X-ray Detectors for Synchrotrons

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*with thanks for contributions from P Fajardo- Th Martin- C. Ponchut- ESRF & H. Graafsma-DESY

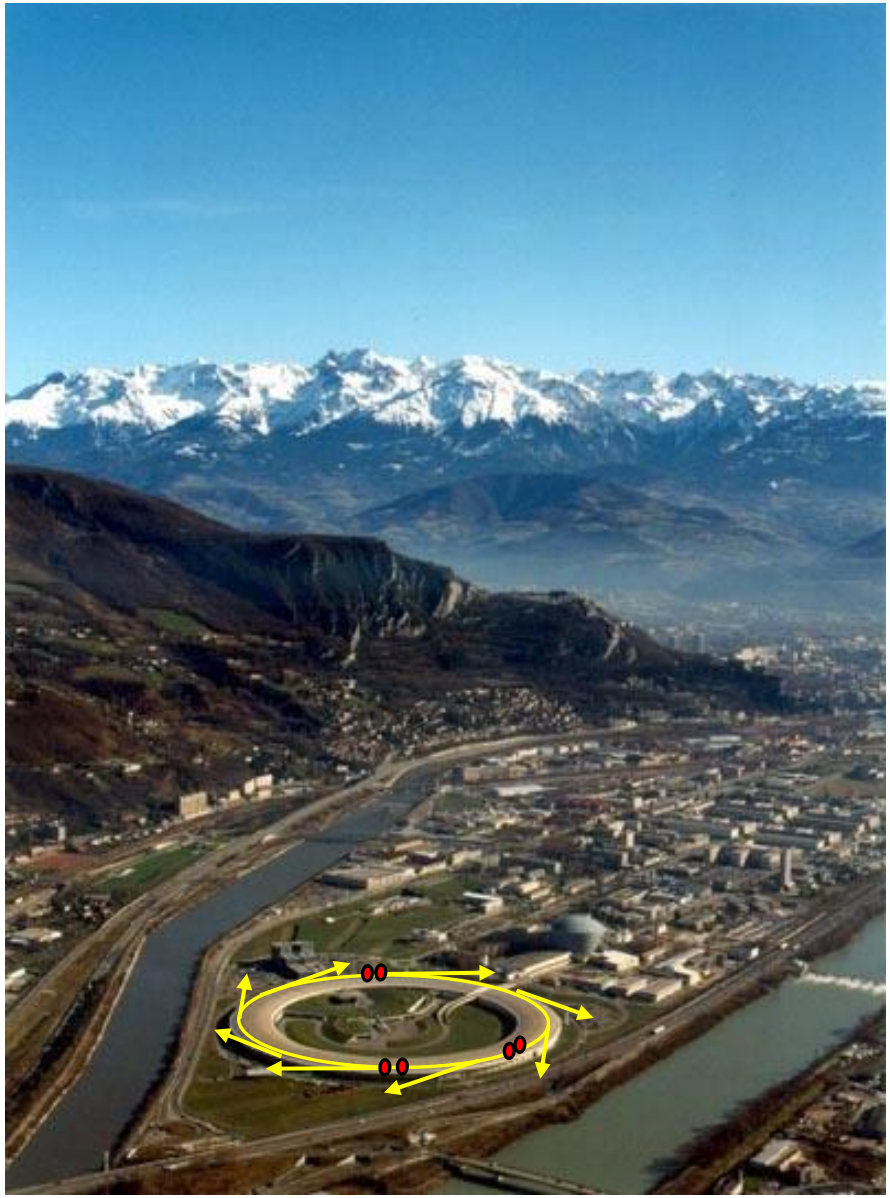
Talk Outline

1. 3rd Generation synchrotron sources: a (very) quick introduction

2. what are we trying to measure?

3. different experimental methods, and some of the major detectors in use

-- all dosed with a little bit of basic X-ray and detector physics.



European Synchrotron Radiation Facility

ESRF

50 beamlines in operation

7000 external users / year

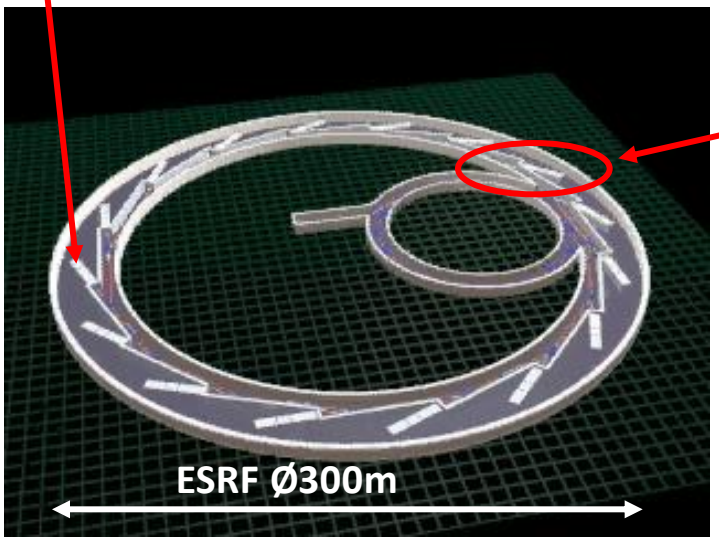
high intensity, semi-coherent X-ray beams
~0.5keV – 0.5MeV for diffraction and inelastic scattering studies

basic and applied research in:

biology (e.g. protein structures…)
materials science
chemistry & catalysis…
2D- & 3D (coherent, diffractive…) imaging
archaeology-paleontology
…

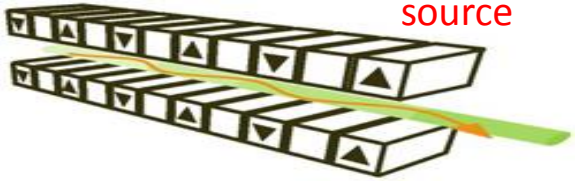
-- at molecular & atomic scales…

~ 50 beamlines



energy tunability

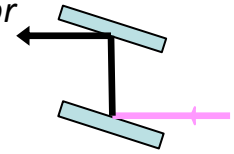
undulator source



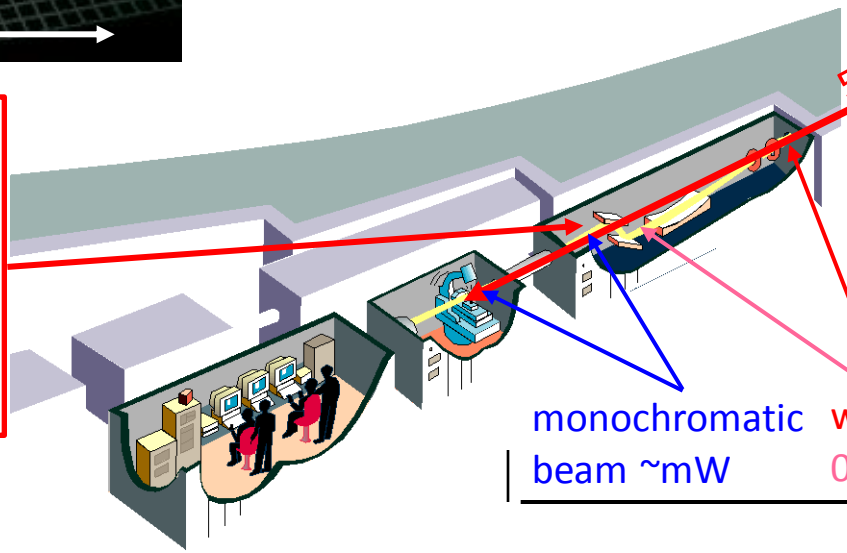
'white' X-ray beam
$\Delta E/E \sim 10^{-2}$

Silicon crystals

monochromator
X-ray beam out
$\Delta E/E \sim 10^{-4}$



Incident pink/white beam radiation



50~100m source to end station

monochromatic beam ~mW

white/pink beam 0.1~1kWmm^{-2}

X-ray beams: some typical numbers

beam *at the sample*

Size:	- Unfocused beam:	few millimeters to a few cm (source is divergent)
	- Focused beam:	<100 *nano*meters to 10's of microns
Energy range:		<0.5 keV to 0.5MeV, but mostly 3 … 100keV
Energy bandwidth (ΔE/E):		10^{-2} to 10^{-8} at sample, typically ΔE ~ few eV @ 20keV
Photon flux (@ ΔE/E = 10^{-4}):		10^{9} - 10^{14} ph/sec ('online' attenuators)

but extremely variable photon rates incident on the detector(s)

from <1 photon /sec up to 'near full beam' flux

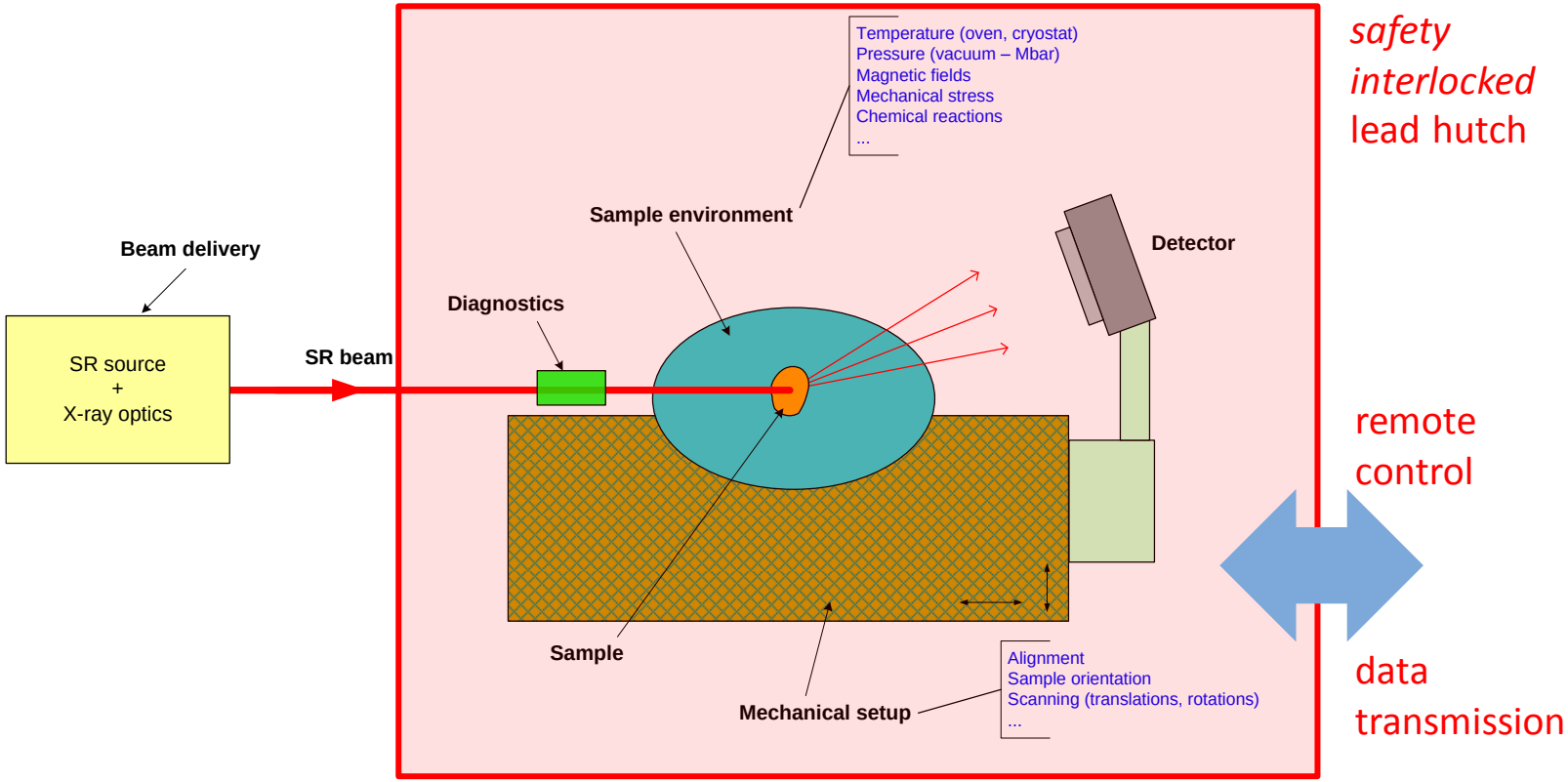
nb. X-ray ionizing radiation dose = sample damage !

e.g. at 10^{12} ph/s and10 keV photons , *dose rate ~10 Gray/sec* in a silicon

experiments are built around the samples to be measured

--> importance of sample environment (temperature; pressure; E and B fields…)

need to physically manipulate sample during measurements (position, rotation…)

