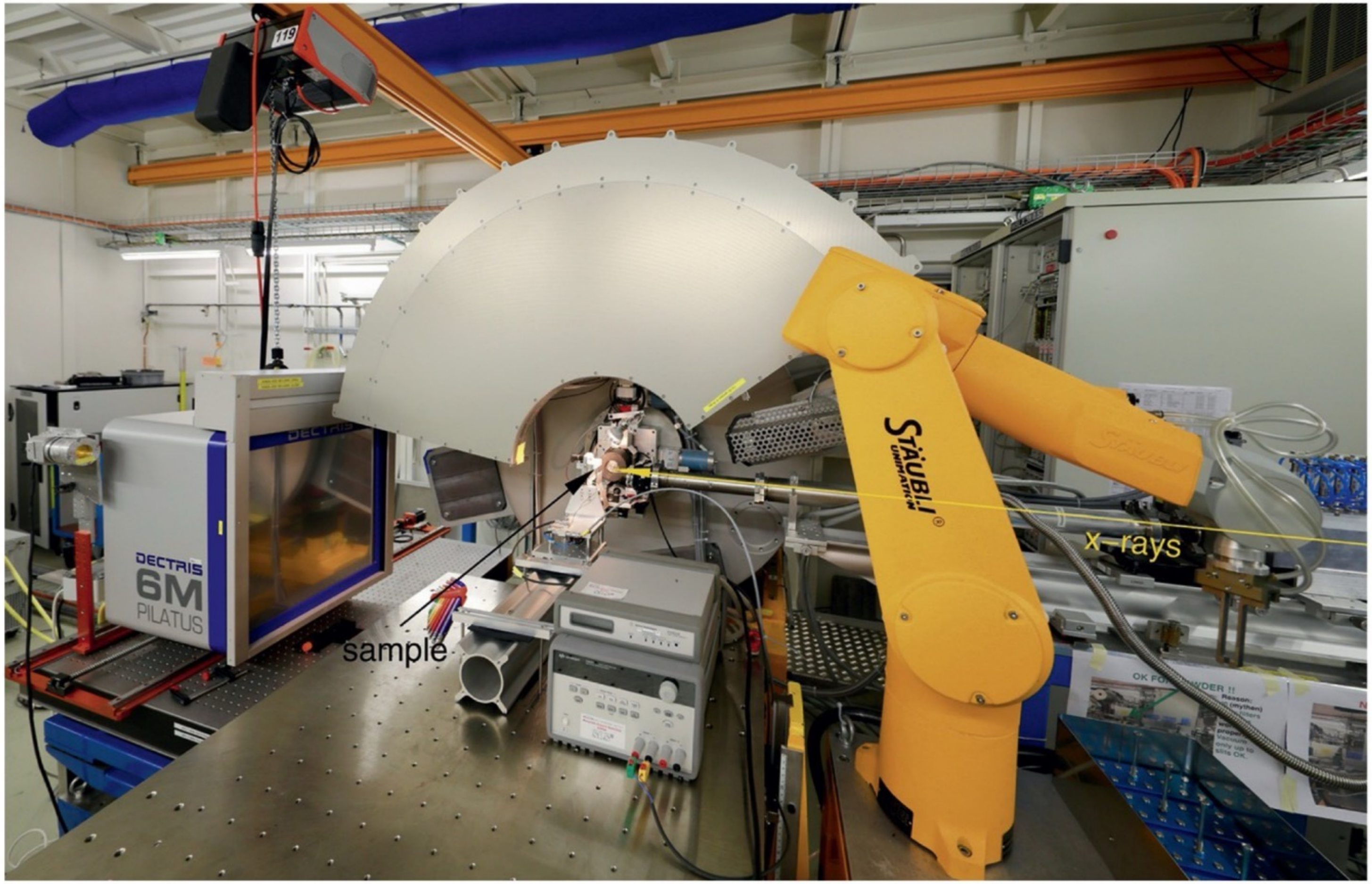
X-ray detectors



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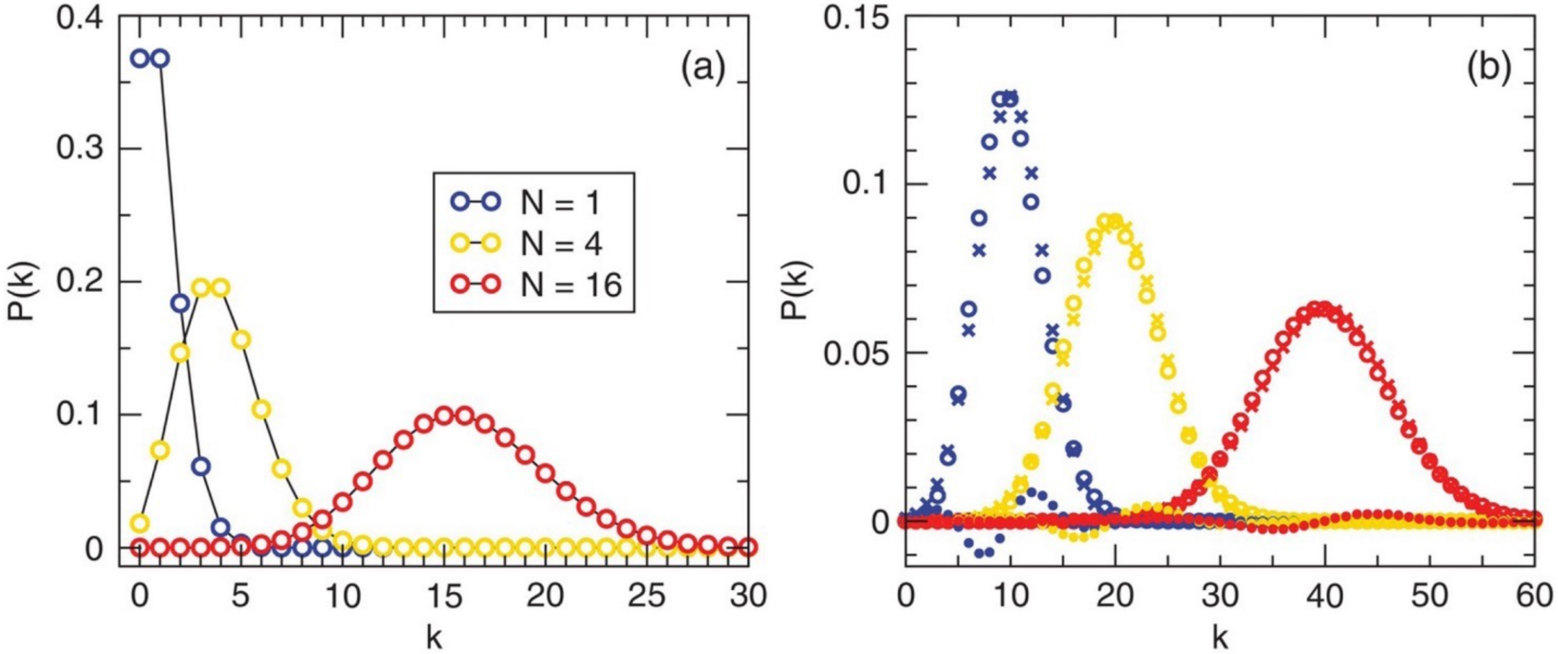
Sources of noise

Any stochastic or random process composed of a series of discrete events, such as the arrival of x-ray photons on to a detector element, is subject to statistical, or ‘Poisson’, noise



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$$P(k) = \frac{e^{-N}\, N^k}{k!}.$$

$$P(k) \approx \frac{e^{-(k-N)^2/2N}}{(2\pi N)^{1/2}}.$$

Scatter ~$N^{1/2}$

To achieve a factor X improvement in the signal-to-noise ratio $\Delta N/N$, one must record X^2 as long

