Energy resolving semiconductor detectors for X-ray spectroscopy

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Detector Unit - ISDD

the increasingly important topic of *wavelength dispersive spectroscopy* 'detectors' will not be discussed here

what I will talk about…

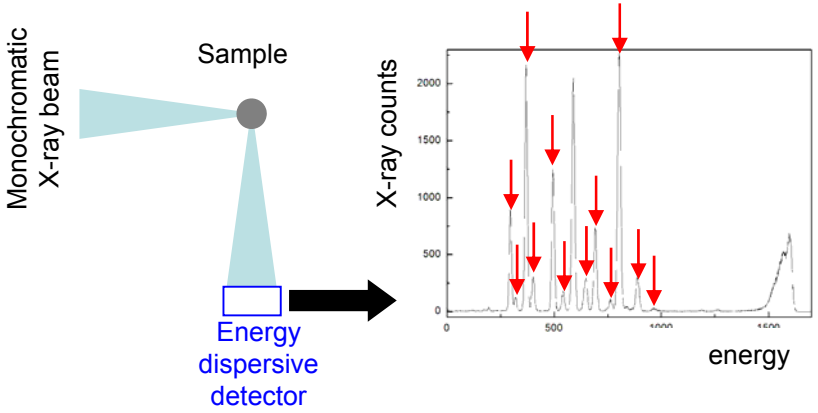
- what are the synchrotron requirements?
- *semiconductor* Energy Dispersive X-ray detectors: principle of operation, material limitations
- energy resolution and Fano statistics
- preamplifier and electronic noise
- signal pulse processing and the pile-up limit
- silicon drift diodes
- multielement arrays and the ‘crosstalk’ challenge
- summary

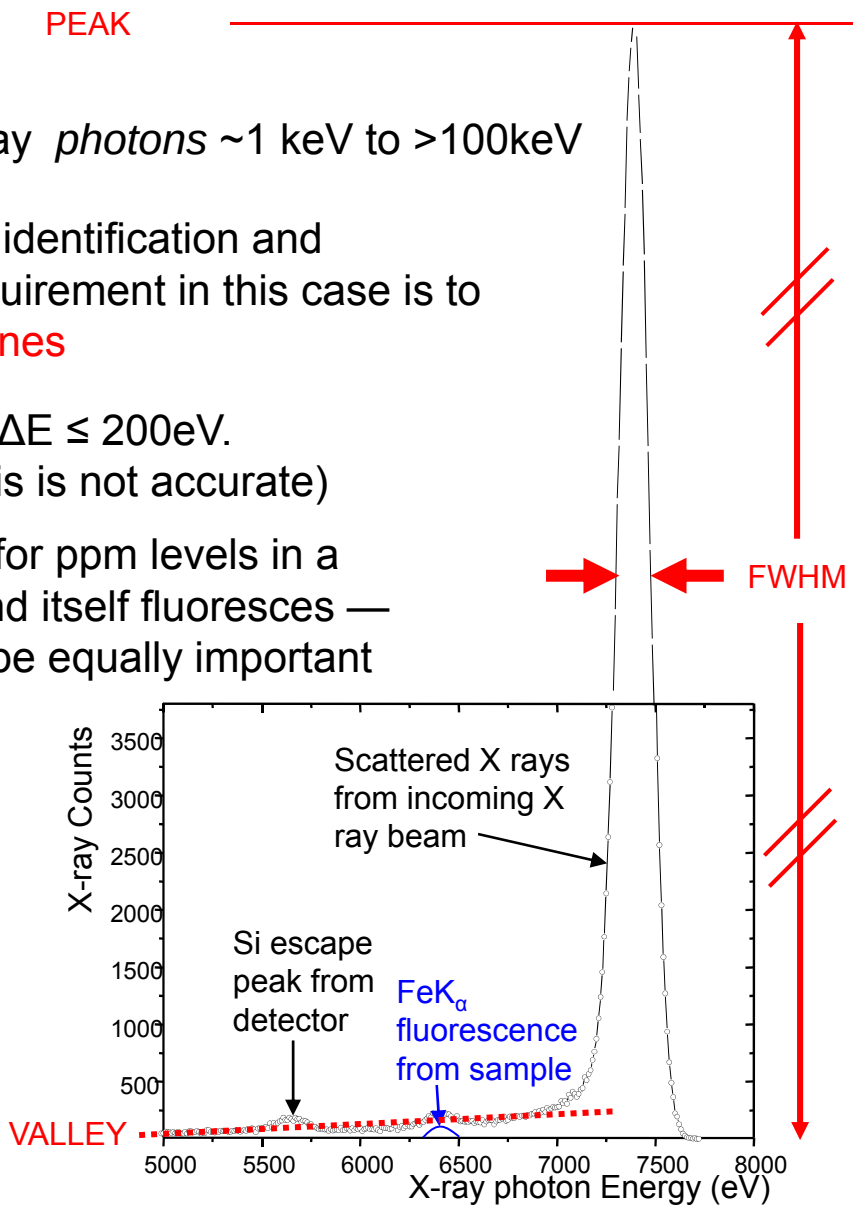
Energy range: '3rd Generation' Synchrotrons, X-ray *photons* ~1 keV to >100keV

Energy *resolution:* many measurements concern identification and quantification of *multiple* elements in sample. Requirement in this case is to resolve-identify individual K, L, (M) fluorescence lines