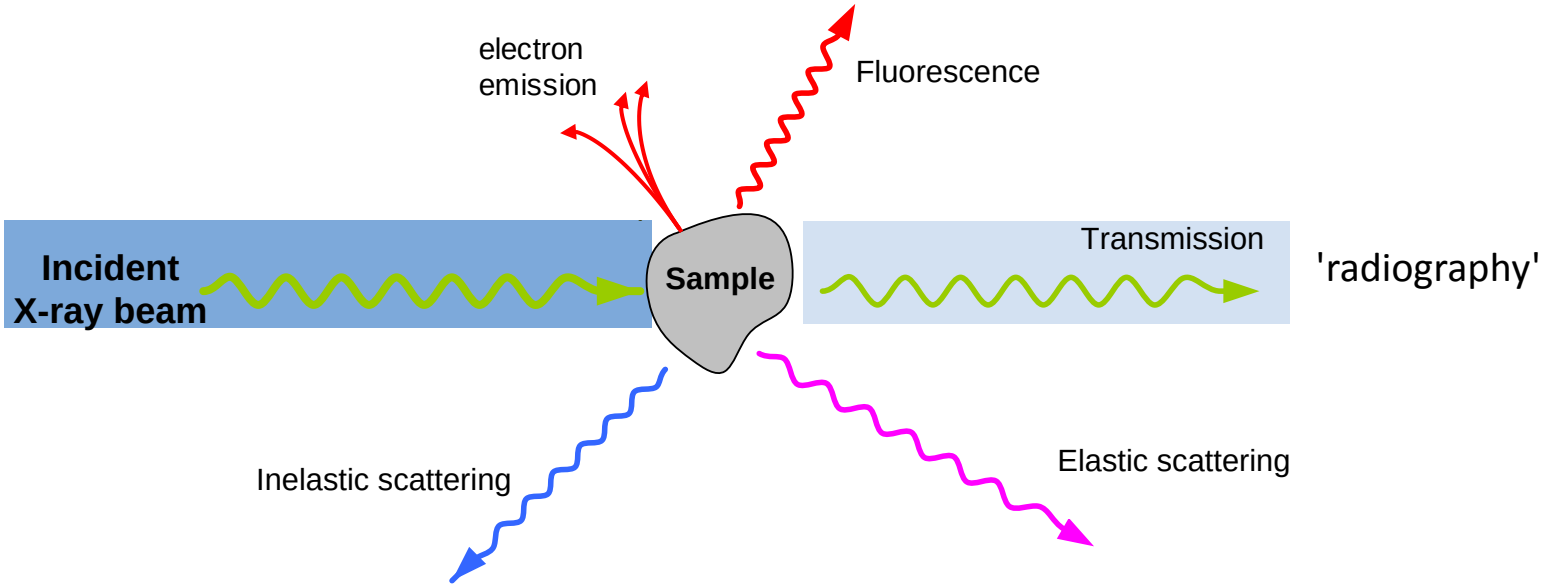


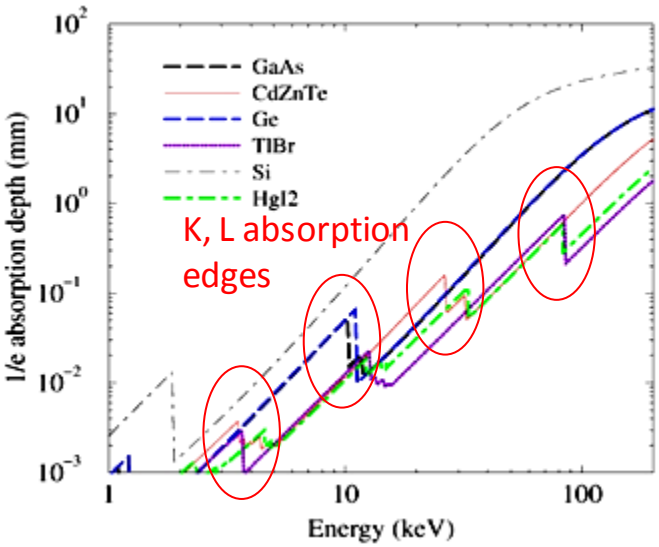
rapid turnover of samples and experiments

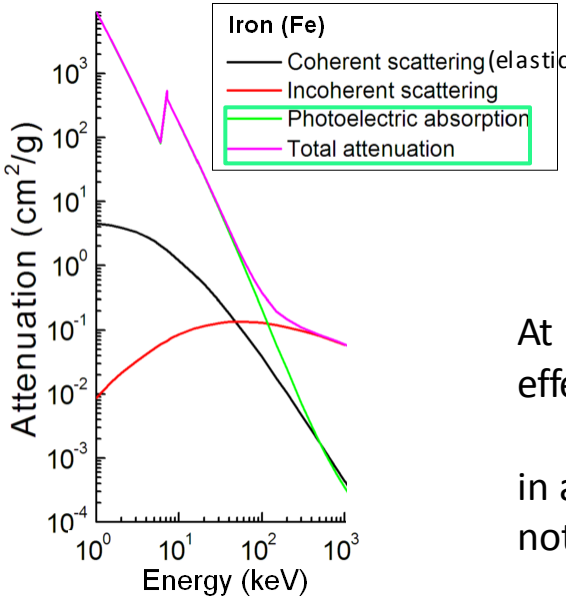
sample(s) in beam for minutes to hours

experiments typically lasts a few days

what is measured ?







Beer's law:

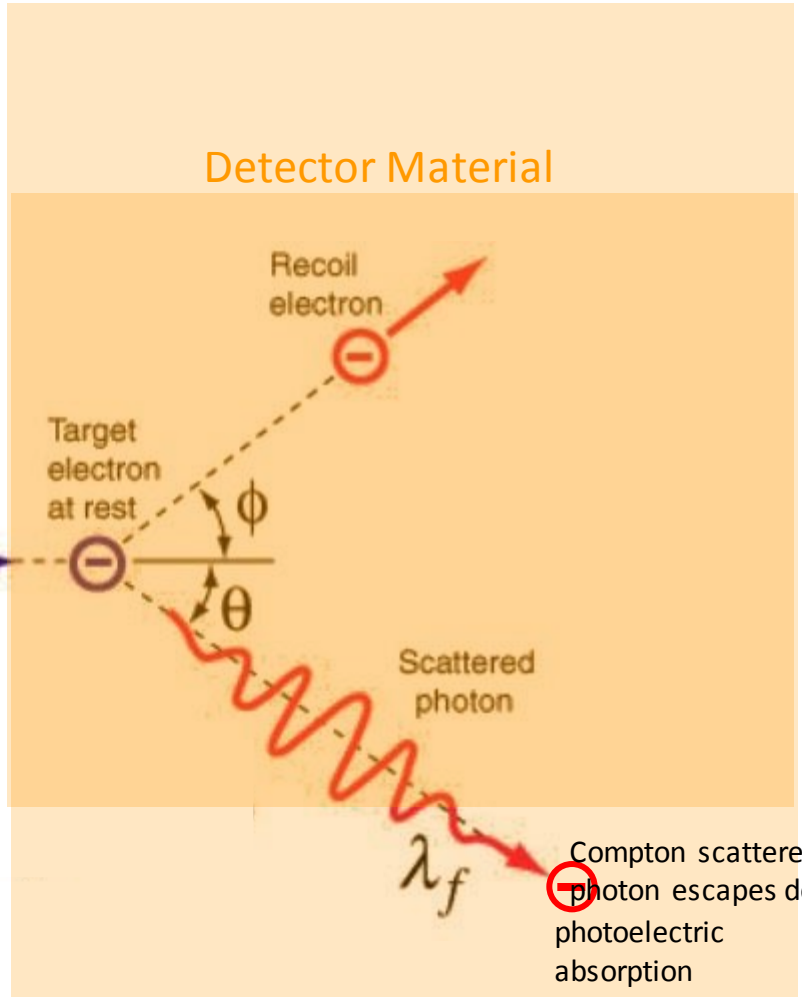
$$I(x) = I_o \exp(-\mu(E).\ x)$$

intensity of a photon beam decreases exponentially with distance into the material,
but the energy of photons in the transmitted beam remains the same.

note that μ(E) is discontinuous at 'absorption edge' energies, corresponding to the atomic shell structure binding energies

At 'low' synchrotron energies, the photoelectric effect is usually the dominant interaction

in a detector, incoherent (Compton) scattering is not desirable…



$$\Delta E = E_0 - \frac{E_0}{1 + \frac{E_0}{mc^2}(1 - \cos 2\theta)}$$

and $E = hc/\lambda$

X measured energy = Compton recoil electron only

✓ measured energy = Compton recoil electron + Compton photon)

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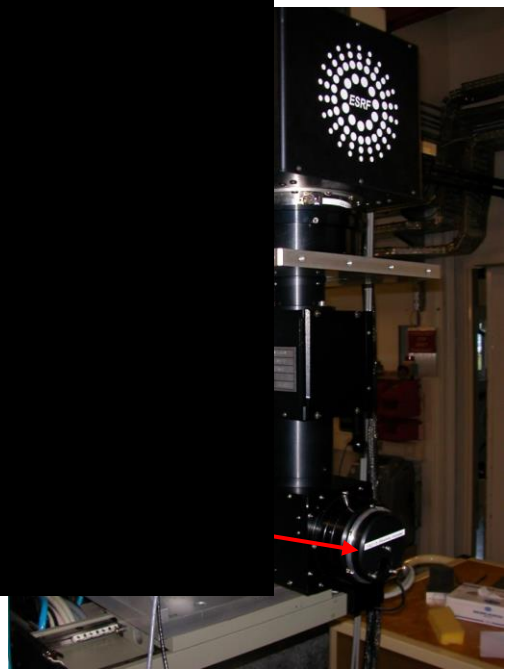


Paul Tafforeau, ESRF

- Scattered photons *conserve their initial energy*, i.e. only momentum changed

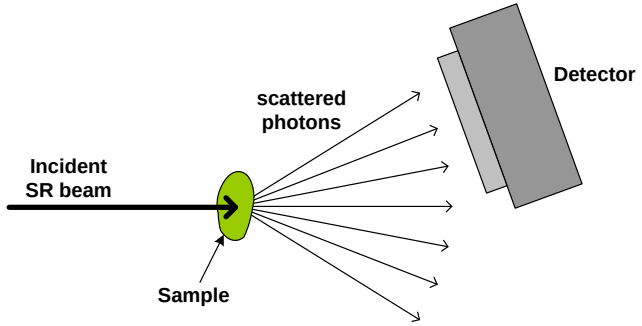
  angular measurements are required, usually measured with 2D spatially resolving detectors

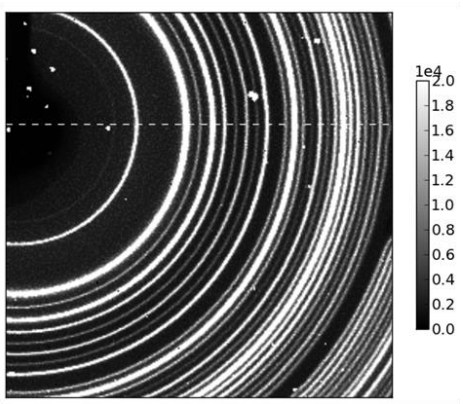
  --> angular resolution can be varied with changing detector-sample distance

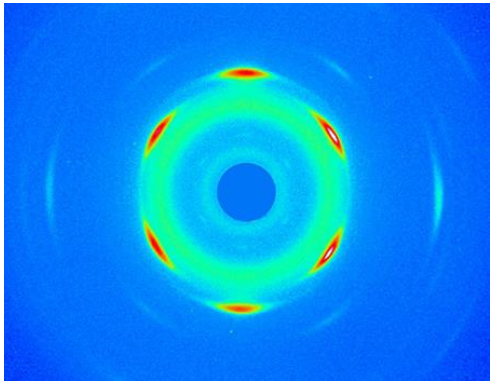
- Large dynamic range may be required (crystal diffraction can cover ~8 orders of magnitude!)