Sources of noise

Dark noise

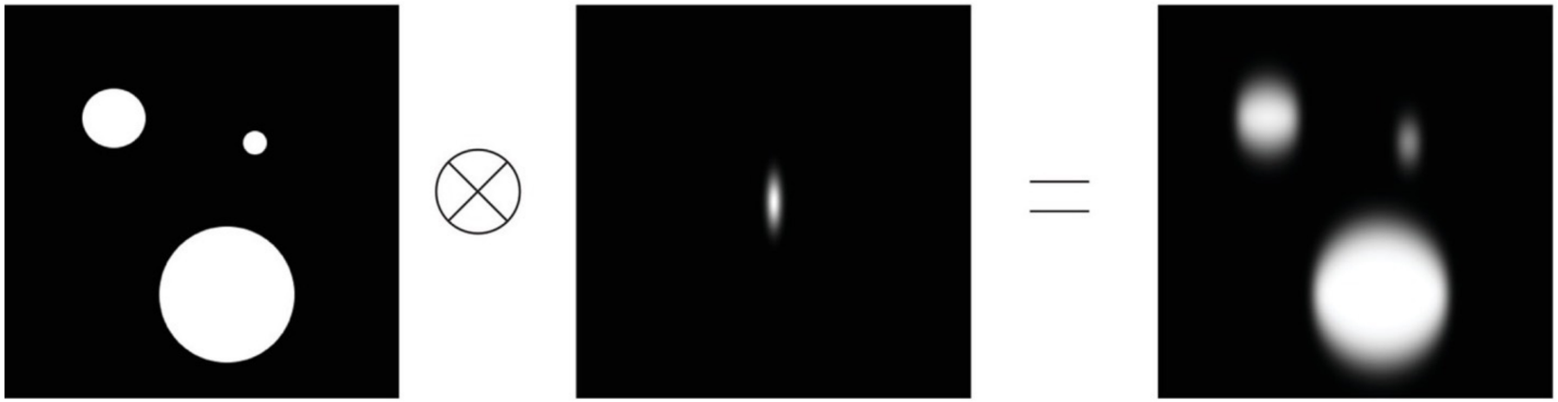
- Noise that is present, even in the absence of any photons
- Varies from pixel to pixel
- Dark images can be recorded to correct of it
- There exist detector with effectively zero dark noise

Readout noise

- Only relevant for weak signal

Point-spread function

- The point-spread function (PSF) describes the response of an imaging system to a point source
- A sharp signal can be smeared out due to various processes (finite pixel size, blooming, …). This less sharp image is the convolution between the original object and the point-spread function