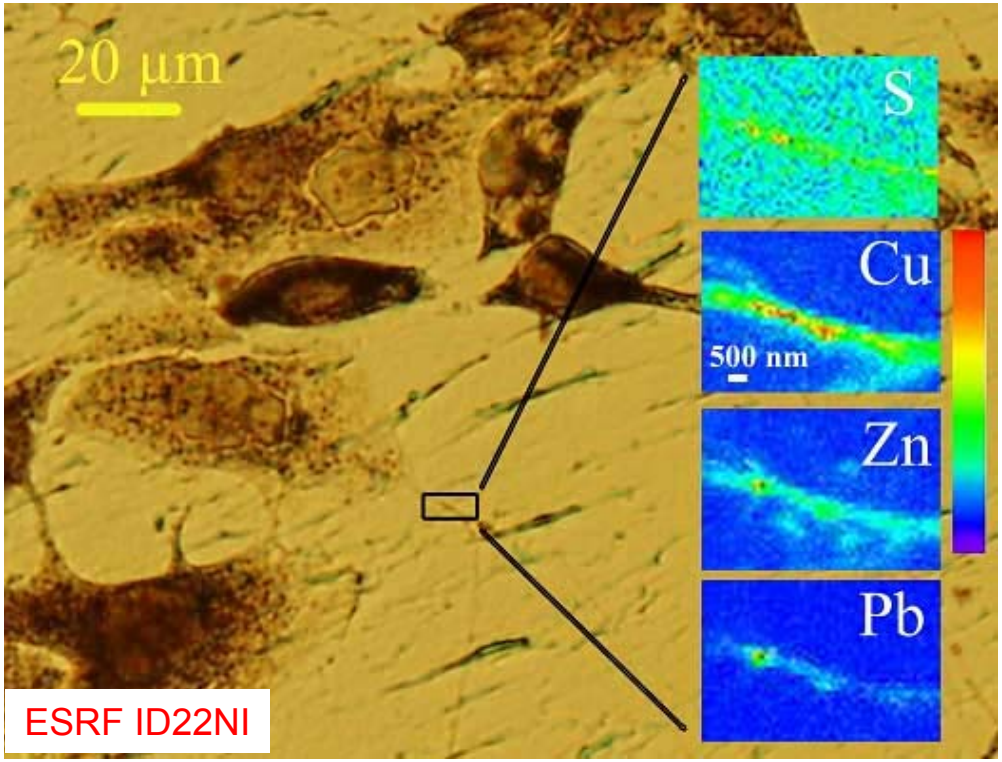
'*FWHM*' is the usual figure of merit, typically need ΔE ≤ 200eV.
A Gaussian line shape is usually assumed (but this is not accurate)

For trace element analysis -- where we may look for ppm levels in a sample matrix that scatters the incoming beam and itself fluoresces — *'peak-to-valley'* performance of the detector may be equally important





Counting rates

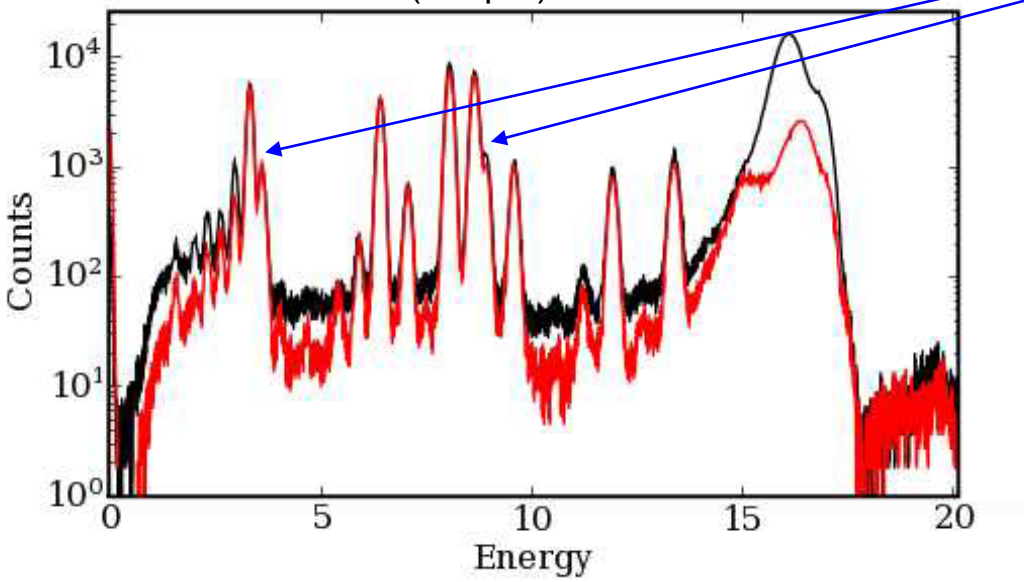


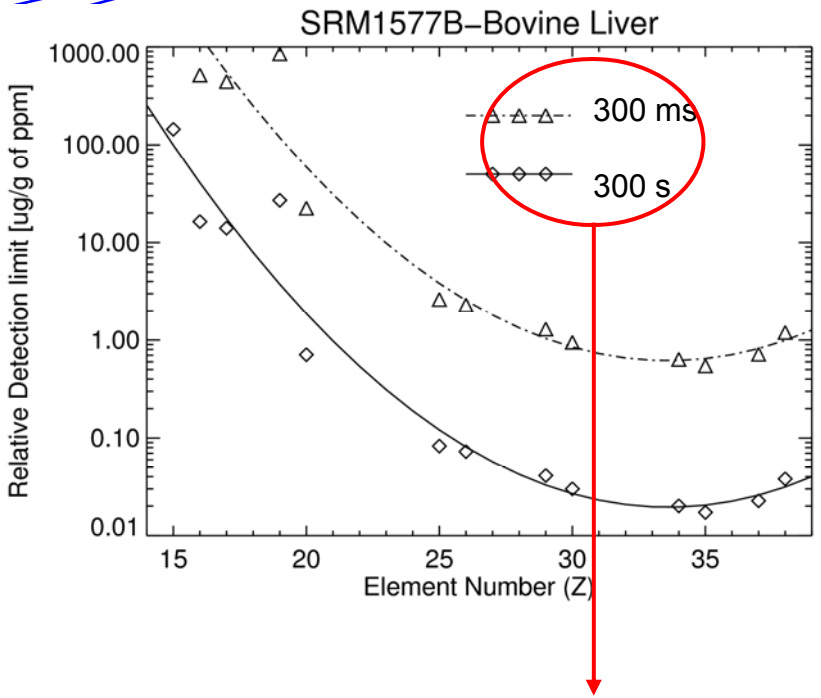
Neurite process
A Carmona et al JAAS (2008)

For analysis of *chemical states* (e.g. SO_4^{n-}… XANESstudies), higher energy resolution may be required.
In this case, the incoming synchrotron beam energy crystal monochromator is energy scanned with ΔE ~1eV to determine spectral response of sample
but an energy resolving detector is still required for dilute samples