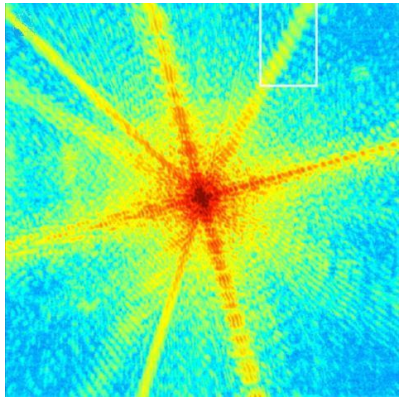
- detectors used:
  - scintillator-PMT; silicon-diode, -APD using diffractometer
  - solid state semiconductors (1D strip, 2D area PADs)
  - 'indirect detection' with flat panel a-Si or CMOS readout
    2D area scintillator-optic-CCD cameras

  (laser read-out phosphor image plates, MWPC gas detectors)

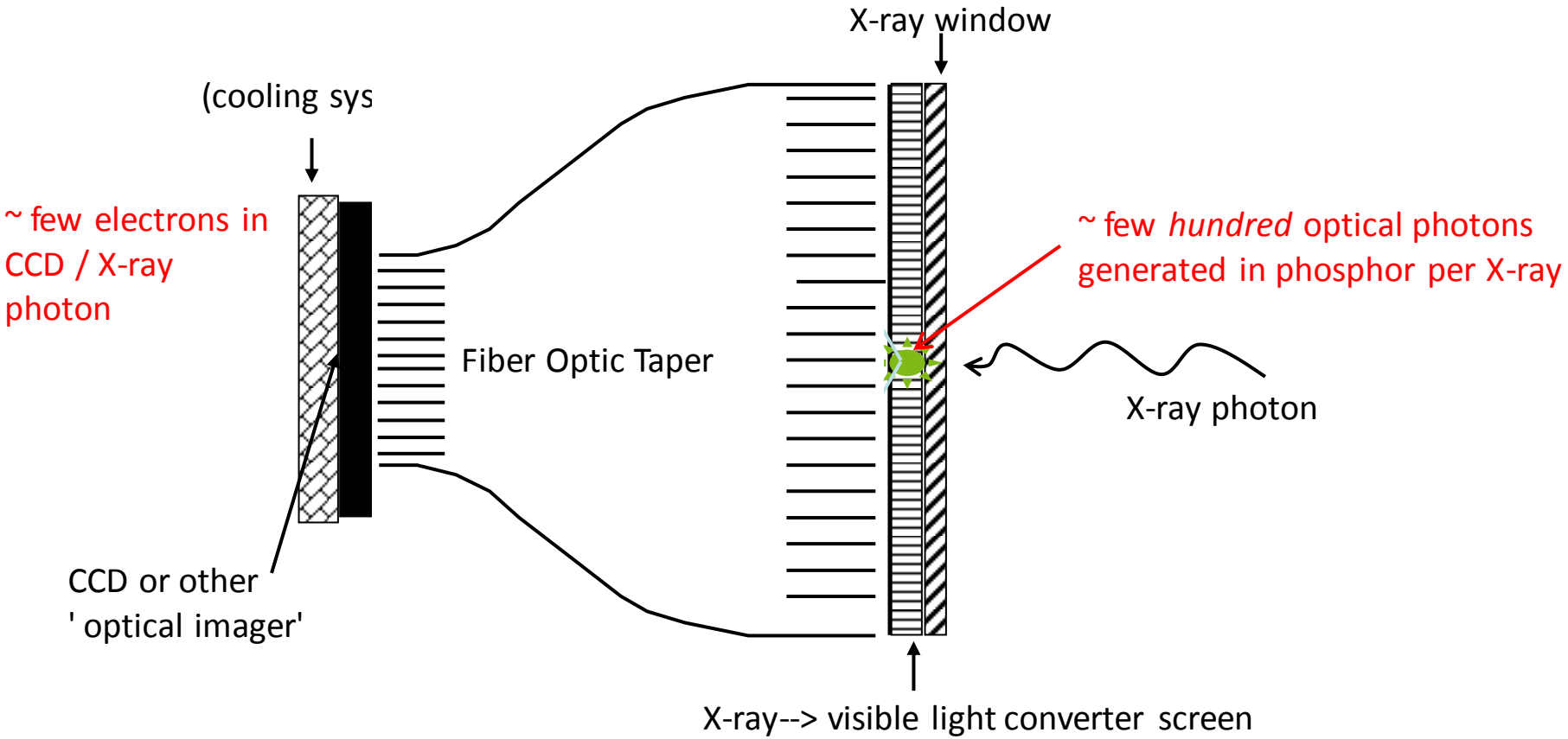






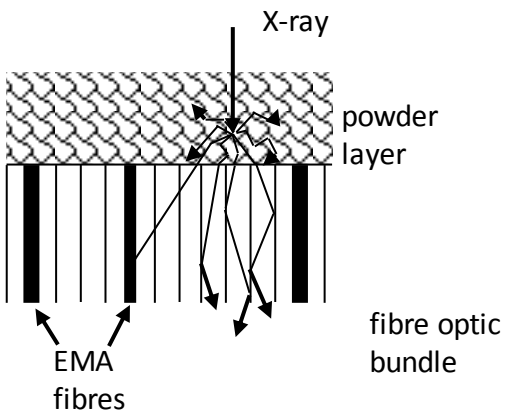


position-resolved 2D area detectors, especially high resolution X-ray imaging



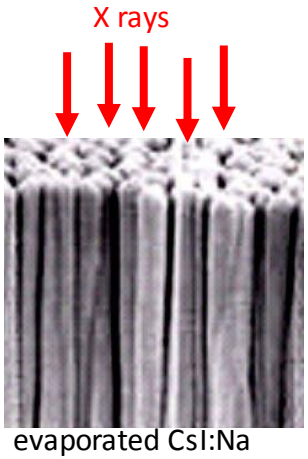
for detector areas above ~1cm^2, fibre optic gives more efficient optical coupling to CCD

powdered crystal 'phosphor' screen



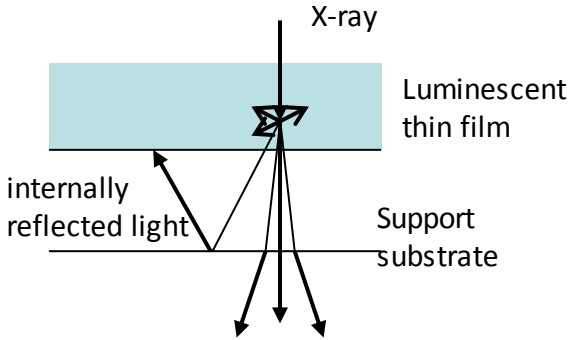
Spatial resolution limit ~5µm fwhm median particle sizes (P43 'Gadox') 2.5µm to 30µm

structured crystal growth scintillator

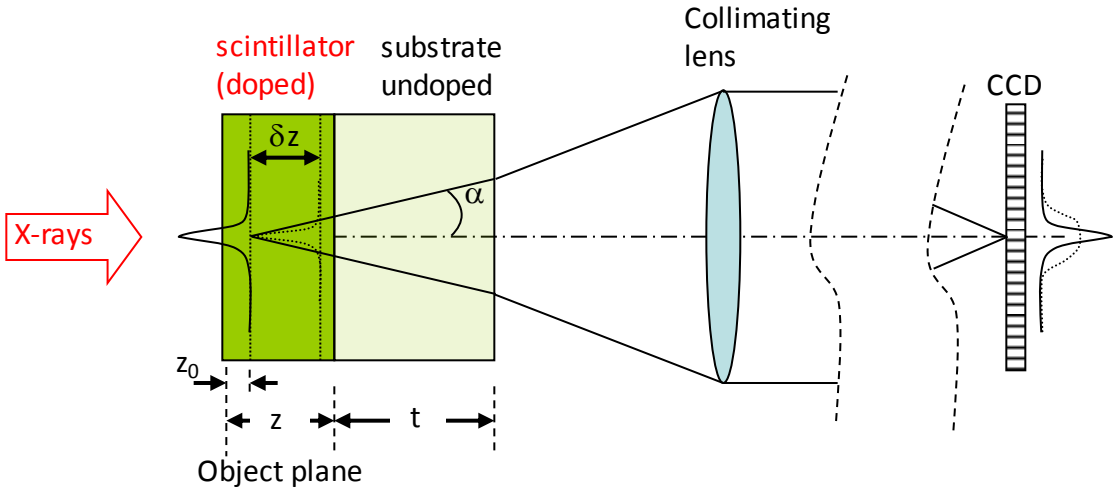


need for encapsulation (fragile, hygroscopic)

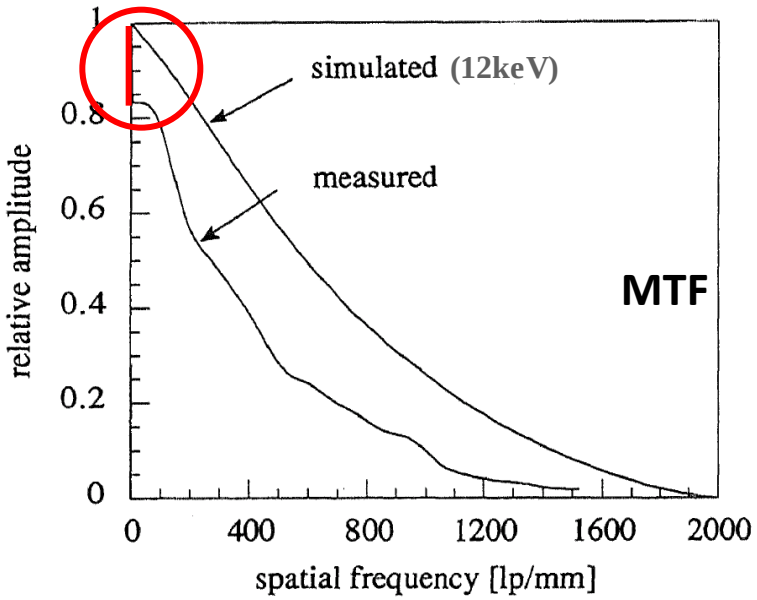
single crystal screen



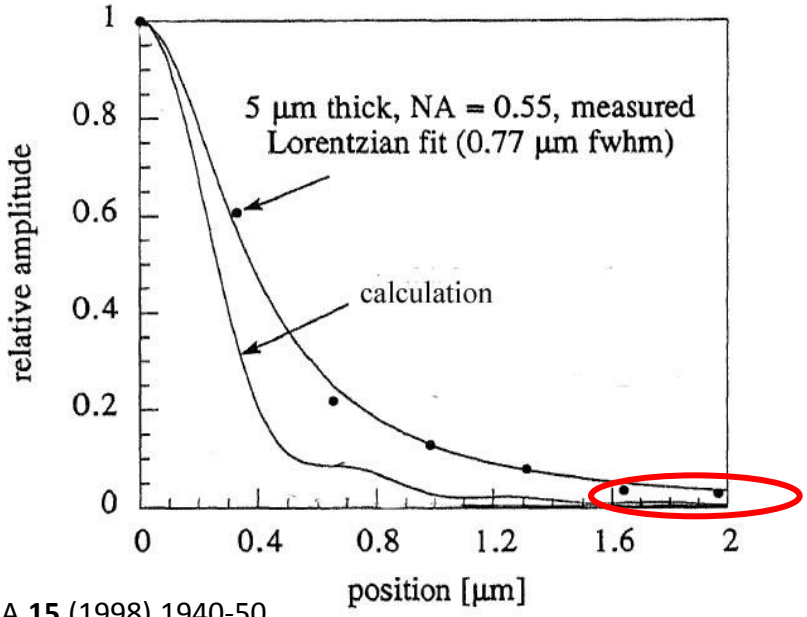
resolution may approach optical diffraction limit, i.e ~0.5µm fwhm in 'thin' screen limit max' size ~1cm



e.g. doped YAG:Ce by liquid epitaxy on undoped 170µm YAG substrate,
X-rays, 100x magnification optics
CCD 24 µm pixel sampling