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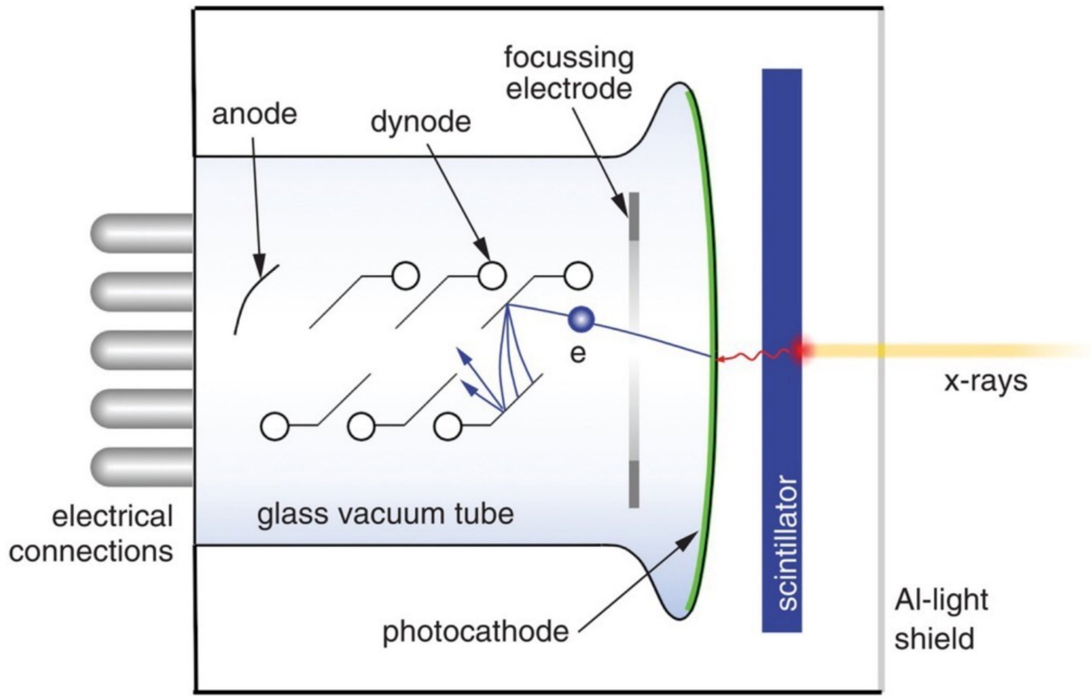
Scintillator detectors

- Typical inorganic scintillator materials are salts or metal oxides doped with high-*Z* materials
- Photons produced by x-rays absorbed in the scintillator material strike photocathode material
- Electrons produced by the photoelectric effect are directed by a focussing electrode towards an array of electrodes.
- On striking a dynode, each electron produces several secondary electrons, which, in turn, are accelerated toward the next dynode, resulting in a cascade production of electrons.
- Accumulation of charge at the anode results in a sharp current pulse corresponding to the arrival of the original x-ray photon at the scintillator



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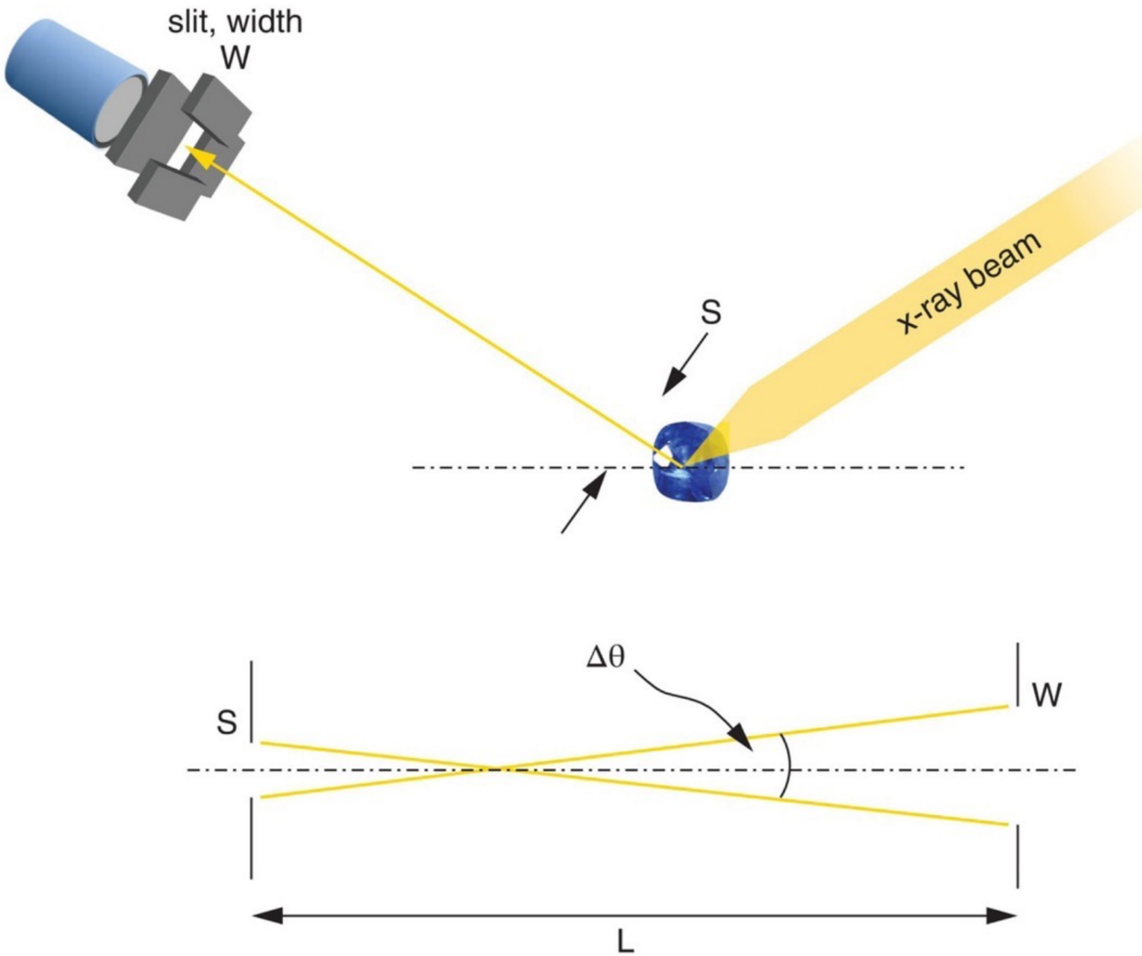
Crystal analysers