For *quantitative element analyis*, Silicon and Germanium semiconductor detectors are used:
- fast photon event counting over all energies in spectrum
- good efficiency possible (solid angle covered by detector)
-adequate FWHM resolutions of known lineshape (needed for *spectrum deconvolution*)

Bovine liver 'thick'(200µm) standard



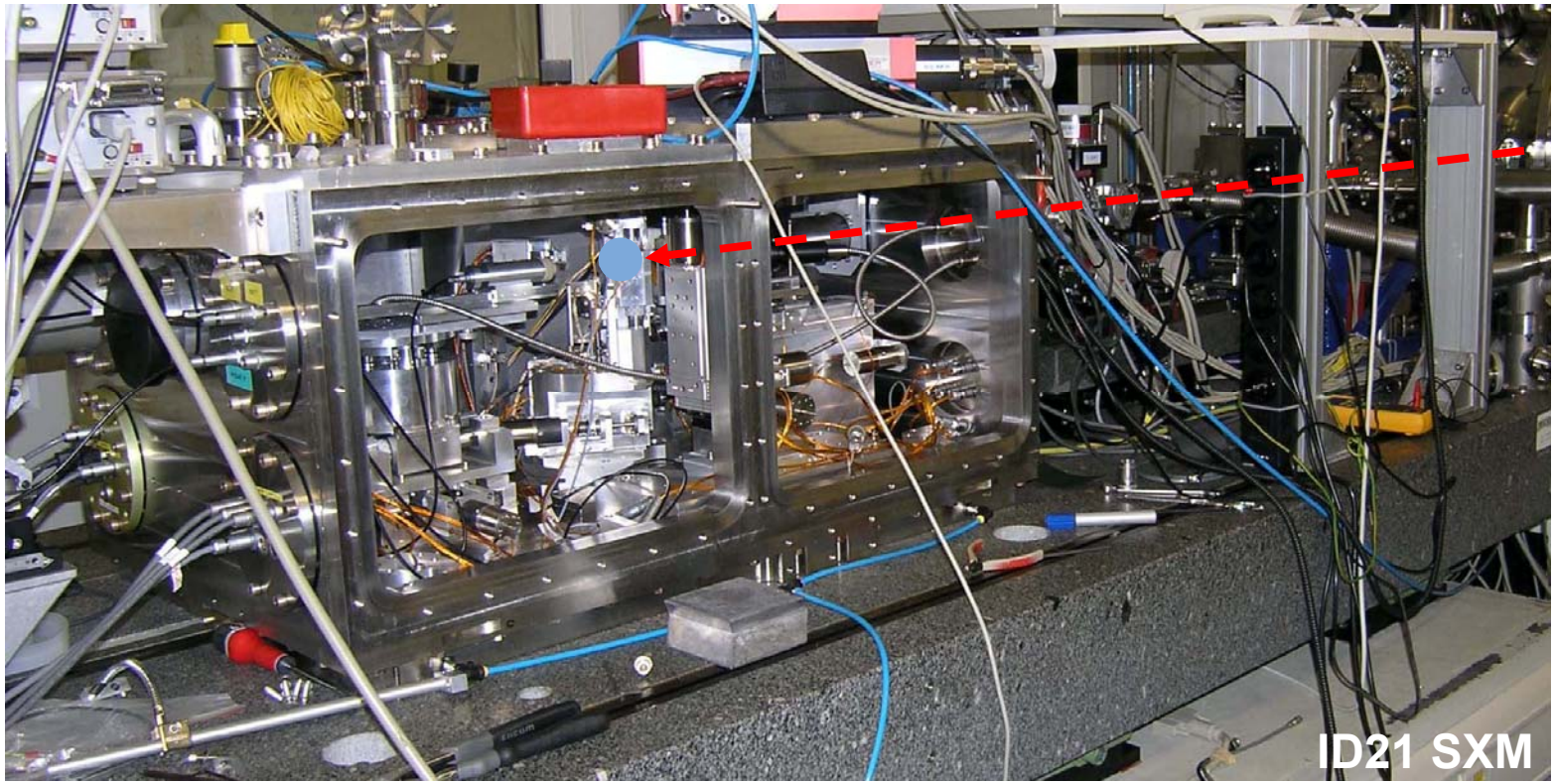


beam normal incidence on sample,
Vortex silicon drift detector detector at 75º
beam 45 deg incidence, detector at 90º