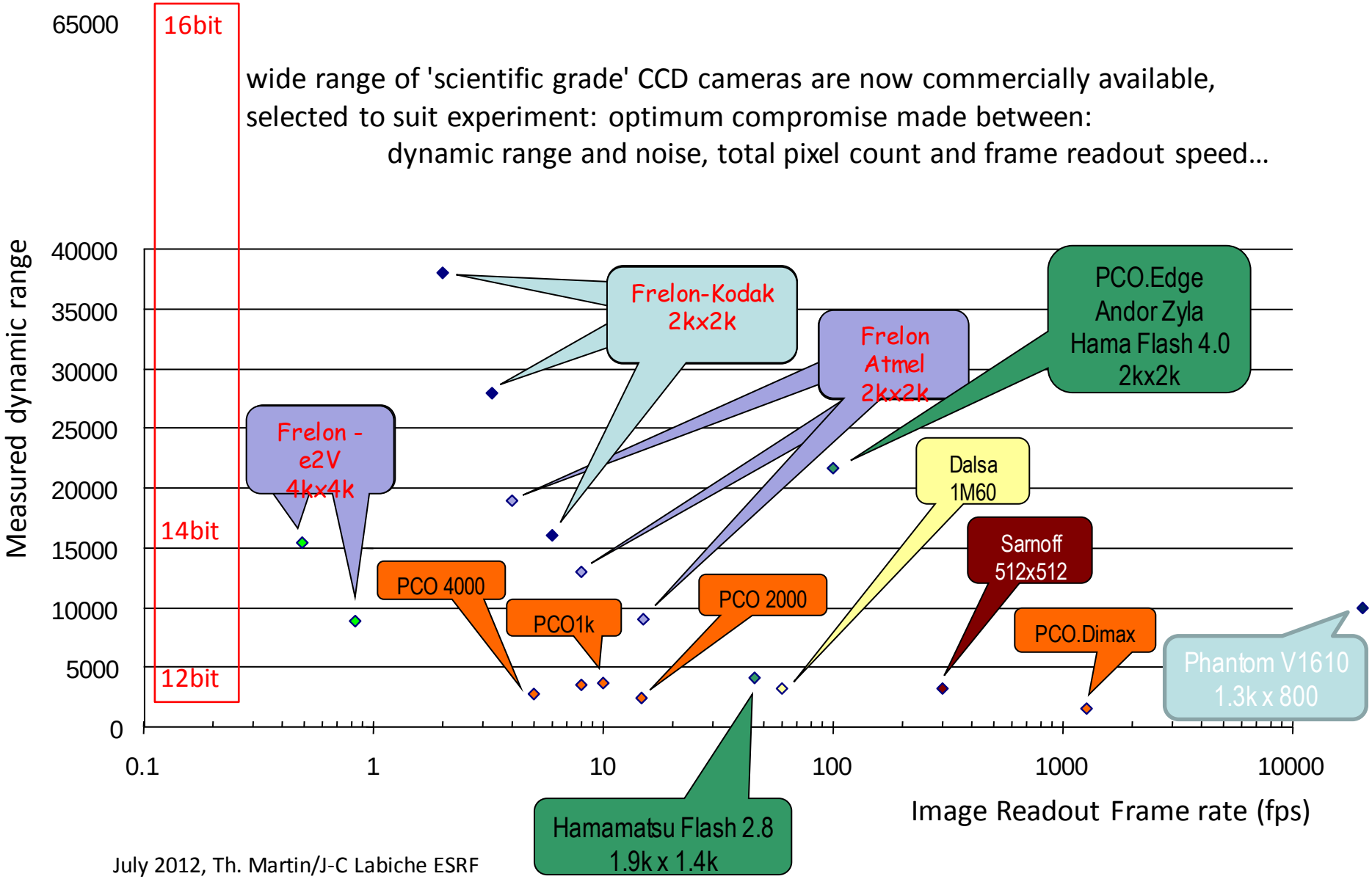


MTF → **LSF**



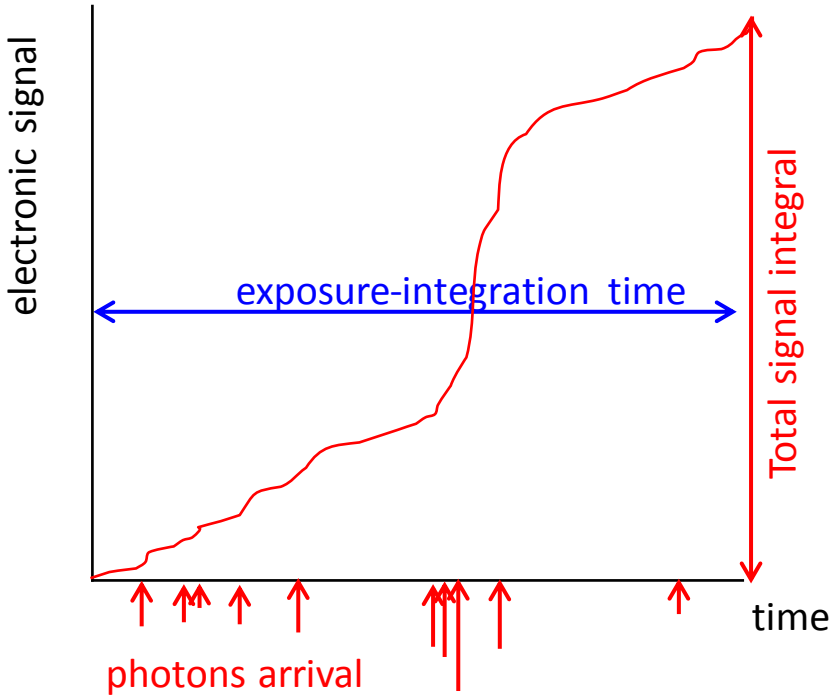
Koch et al, J. Opt. Soc. Am. A **15** (1998) 1940-50

CCD camera performance



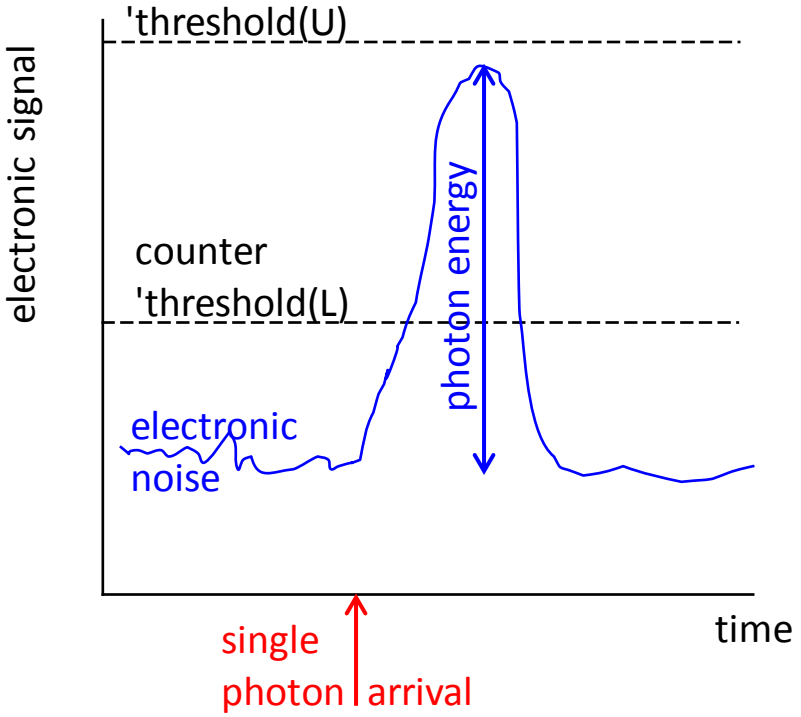
July 2012, Th. Martin/J-C Labiche ESRF

integrating detector:
signal resulting from arrival of (usually) many photons is only sampled at the end of an 'exposure' period



signal response *remains linear even at very high photon rates*,
unless detector 'saturates'

energy resolved and counting detectors:
individual X-ray photons are detected 'one at a time'



removes detector noise contribution to signal,
permits *energy resolved photon detection*
…but signal response shows 'count pile-up'

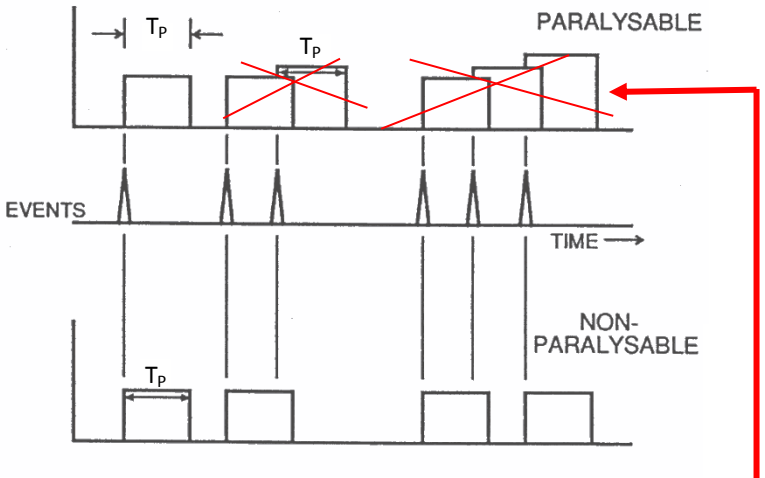
Counting detectors are usually 'non-paralysable': two events are counted as a single event if they are separated in time < T_P the event 'dead-time' . Measured count rate response is non-linear, reaching a plateau value for high event rates.

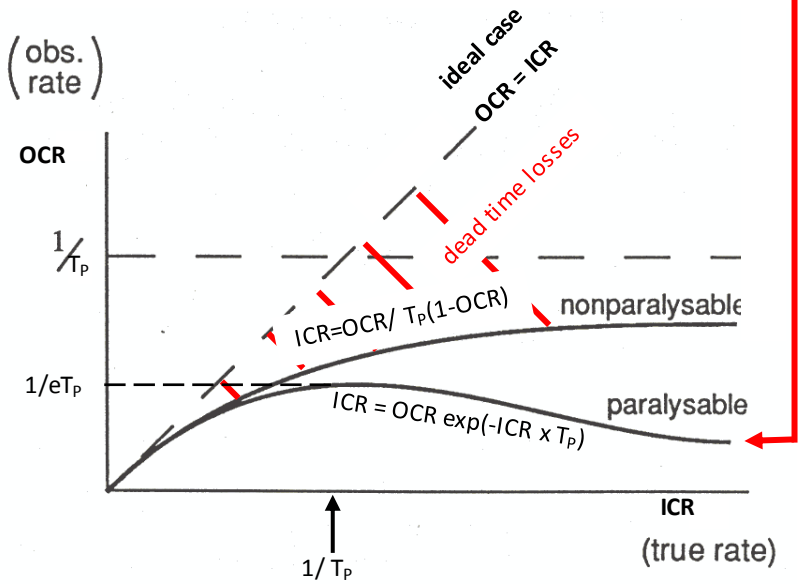
X-ray *energy resolving* detectors *must be* 'paralysable' i.e. time-overlapped pulses must be rejected, else they will be recorded as false events in the measured energy spectrum histogram

For Poisson time-distributed events, e.g. from a radioactive source, the true input event rate (ICR) at the detector can be obtained from the output spectrum:

$$ICR = OCR \exp(-ICR \times T_P)$$

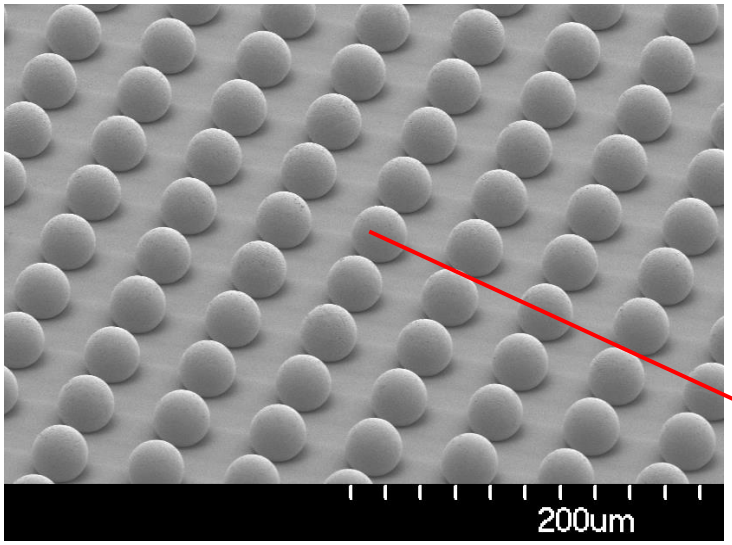
$T_P \approx$ 5x filter 'shaping time' of the pulse