- The angular resolution of a point detector is defined by the angles subtended by the irradiated sample volume and by the slit width at the detector.

$$\Delta\theta = \frac{S + W}{L}$$

- Many detectors, including scintillators, cannot distinguish x-rays with different energies.
- Solution: crystal analysers



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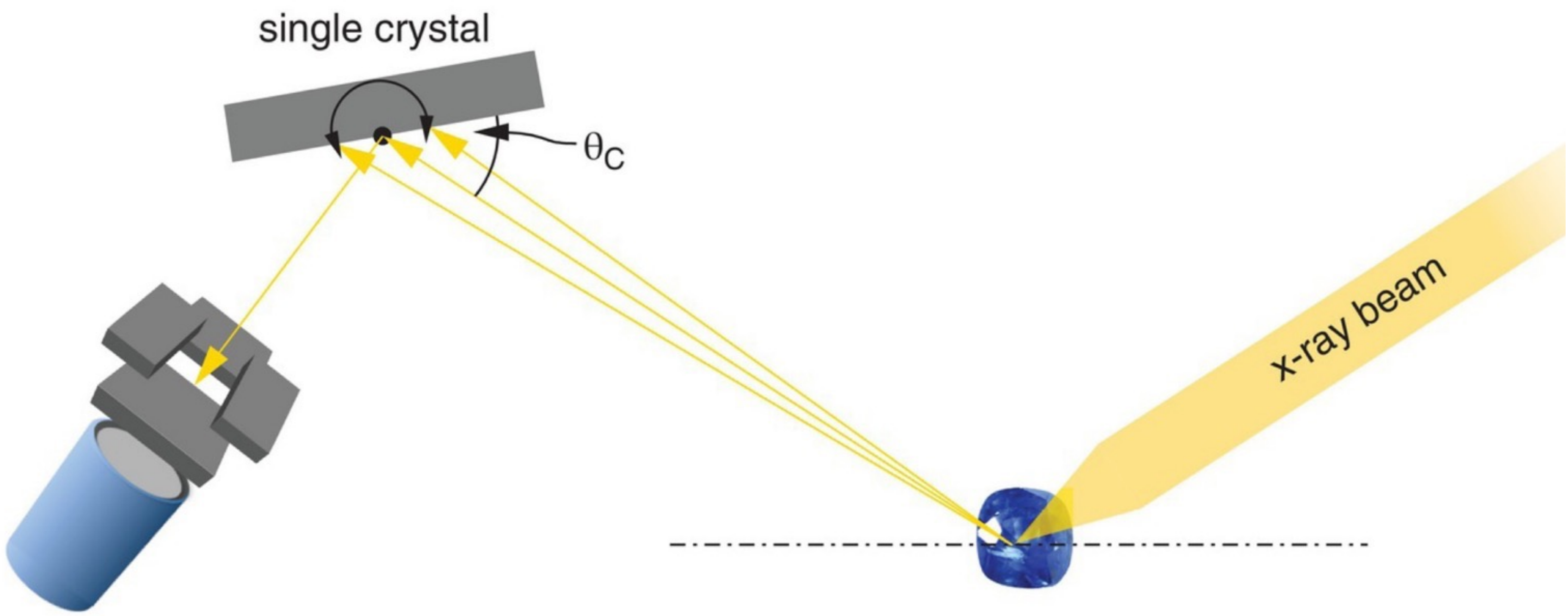
Crystal analysers

The introduction of a single crystal in the detector system to select the monochromatic diffracted signal can improve the resolution by precisely defining the angle between the incoming beam and the diffracted signal. In addition, it suppresses lower-energy fluorescence signal.