P Cloetens, ESRF-ID22N

detection limits are set by counting statistics

Synchrotrons X-ray beams are *focused* onto sample

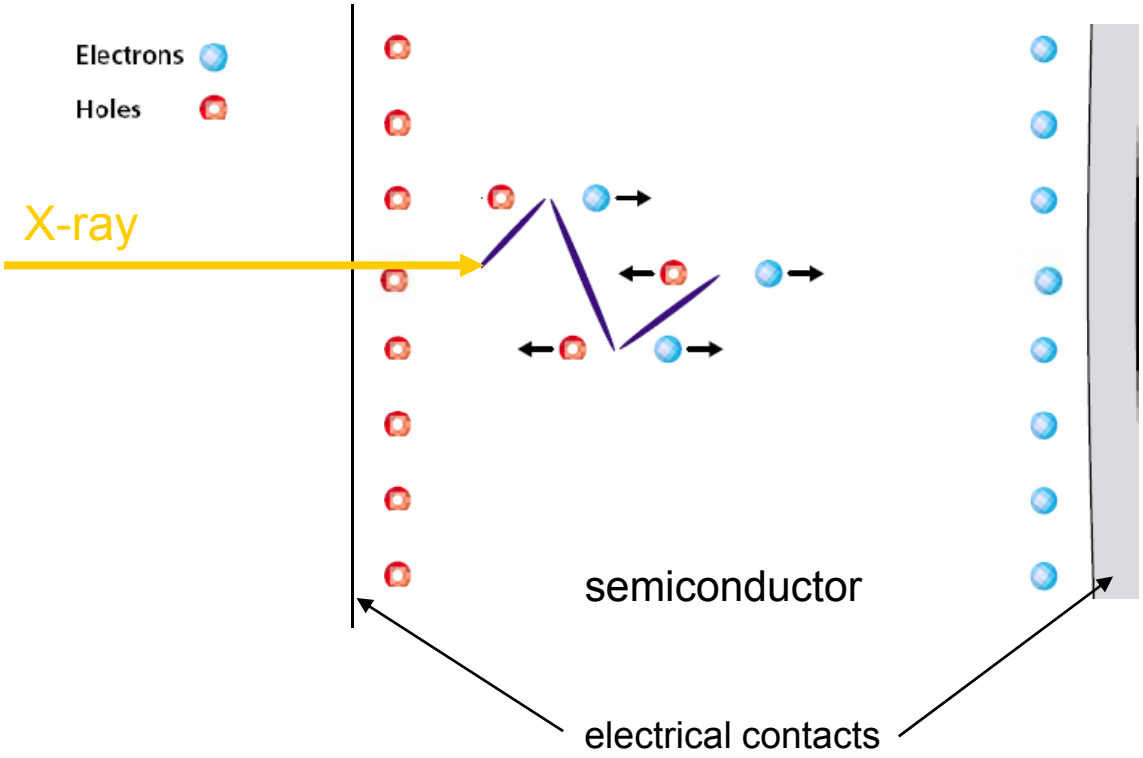


other practical challenges for optimum detector operation:

-vibrations – accoustics

-electrical interference from other equipment… *Electro- Magnetic Compatibility* (EMC)

Semiconductor material, e.g. crystal of Si or Ge, with thin X-ray transparent contacts.
An applied electric field can deplete bulk of (thermally generated) free charges.



- X-ray interacts (photoelectric effect or Compton scatter), generates 'hot' electrons which rapidly thermalize (in ~psec timescale),
- hole, electron charges drift in applied field towards electrodes (~nsec to µsec)
- electrical signal develops while the charge drifts in the bulk…

"*The crystal counter: a new instrument in nuclear physics*",
P.J. Van Heerden, PhD Dissertation, Rijksuniversiteit Utrecht *July 1945*

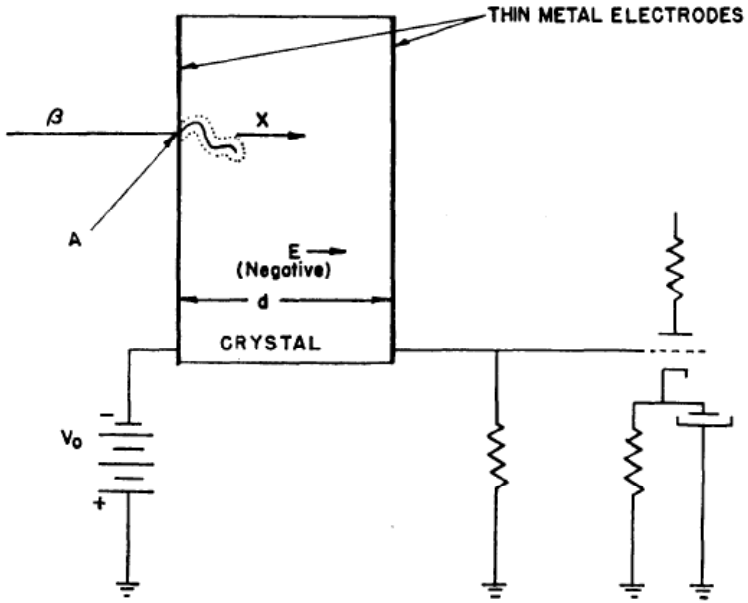
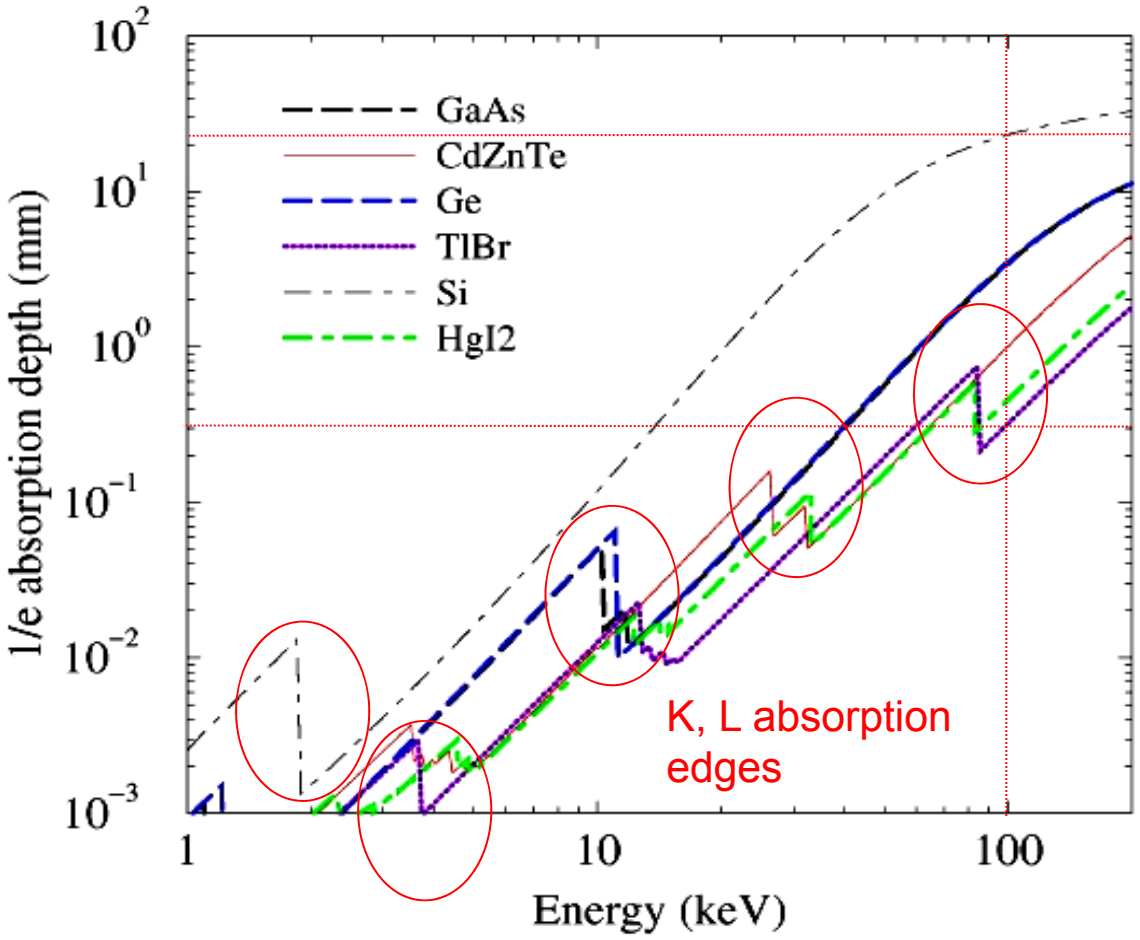