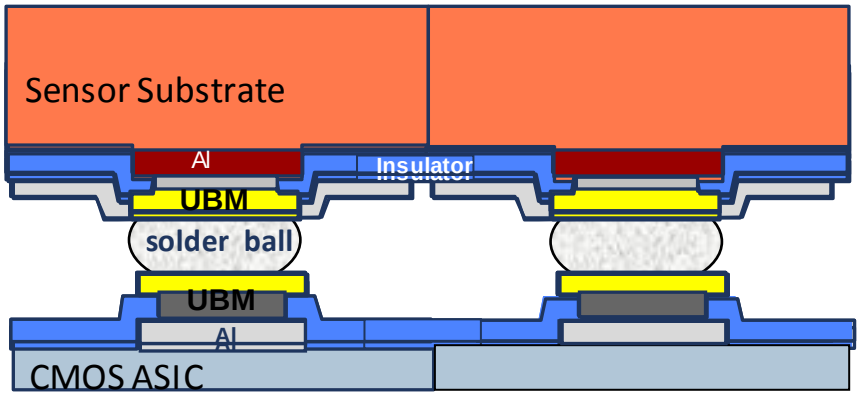


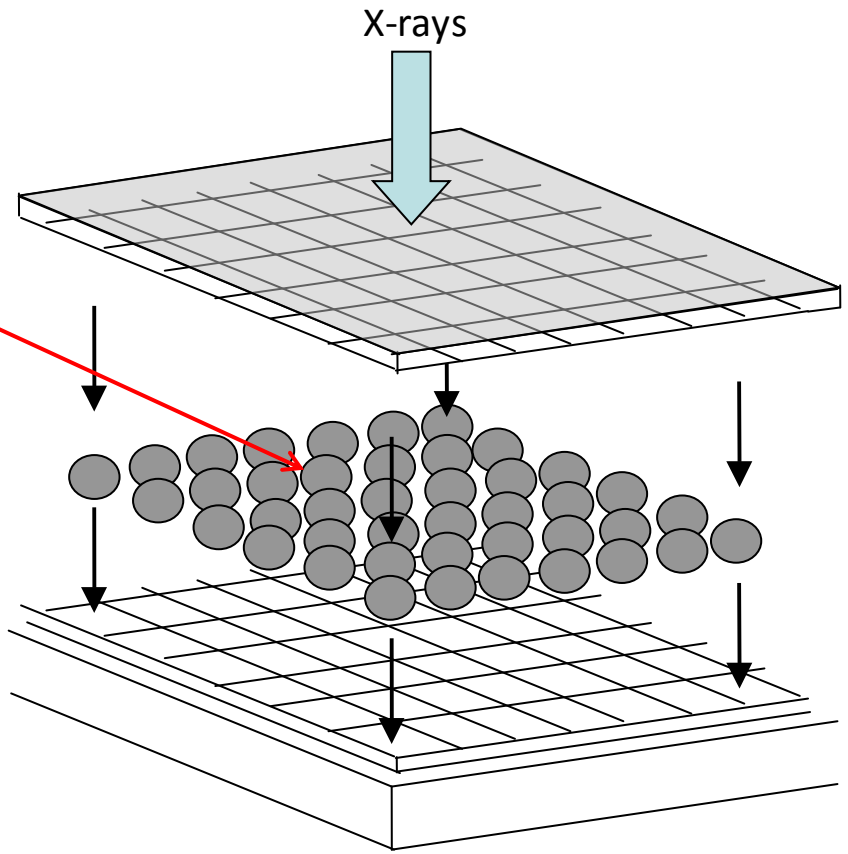


to count faster, use multichannel, parallel-counting 'pixel detector ' systems

Pixel Array Detectors (PADs)-1

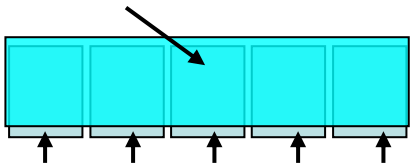






ESRF ' Maxipix' system , uising Cern 'MEDIPIX-2' ASIC and silicon sensor

monolithic Si sensor

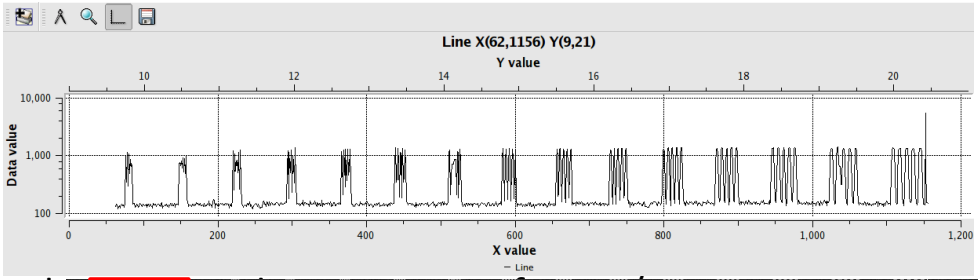


ASIC readout chips

5x1 assembly: 1280 x 256 pixels
71.2 x 14.1 mm^2 active area

350 Hz frame rate (5x1 chips) using parallel readout mode

55 x 55 µm^2 pixel size --*high spatial resolution*



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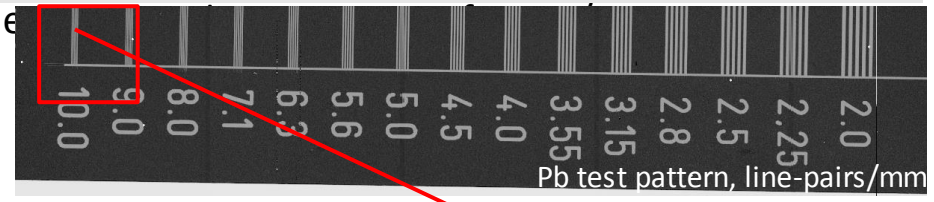
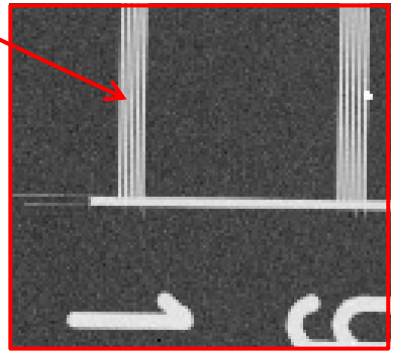


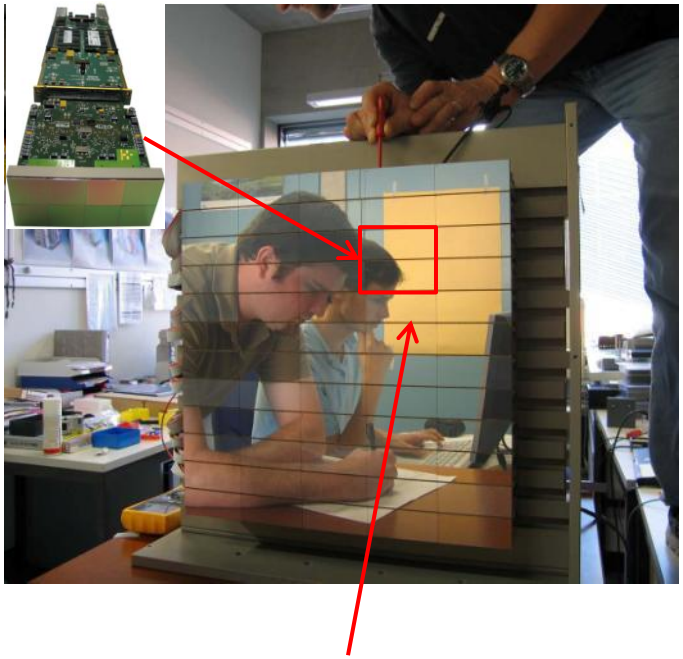
Pb test pattern, line-pairs/mm

Moiré effect, pixel sampling of spatial frequency close to or below the *Nyquist sampling limit = 1/2f*

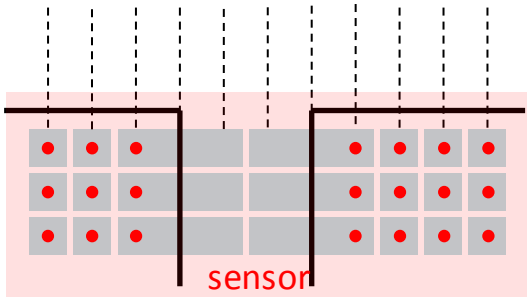
f =spatial modulation frequency of signal



Paul Scherrer Institute developed ASIC (counter) and silicon sensor 'Pilatus' Systems sold by Dectris Ltd.



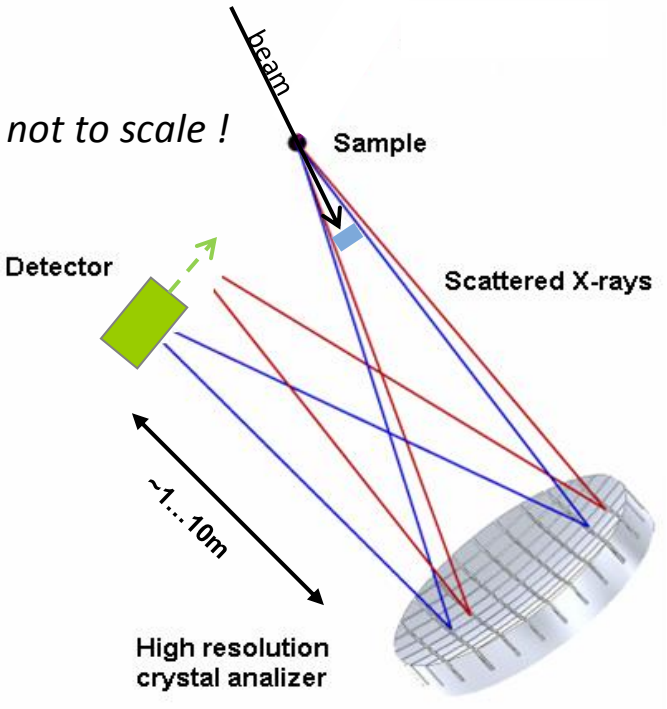
insensitive areas, typically ~1mm between ASICs:



for large areas and fast readout, silicon PADs have completely replaced MWPC gas detectors at synchrotrons:
higher global count rate capacity, reliability …

Inelastic scattering

- measurement of 'recoil' energy transferred to the sample by the individual X-ray photons
- energy transfer is small: high energy resolution required: 1meV ~ 1eV (e.g. for 10keV X-rays)
- Use of diffractive crystal *energy analyzer* detection arrangement:



- highly monochromatized beam and weak interaction cross sections
  --> low photon detection rates
  beam on sample 10^{12} photons/sec, but possibly < 1 photon/sec on detector
- use of a position sensitive detector may 'improve' effective energy resolution *and* useful count throughput of crystal analyzer
- detector energy resolution useful to reject stray X-ray background

Possibly measured simultaneously:
- sample absorption
- sample X-ray fluorescence
and polarization dependence (e.g. sample *dichroism*)

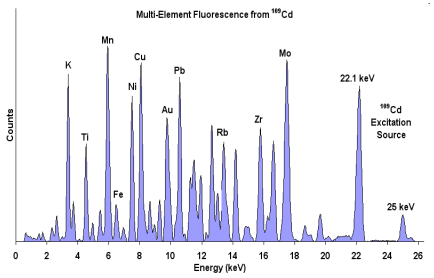
often *measured as function of incident beam energy*, sample environment…

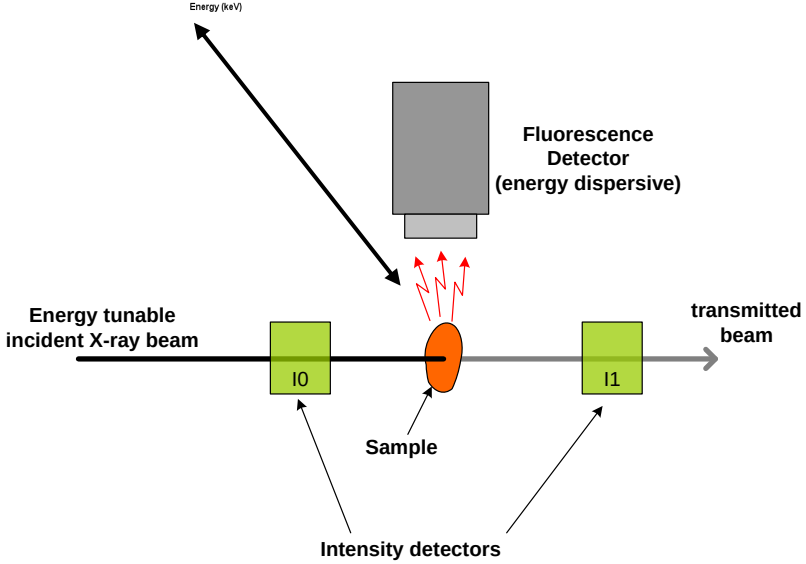
detectors:

for transmitted beam intensity:
gas ionization chambers, photodiodes

for emitted fluorescence:
Si, Ge semiconductor detectors

diffractive crystal analysers
cryogenic bolometers / STJ's



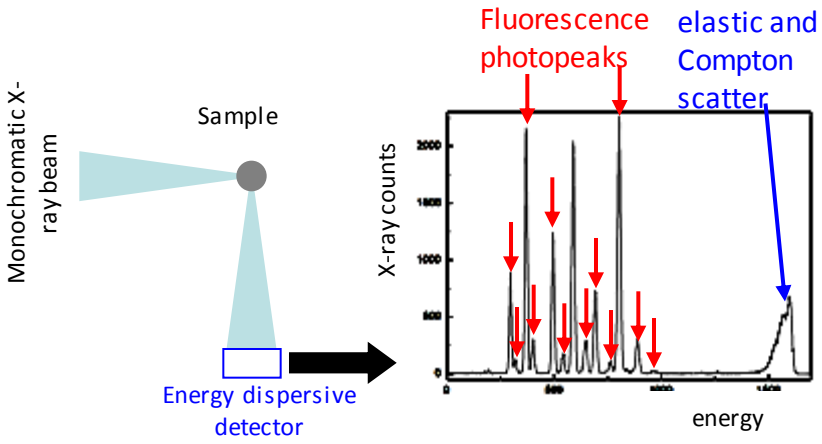


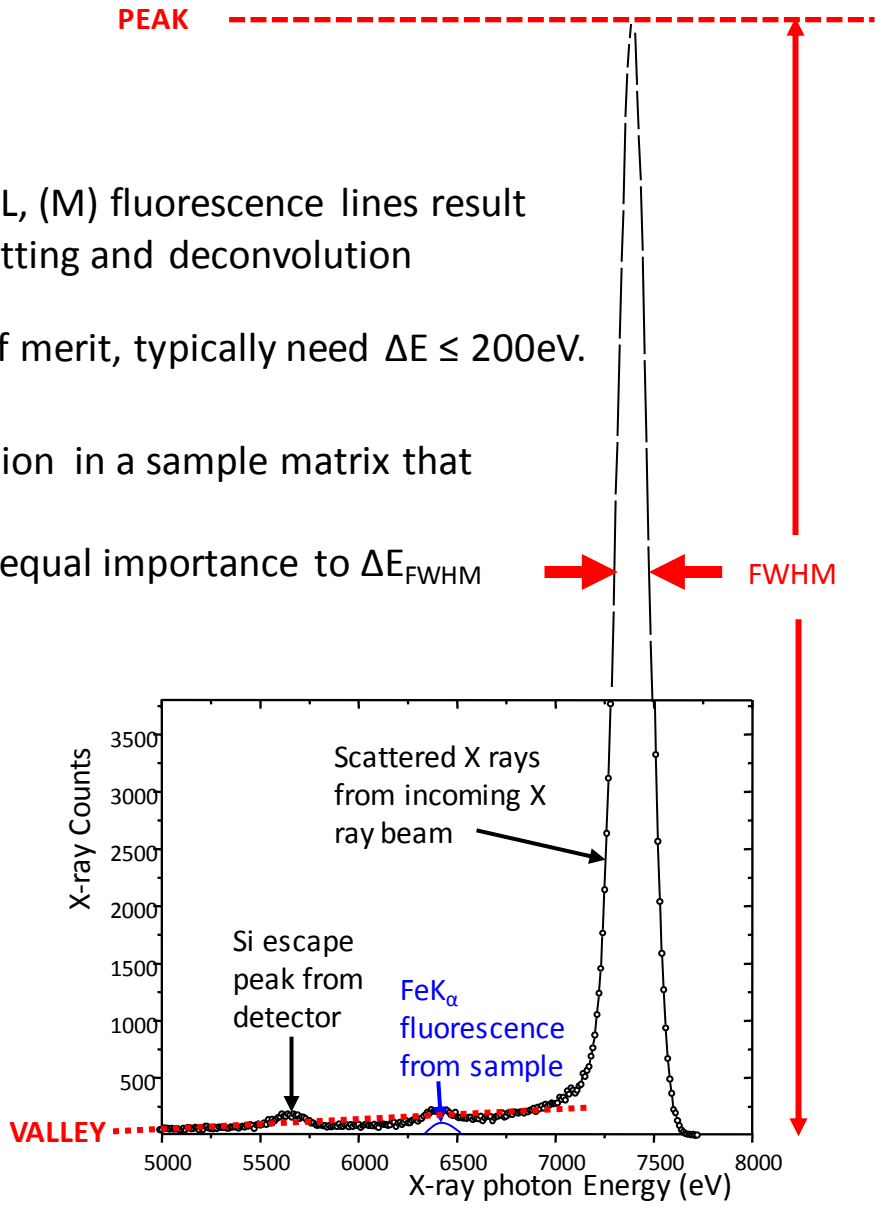
Often need to identify/quantify elements in the sample.