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Charge-coupled devices (CCD)

A CCD records images by converting photons of light into electrons

The electrons are temporarily accumulated in pixels on a photosensitive semiconductor chip.

At the end of an exposure, the accumulated charges are read off the chip

The signal is only read out after the exposure, CCDs are therefore termed ***integrating*** devices.

CCDs are used in conjunction with an overlayer of scintillator material typically a few tens of microns thick.

Advantage: small pixel sizes possible, well-established technology

Issues:

- Blooming causing streaks
- Dark noise
- Limited dynamic range

Single photon counters

Electronics comprise of a preamplifier, a comparator, and a counter.

- The preamplifier amplifies the charge generated in the sensor
- The comparator produces a digital signal if the charge exceeds a predefined threshold
- Counter for digital storage

No readout noise, no dark noise

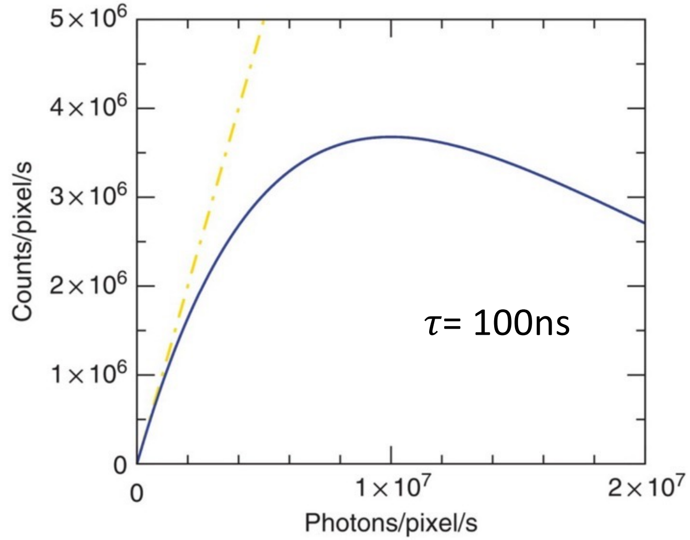
Can suppress x-rays with energies lower than a pre-defined value

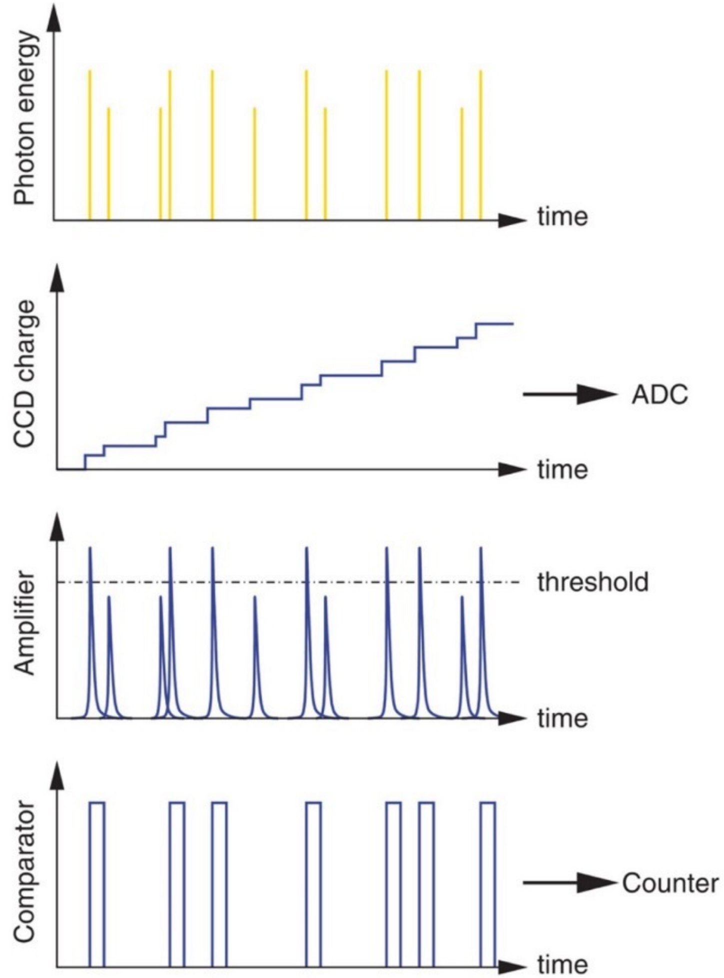
Cannot be used for XFEL experiments – too slow

Single photon counters

Further advantages:

- fast readout times (down to μs range)
- High dynamic range
- a high detector quantum efficiency
- Low dead time τ