Fig. 1—A schematic diagram of a crystal conduction counter.

but in practice needed development of

- materials in which photoelectric charge is not 'lost in transit', i.e. by trapping at crystal structure defects or impurity sites (→ Ge(Li), Si(Li)… high purity Ge, Si crystals)
- development of (surface) electrical contact technologies
  (problems of time dependent 'polarization' effects; charge injection-leakage current…)

X-Ray absorption in various detector materials



Beer's law:

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

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

At 'low' energies, photoelectric effect is dominant:

$\mu(E) \sim E^{3\ldots4}$

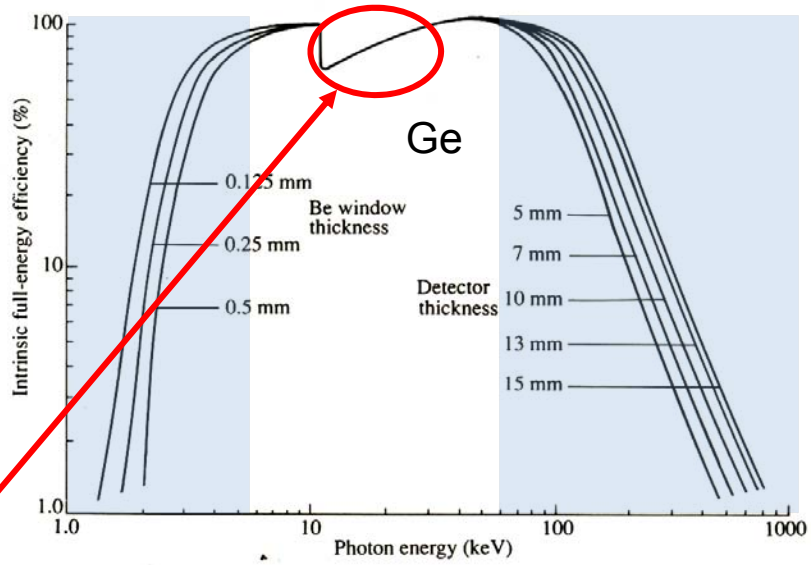
but μ is discontinuous at 'absorption edges' corresponding to atomic shell structure binding energies

40μm of Ge (or GaAs) has same total X-ray absorption as ~500μm Si

Material absorption effects on energy spectrum

Useful detector energy range is set by photon absorption range in material (s)

- -'window' transmission cut-off (need for a detector vacuum window)
- - inefficient charge collection for absorption at front contact of the semiconductor crystal
- - transmission loss at higher energies
- - incomplete energy absorption (loss by *Compton Scattering*)

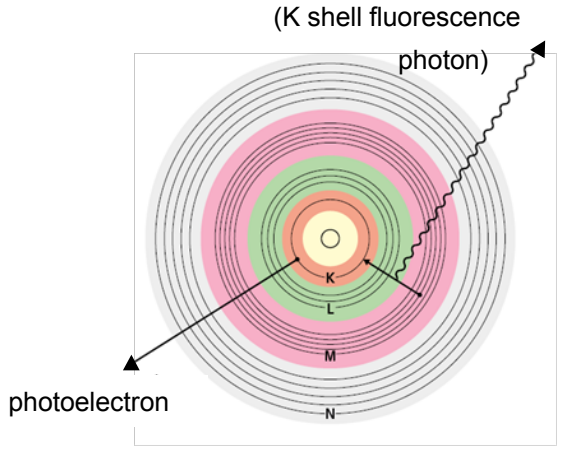


Abrupt absorption efficiency loss occurs at *binding energies* of electrons corresponding to shell levels. This is associated with probability of *fluorescence emission*

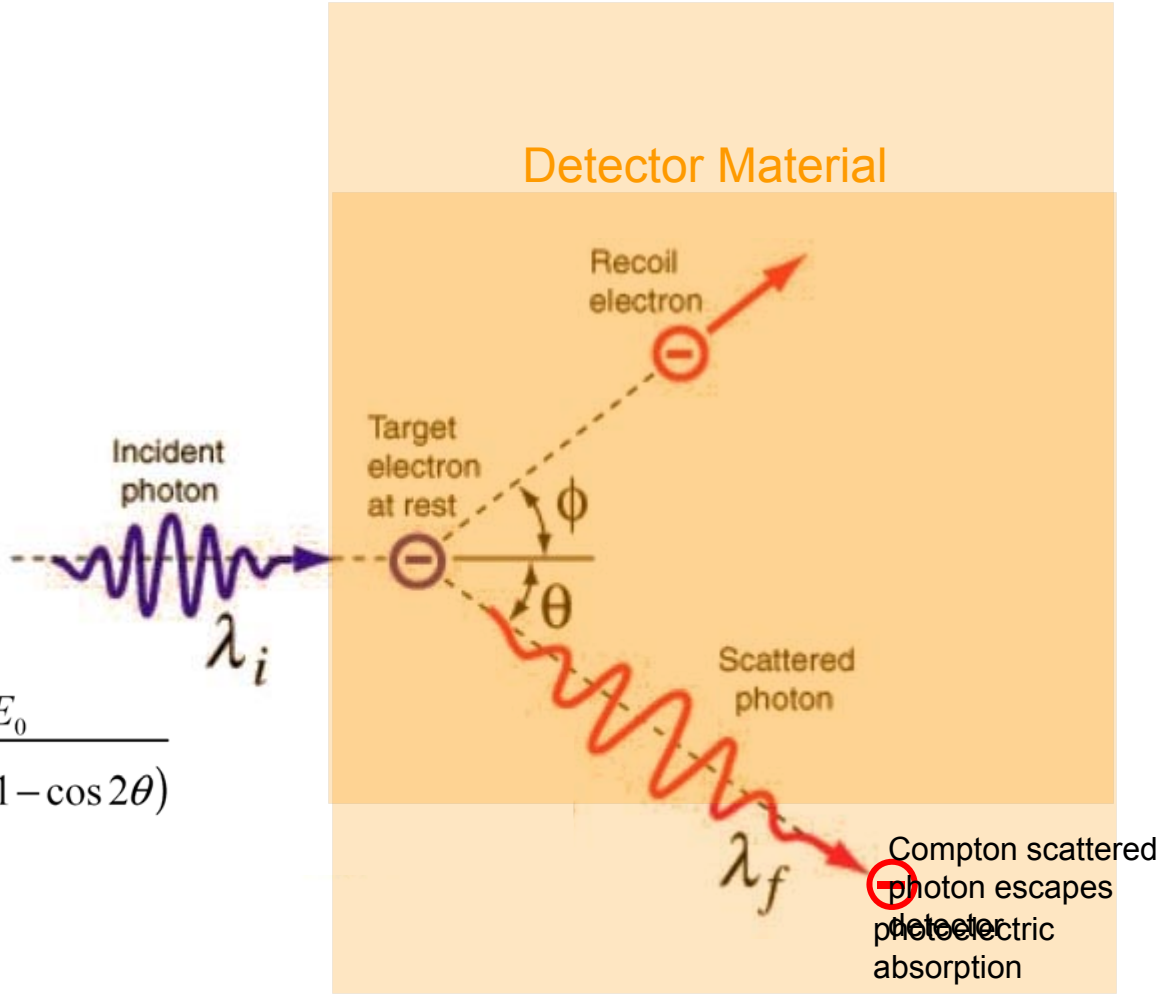
'Escape' peaks appear in detector energy spectrum at energies (E_{Xray}- E_{fluo}), where E_{fluo} transition energy for electron falling from L, M… levels to inner K shell energy level