If the sample contains many elements, then multiple K, L, (M) fluorescence lines result in complex energy histograms requiring accurate peak fitting and deconvolution

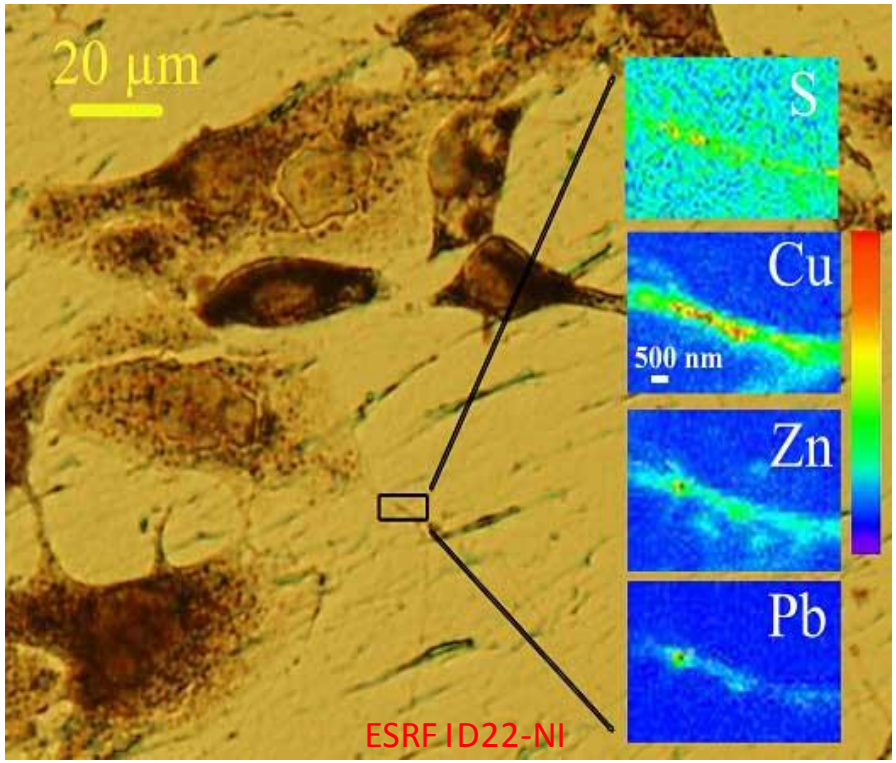
Energy resolution '*FWHM*' is the usual detector figure of merit, typically need $\Delta E \leq 200eV$.

For trace element analysis we may seek < parts per million in a sample matrix that itself fluoresces *and* scatters the incoming beam…
'peak-to-valley' performance of the detector may be of equal importance to ΔE_{FWHM}





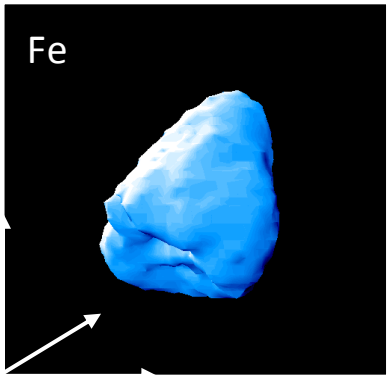
Fluorescence 2D energy resolved 'imaging' of samples

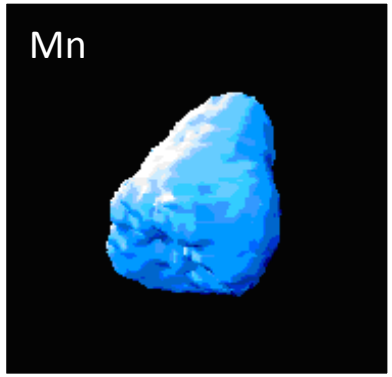


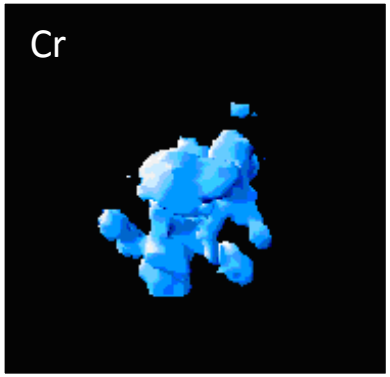
Neurite process
A Carmona et al.
JAAS (2008)

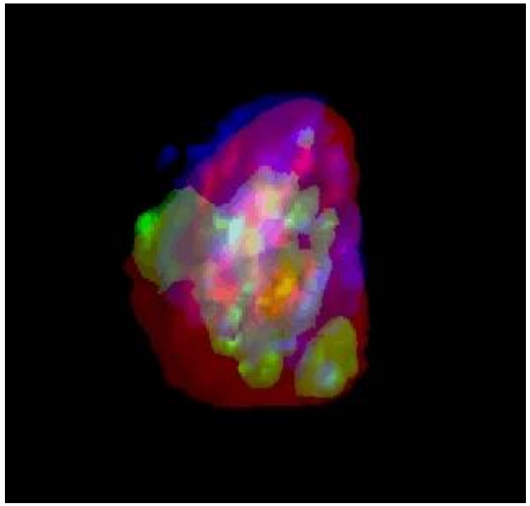
Rotation of the sample enables *3D mapping* of element distributions

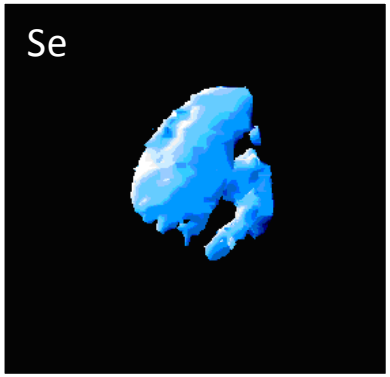
NASA *'stardust'* sample 0044-Track 3/terminal particle: heterogeneous on the submicron level, Fe-rich olivine

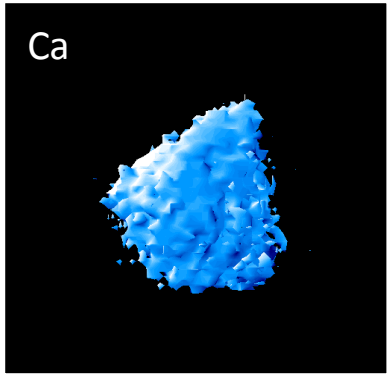


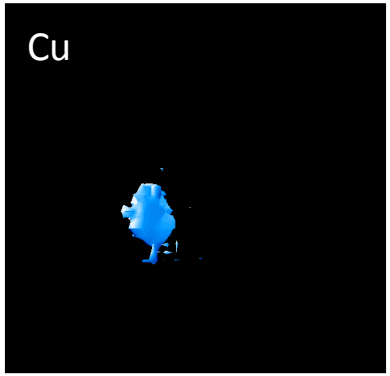










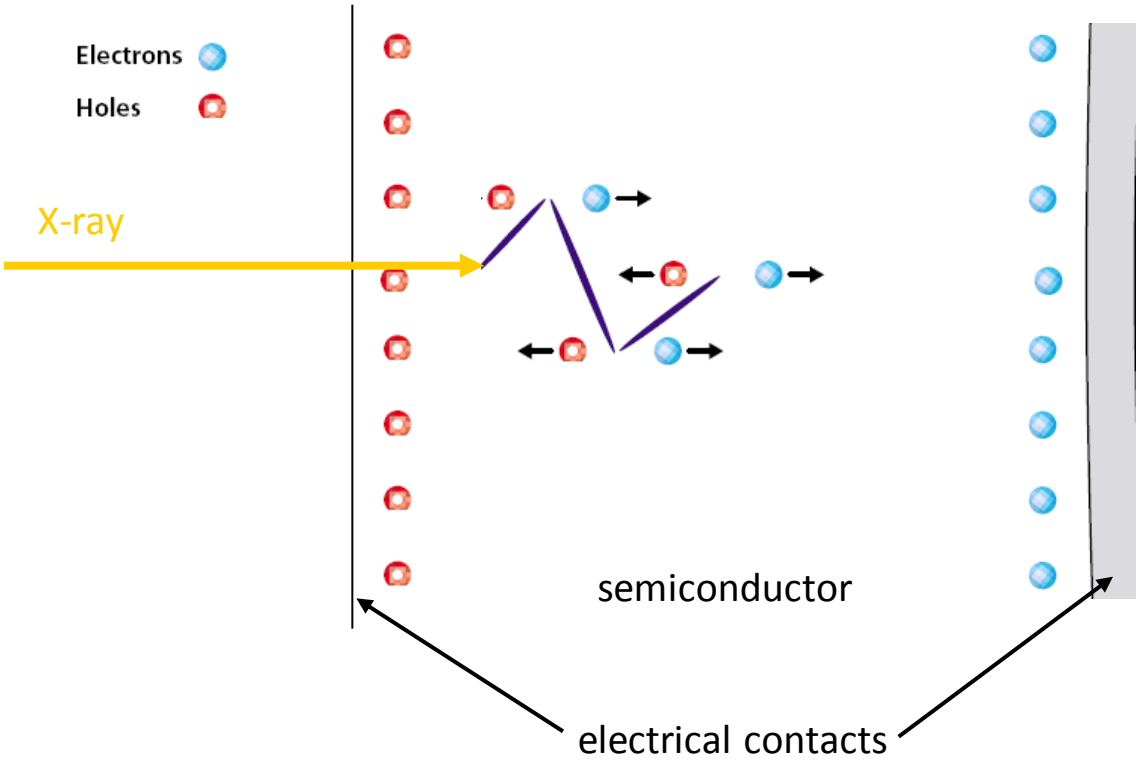


3D tomographic reconstruction shows distribution of *Fe*, *Cr*, *Se*.

~ 2 μm

ESRF-ID13', courtesy of Laszlo Vincze, Univ. Ghent

Semiconductor material e.g. high purity silicon with X-ray 'transparent' doped p and n contacts. Applied reverse-bias creates electric field depletes crystal bulk of (thermally generated) charges carriers.



-X-ray interacts (photoelectric absorption or Compton scatter), generates a 'hot' electron which rapidly thermalizes (~psec timescale) creating a cloud of charge .

- charges drift in the electric field towards electrodes over ~nsec to µsec timescale

- electrical signal is developed *while the charge drifts in the bulk… (Ramo, image charge…)*

For a *semiconductor* detector, the *statistical limit* to energy resolution is given by

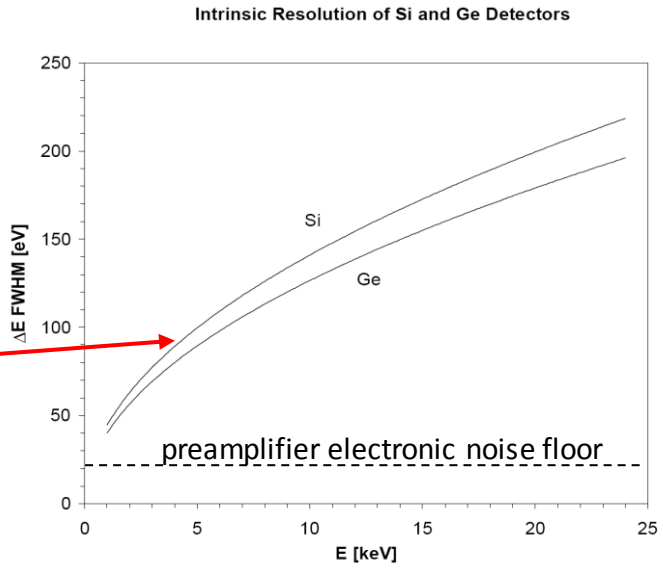
$\Delta E_{FWHM} = 2.35 \sqrt{F \varepsilon E}$ where ε =3.63 eV per electron-hole pair *for silicon*

Fano factor F ≈ 0.11 for Si and Ge (F is *not* a constant, but can usually be considered as one)

U. Fano, Phys. Rev. 72 (1947) 26

spectral resolution *measured* is the quadrature-sum of the Fano statistics and preamplifier electronic noise contributions:

$\Delta E_{total} = \sqrt{(\text{Fano})^2 + (\text{electronic noise})^2}$



Charge **q** created by X-ray absorption is

$$\mathbf{q} = 1.6 \times 10^{-19}\, E_{xray}(eV)/\mathcal{E} \qquad \text{(Coulombs)}$$

For a silicon detector with a *charge preamplifier* of feedback C_f = 0.1pF, an Xray of 10keV energy gives a voltage- step signal of just 0.4mV, but we need to measure this step with a precision of ~1% !