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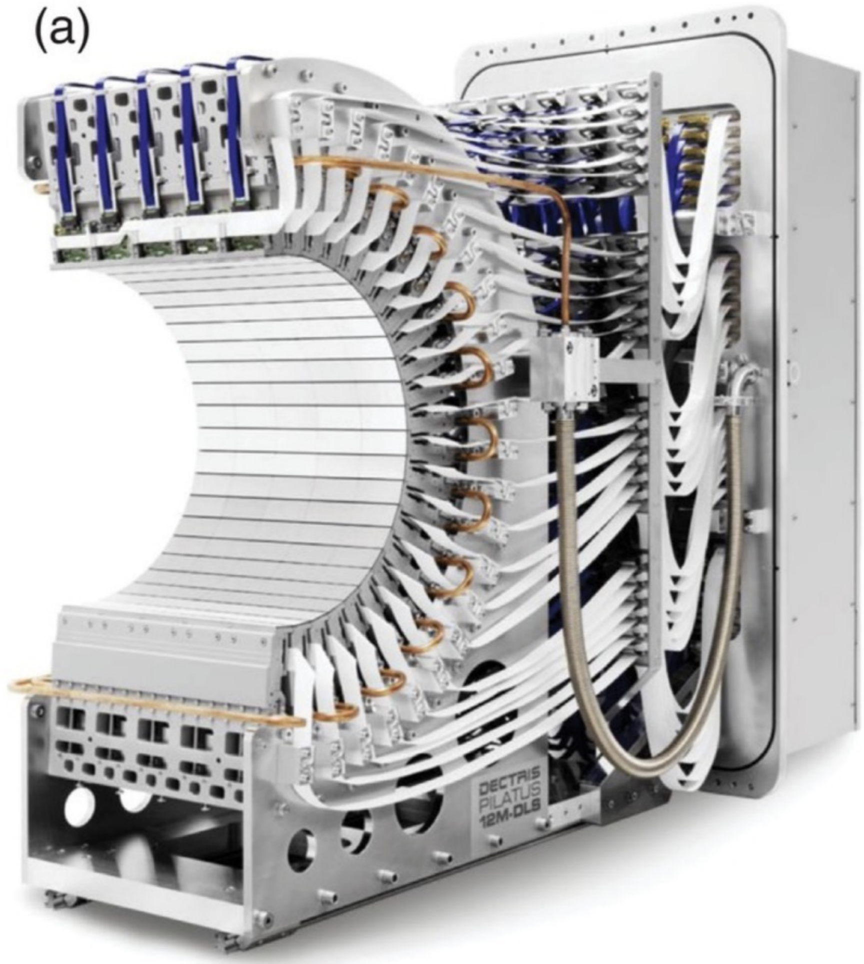
Single photon counter

- Custom-made vacuum-compatible Pilatus 12M detector for the I23 low-energy macromolecular crystallography beamline at the Diamond Light Source, UK.
- The detector is composed of 5 × 24 modules, each module containing approximately 100’000 pixels.
- The detector covers an angular range of 200 degrees



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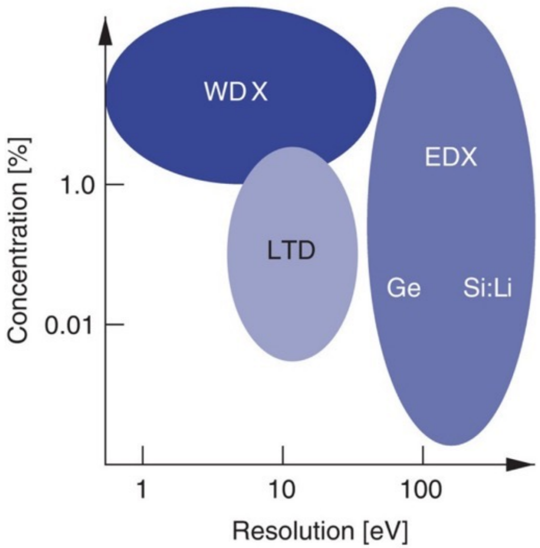
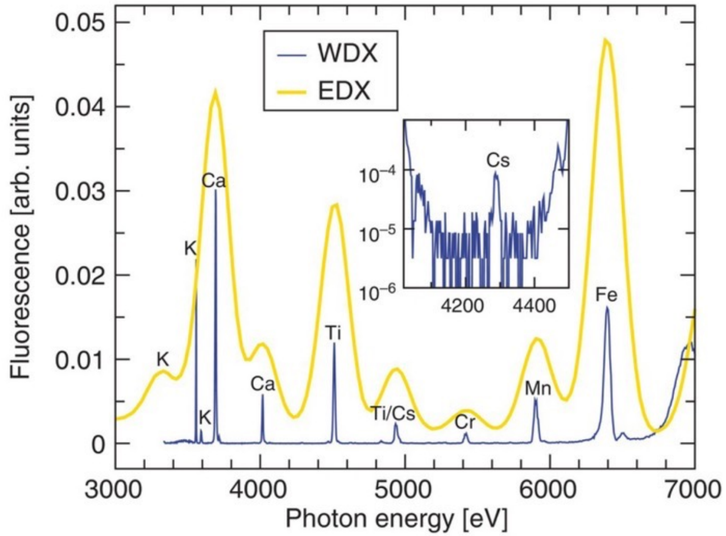
Energy dispersive detectors

Solid state detector

- Si(Li) or Ge technology
- Upon interaction with an x-ray photon, electron–hole pairs are created that drift in an applied high electric field.
- The charge is collected in a manner similar to the charging of a capacitor
- The voltage increment due to the collected charge is proportional to the photon energy