e.g. for Ge Efluo ≈ 9.9 keV (Kα), 1.2 (Lα1)
for Si ≈ 1.74 (Kα)

Escapes complicate spectra with multiple peaks, and information may be 'lost' by peak overlaps



Compton Scattering and energy loss



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

$$E = hc/\lambda$$

~~all~~ measured energy = Compton recoil electron only (Compton photon)

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			

Materials already investigated as radiation detectors

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

Absorbed radiation energy E is shared between crystal lattice excitations (~2/3) and generation of charge carriers (~1/3)

this ratio is almost constant for semiconductor materials

Lower bandgap materials can offer better resolution due to better *Fano statistics*

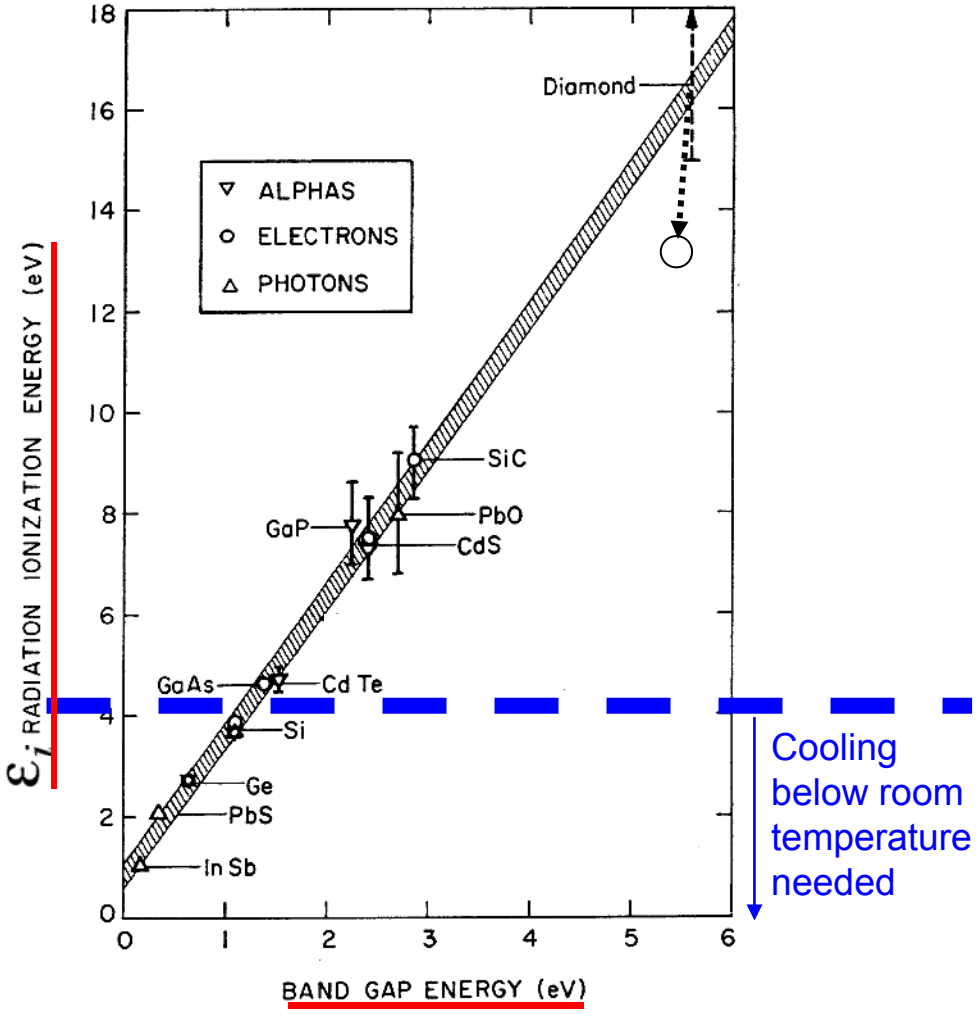
$$\Delta E = 2.35 \cdot \varepsilon_i \sqrt{F N_Q}$$

N_Q is number of generated charge carriers,

F defined as 'Fano factor'

$$= 2.35 \cdot \varepsilon_i \sqrt{F \frac{E}{\varepsilon_i}} \qquad = 2.35 \cdot \sqrt{F E \varepsilon_i}$$