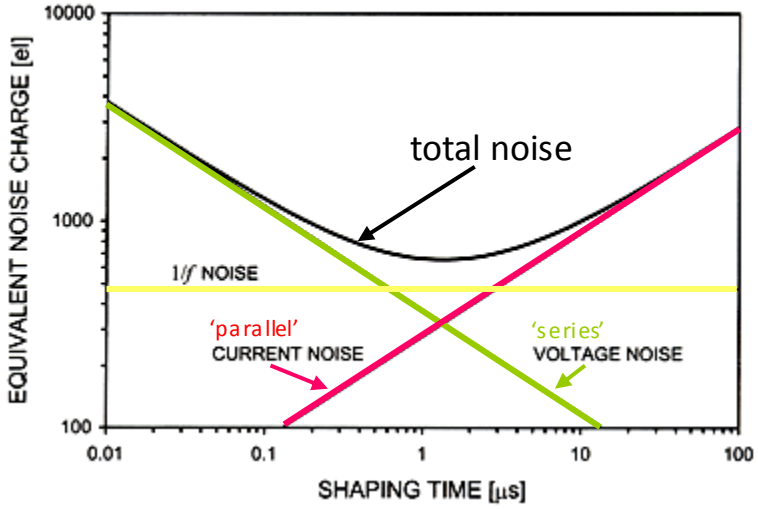
NOISE contribution of the detector preamplifier must be minimized:

this is done by reducing measurement bandwidth using an optimized weighting filter

For a charge preamplifier, the rms 'Equivalent Noise Charge' is

$$ENC \approx \sqrt{\left(\frac{kT}{2R_P} + \frac{eI_D}{4}\right)\tau + \left(\frac{kTC^2}{2g_m}\right)\frac{1}{\tau} + AC^2}$$

parallel noise series noise 1/f noise



where τ is the signal 'shaping time' (CR-RC filter)

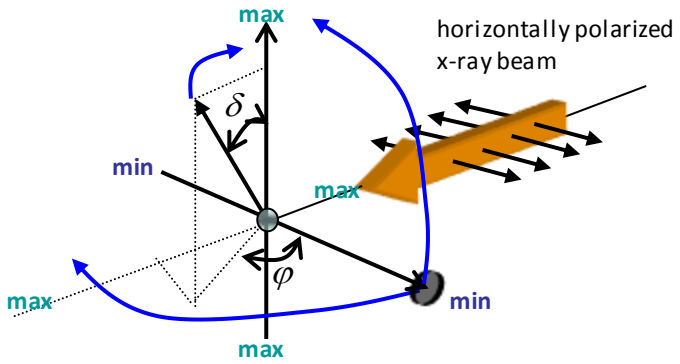
Synchrotron undulator beams focused on the sample are typically ~ 99% linear polarized

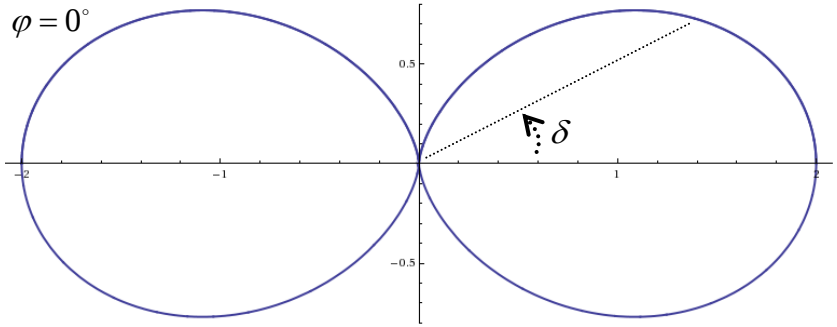
--> strong angular dependence of both Rayleigh (elastic) Compton (inelastic) scattering

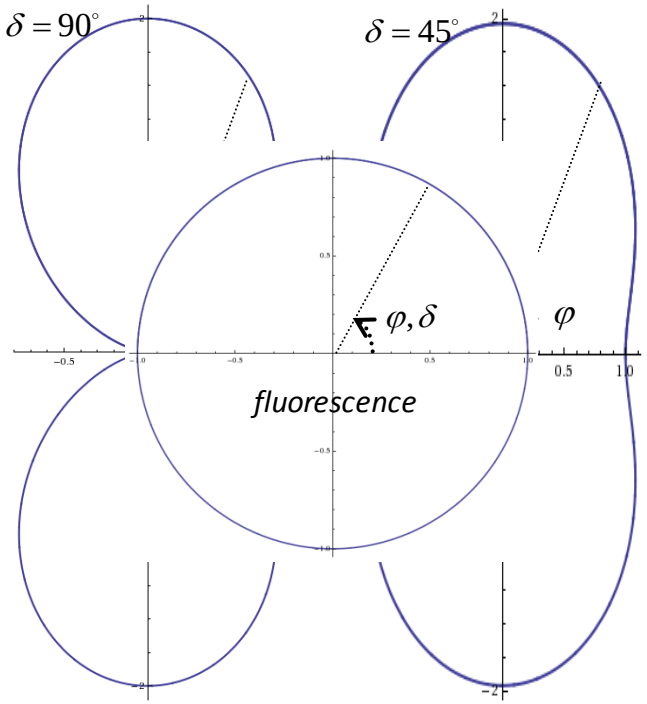
Polarization dependent elastic scattering cross section:

$$\left(\frac{d\sigma}{d\Omega}\right)_{\delta,\varphi} = \frac{r_e}{2}\left(1+\sin^2\delta\cdot\sin^2\varphi + P_0\left[\sin^2\delta\cdot\cos^2\varphi - \cos^2\delta\right]\right)$$

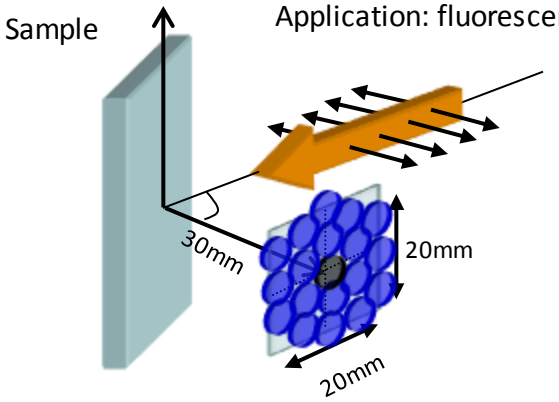
R. E. van Grieken, A. A. Markowicz, Handbook of X-ray Spectrometry (2002).

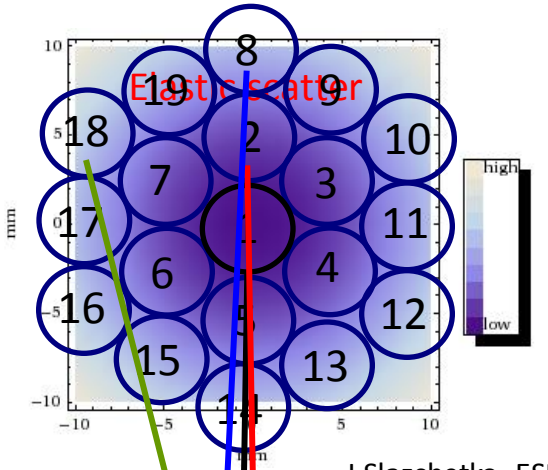






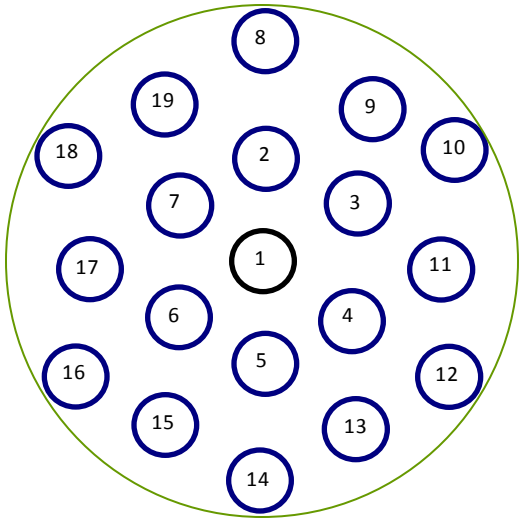
recall that cross section for fluorescence radiation is (almost) isotropic, i.e. independent of φ, δ

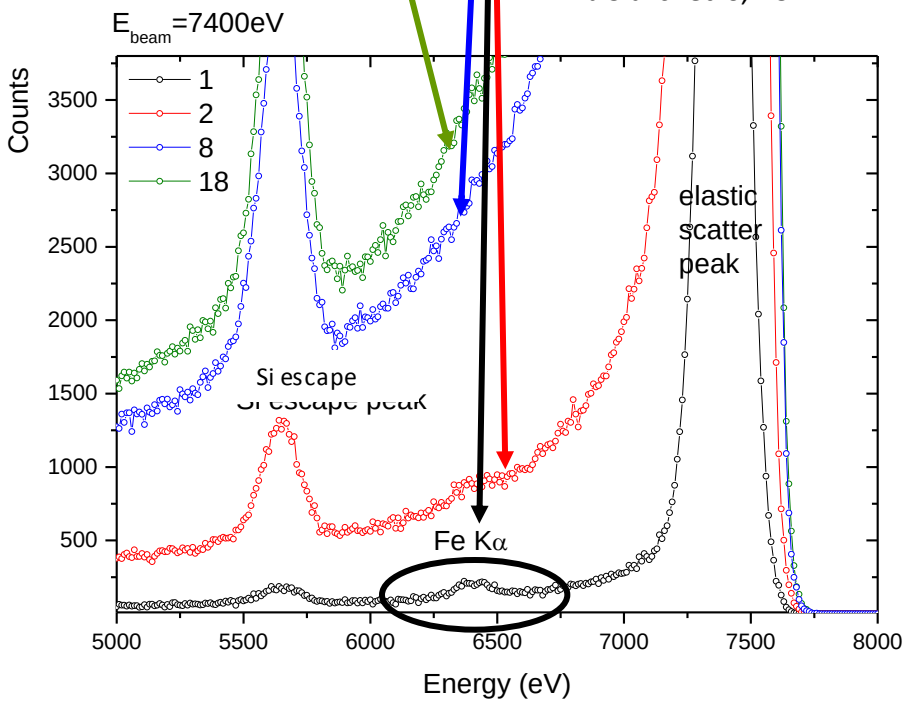




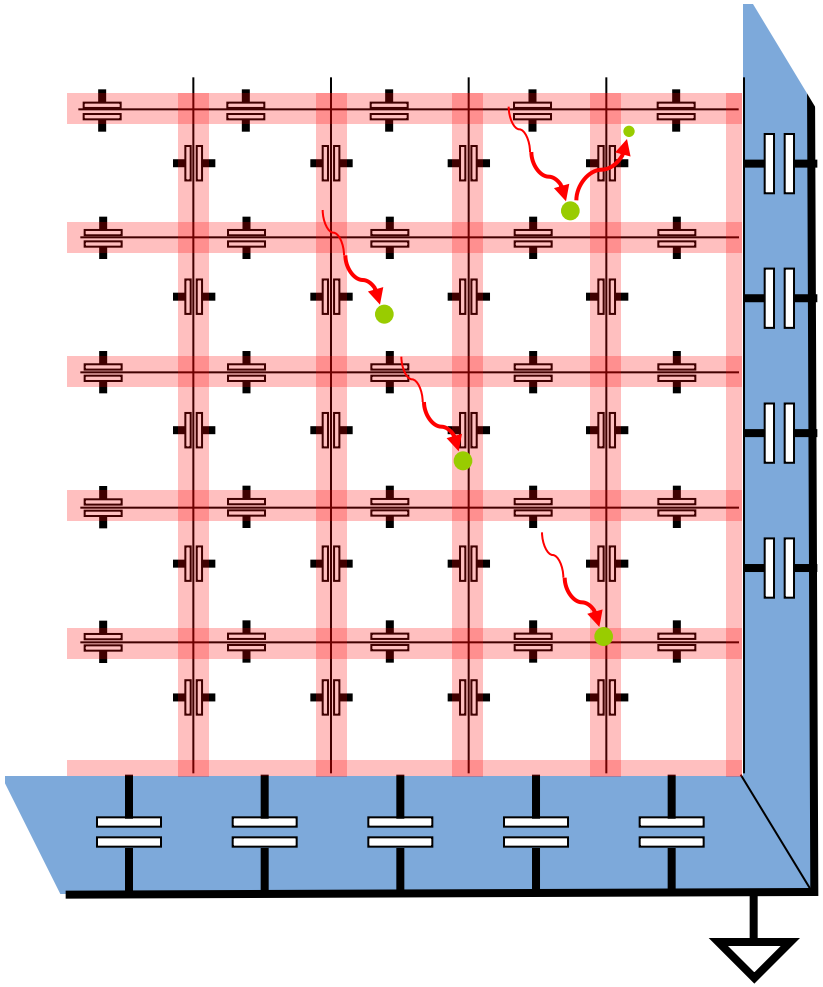
→ importance of 'packing factor' of the elements

typical case for Ge 'discrete element' detectors





using lithographic masked doping, a matrix of individual sensing areas can be made on semiconductor detectors. This can give a 100% sensitive area, *but there are challenges:*



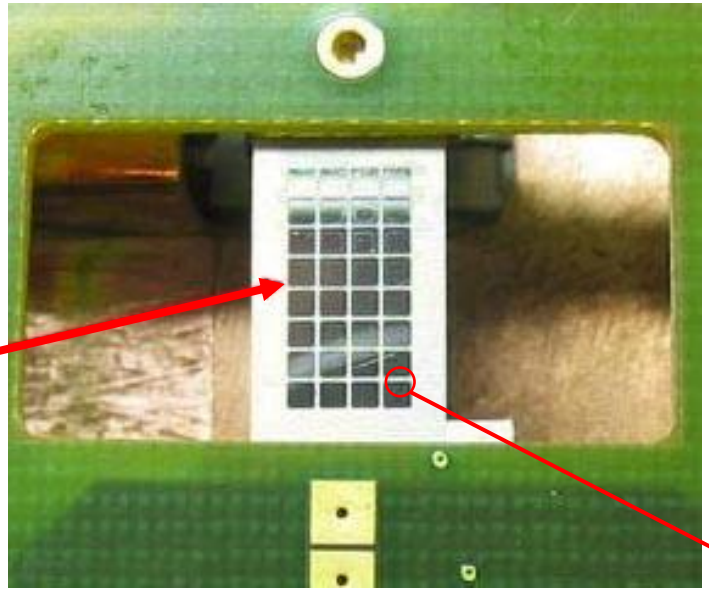
if an X-ray is absorbed near a pixel border, diffusion creates a 'cloud' of electric charge which splits the signal between neighbour pixels

Alternatively, a fluorescence photon may be emitted and absorbed in a neighbour pixel

These *physical crosstalk* effects become more serious as the individual 'pixel' area is reduced. Partial solution is use of a *grid collimator* which hides the pixel edges.