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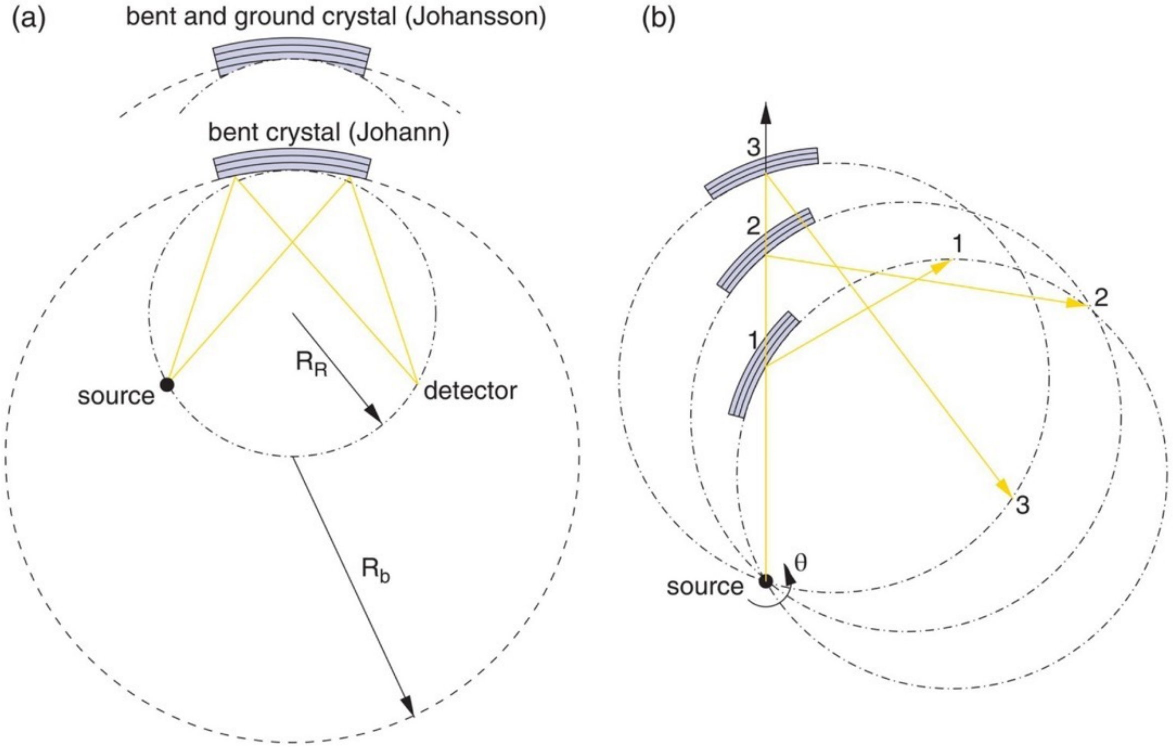
Energy dispersive detectors

Wavelength-dispersive spectrometers (WDS)

- the photon energy is selected by diffraction on a single crystal or a grating
- High energy resolution



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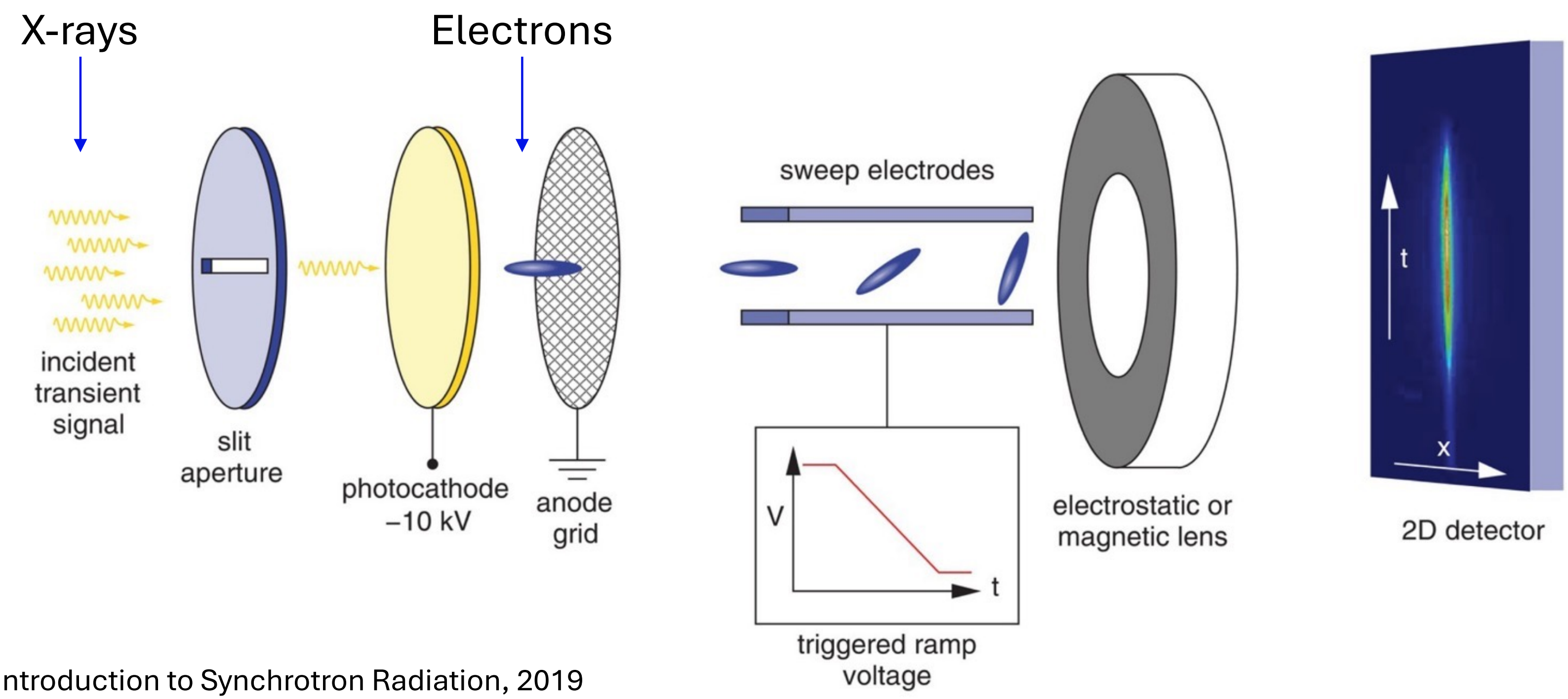
Streak camera