But low bandgap materials must be cooled to limit noise from thermal generation of carriers ~exp(ε_i /kT)
and often suffer from 'charge trapping'



C.A. Klein, J. Applied Physics **39** (1968) 2029

Recall, 'physics-statistics' energy resolution limit ΔE is set by Fano statistics:

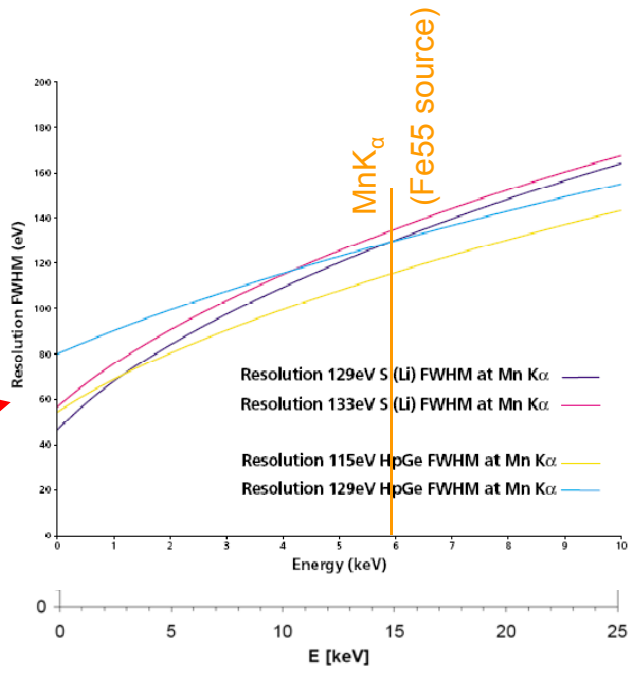
$$\mathbf{FWHM = 2.35\ \sqrt{F\varepsilon E}} \qquad \boldsymbol{\varepsilon} = 3.63 \text{ eV/e-h for Si}$$

Fano factor **F** ≈ 0.11 for Si and Ge (**F** is *not* a constant)

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

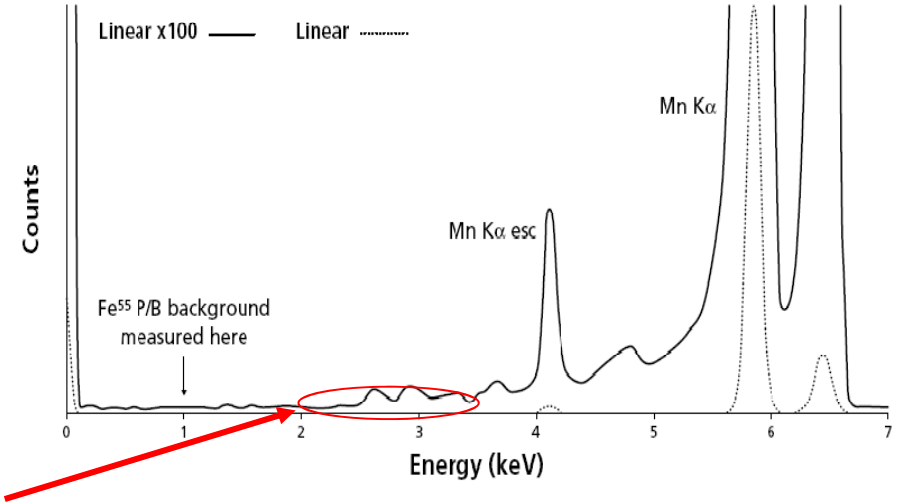
But *measured* spectral resolution **R** is quadrature-sum of above Fano statistics and electronic noise :

$$\mathbf{R = \sqrt{(Fano)^2 + (electronic\ noise)^2}}$$



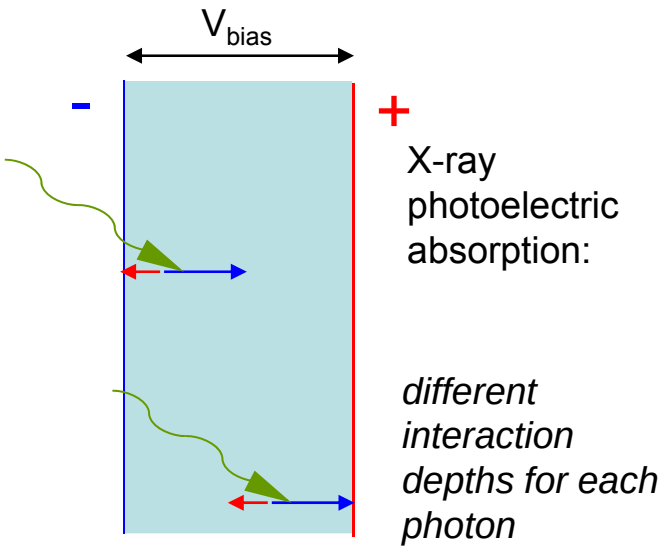
R should have ~Gaussian symmetric shape, but rarely does at ≤ 1% level… multiple causes:

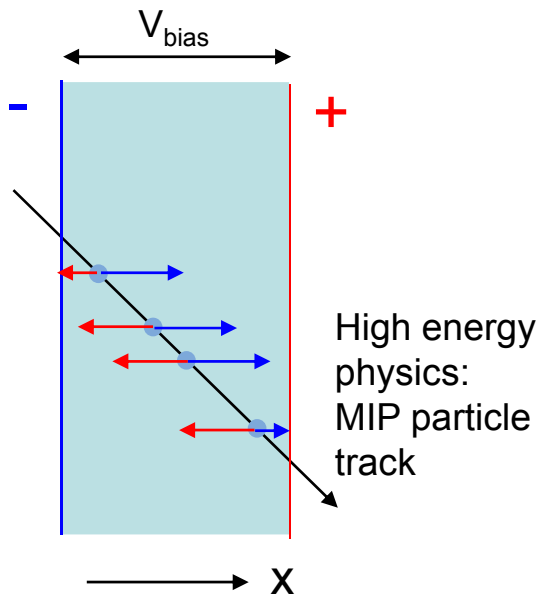
- near surface X-ray absorptions with incomplete charge collection
- 'ballistic deficit' associated with charge collection and pulse filtering time
- 'external' noise sources
- pulse processor effects (pile-up and baseline *degradation at high count rates*)