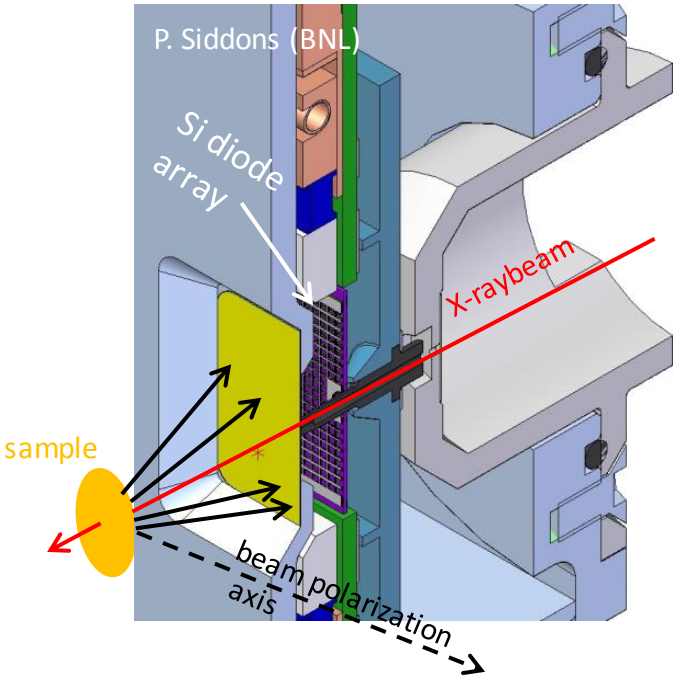
Each sensing area is capacitively coupled to its neighbours: this results in additional *electronic* crosstalk

Molybdenum mask on planar silicon detector developed at NSLS-BNL

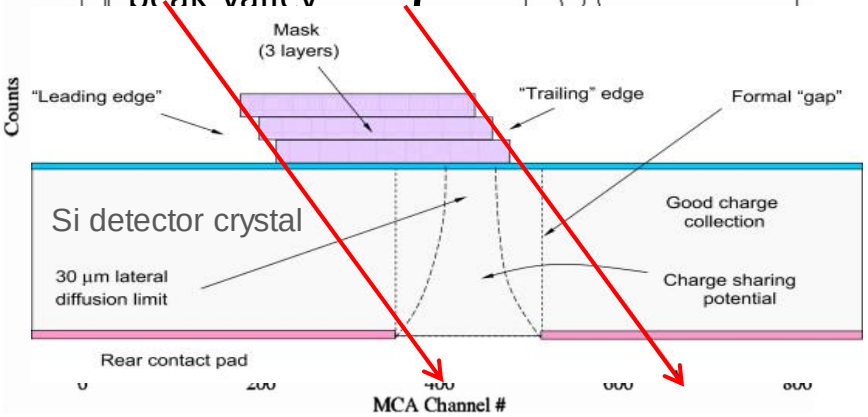


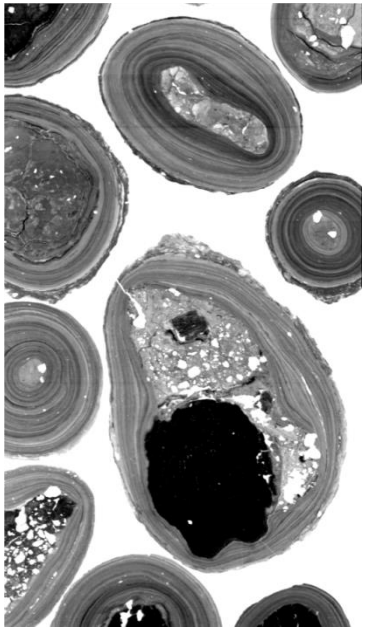
MAIA development: prototype with 32 element pin diode monolithic Si crystal

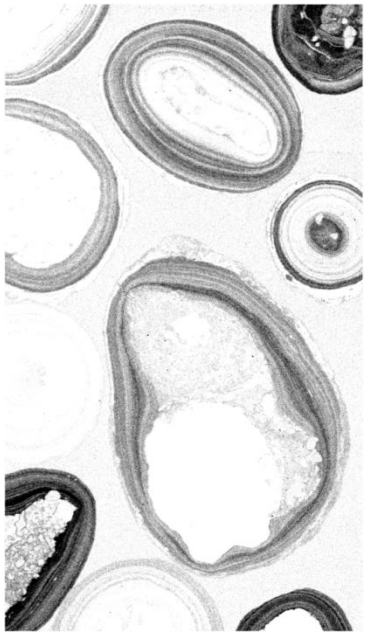
C.G. Ryan et al.
Nucl. Instr. and Meth. Phys. B 260 (2007) 1–7



staggered mask, alignment with diode edges
in final MAIA detector design (384x1mm² diodes)







first 96 element MAIA detector: concentrated iron (left) and trace yttrium (right) in iron-oxide nodules: grey scale is element abundance. Sample mapped with 5msec dwell time per 'pixel' position, total of 1625 x 2625 x7.5µm pixels, corresponding to 13 x 21 mm^2 scanned area. Beam energy 17.2 keV.

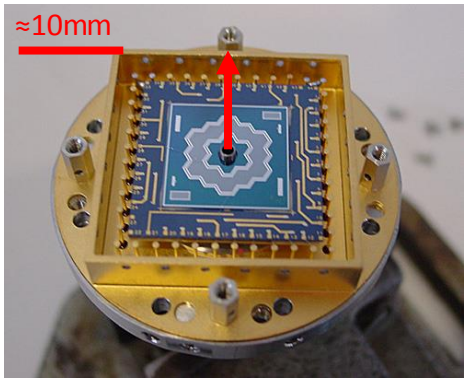
Ryan CSIRO, Siddons BNL et al, 1990

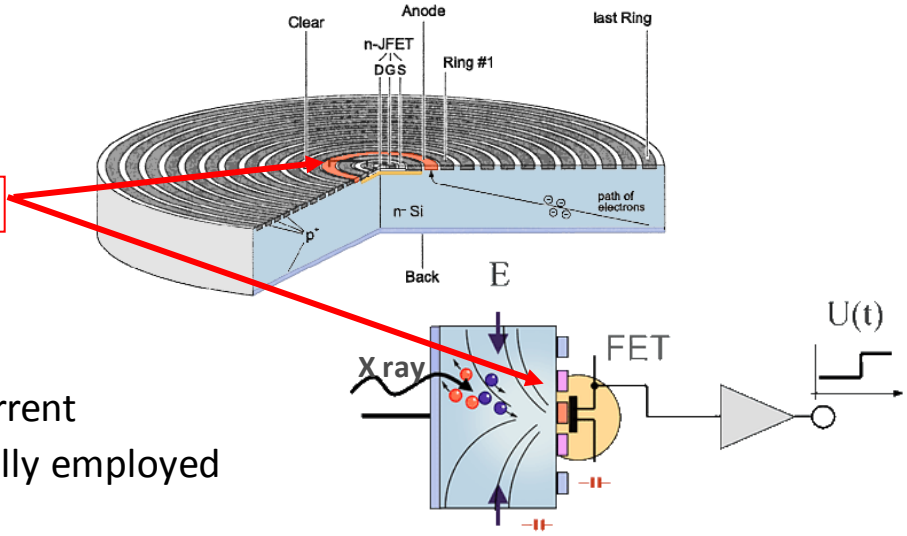
Zn, Ca trace metals in a mouse embryo cross-section image from latest 384 element MAIA detector at the Australian Synchrotron --event detection rates to 10 Mcps –> pixel dwell times ->50µs using *'on the fly' sample motion -data acquisition for 2D mapping* the sample; online FPGA based spectrum deconvolution.

CSIRO website May 2013

Planar silicon technology, multiple electrodes
establish transverse drift field ,
low capacity charge collecting anode / FET ~100fF

preamplifier may be (partly) integrated on detector

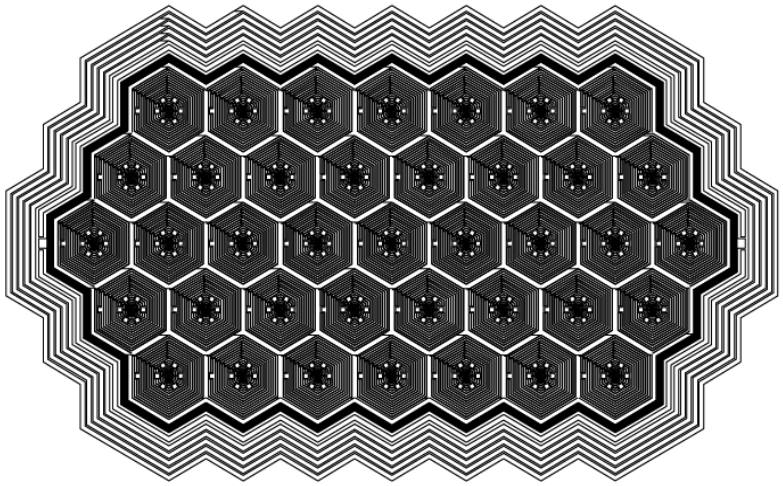




high resistivity silicon
⇨ low bulk leakage current
⇨ Peltier cooling usually employed
-10°… -50°C
⇨ *compact, lightweight systems,*
high rate capability ~1Mcps/channel, with 'Fano limited' energy resolution

Near wafer-scale lithographic processing
large, tightly-packed arrays possible
crosstalk issues

but large cell counts ⇨ yield issues
⇨ multi channel pulse processors
⇨ fabrication complexity / cost



39 cell detector with on-chip FETs, total active area 195mm^2 (after Struder, MPI-Garching)

Summary

We have presented some of the major detector types used at synchrotron sources and given a basic summary of their operation, but there are *many* other detector types in use, not described here for lack of time.

For many of these 2D (and '3D') detectors, fast data transfer with online visualization and analysis have become major electronic and software engineering challenges.

High performance, high efficiency detectors are as important as the beamline and its X-ray optics:

the X-ray beam intensitiy can be so high that for many experiments we are limited by the *radiation tolerance limit of the sample…* so we need to make sure that every photon 'counts'.

Semiconductor materials for γ and X-ray detection

Fano energy resolution, leakage current (noise)

$$\frac{\Delta E}{E} \propto \frac{1}{\sqrt{N}}$$

Signal development time (max. counting rate)

stopping power, X-ray absorption length

Material	Z	Bandgap [eV]	Mobility [cm²/Vs] electrons	Mobility [cm²/Vs] holes	Density g/cm³
Si	14	1.1	1350	480	2.3
Ge	32	0.7	3800	1800	5.3
Diamond	6	5.5	4500	3500	3.5
GaAs	31-33	1.5	8600	400	5.4
AlSb	13-51	1.6	200	700	4.3
GaSe	31-34	2.0	60	250	4.6
CdSe	48-34	1.7	50	50	
CdS	48-16	2.4	300	15	4.8
InP	49-15	1.4	4800	150	
ZnTe	30-52	2.3	350	110	
WSe_2	74-34	1.4	100	80	
BiI_3	83-53	1.7	680	20	
Bi_2S_3	83-16	1.3	1100	200	6.7
Cs_3Sb	55-51	1.6	500	10	
PbI_2	82-53	2.6	8	2	6.2
HgI_2	89-53	2.1	100	4	6.3
CdTe	48-52	1.5	1100	100	6.1
CdZnTe	48-30-52	1.5-2.4			

monoelemental crystals, excellent charge transport

Binary and ternary compounds

Stochiometry etc

→ *trapping of charge during drift*

μτ products, schubweg

τ_e, τ_h carrier lifetimes

Materials already investigated as radiation detectors