- 2D image with one coordinate corresponds to time and the orthogonal axis to space
- Time resolutions down to 250 fs



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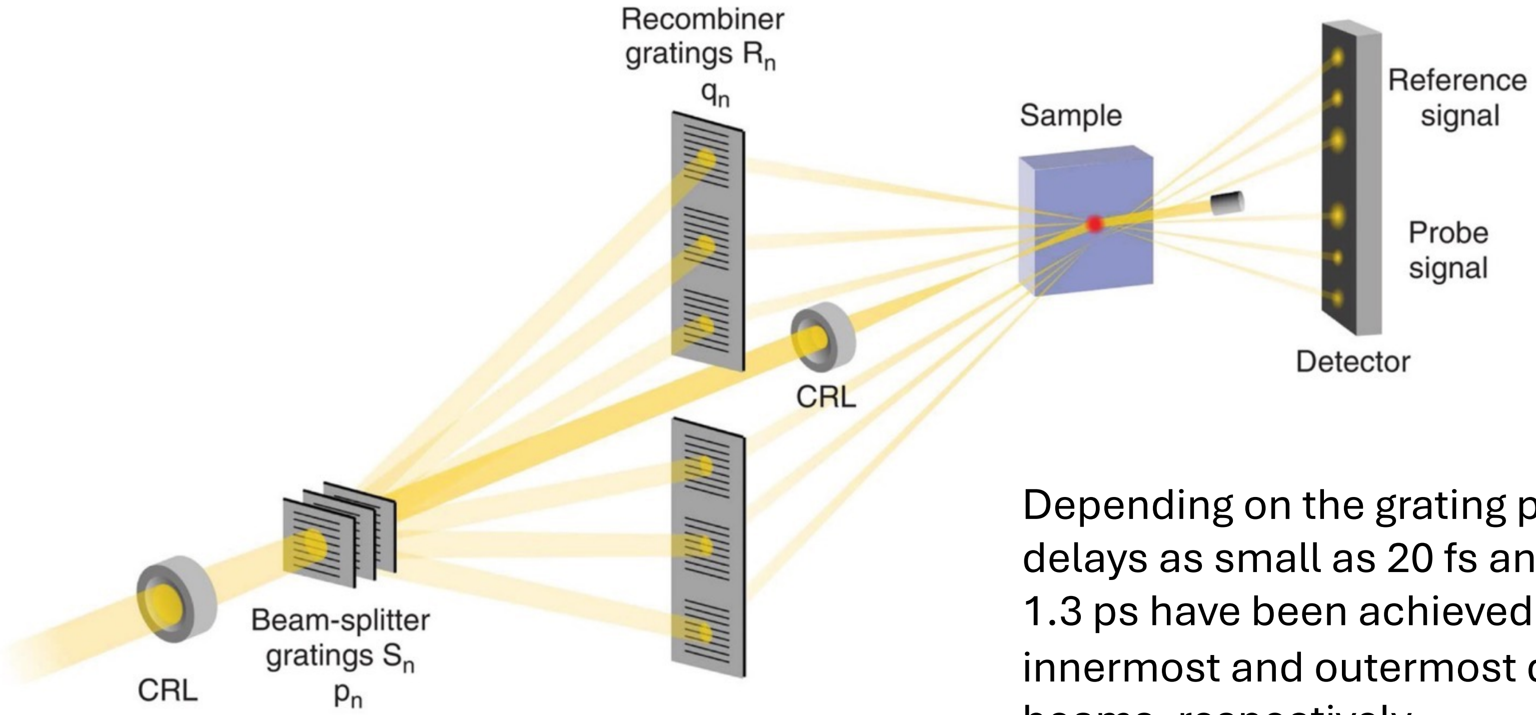
Streaking at XFEL

Split and delay principle



Depending on the grating periodicity, delays as small as 20 fs and as long as 1.3 ps have been achieved for the innermost and outermost diffracted beams, respectively.

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