Peak-valley performance may be critical

Signal: time development

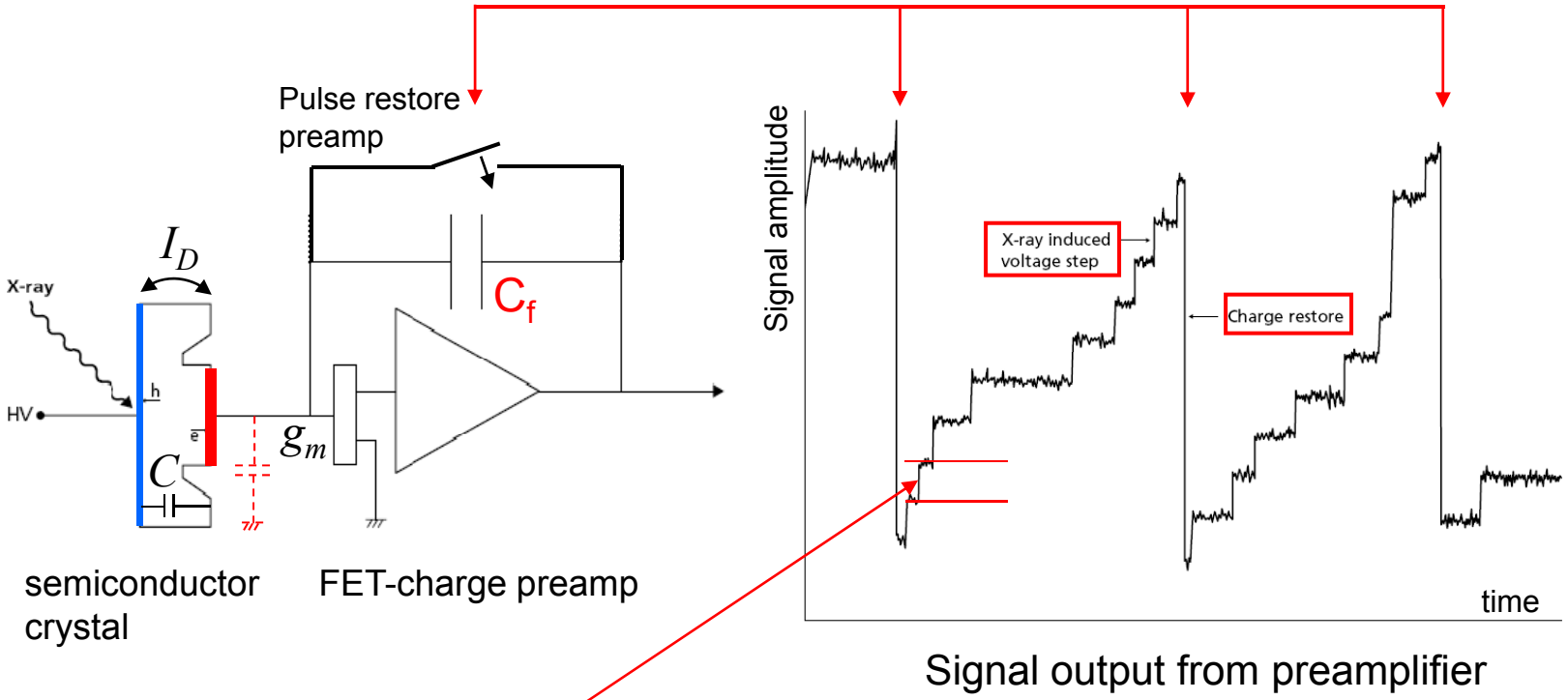




for photons → variation in signal-time development according to photon interaction point

In spectroscopy measurements, problem is avoided by use of *charge sensitive preamplifier* which integrates the *i(t)* signal current

assuming no charge trapping!



charge preamp, *signal* out (volts) = $\frac{\text{charge in (from X-ray photo-conversion)}}{\text{preamp feedback capacitance } C_f}$

Charge preamplifier →

Signal amplitude is proportional to collected photoelectric charge (i.e. to X-ray energy)
…and independent of detector bias, interaction point (charge drift time variations)

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

$$\mathbf{q} = 1.6 \times 10^{-19} E_{xray}(eV)/\varepsilon_i \quad \text{(Coulombs)}$$

ε_i = 3.63eV / electron-hole pair for Si , 2.9eV / electron-hole pair for Ge

→ for germanium detector preamp' with typical feedback C_f=0.1pF, a 10keV Xray gives a voltage step signal of only 0.5mV and we need to measure this with a precision of <1% !

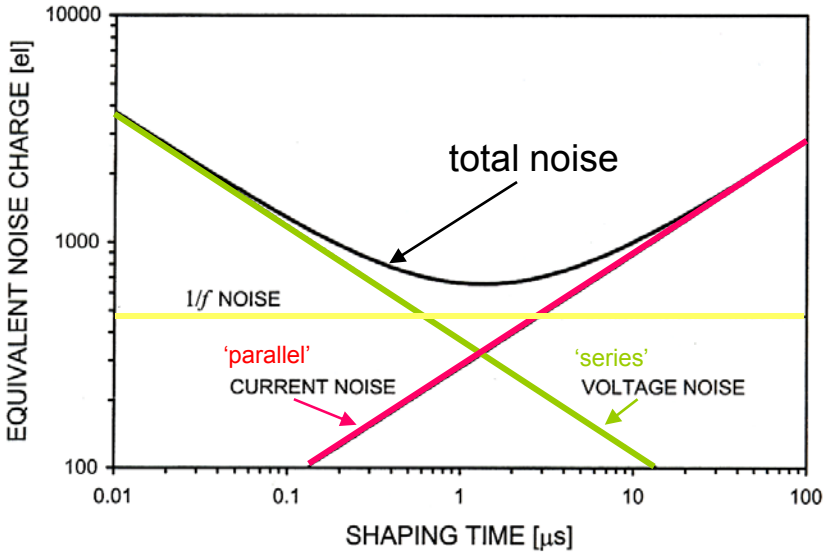
NOISE contribution of electronics (preamplifier) must be minimized

For charge preamplifier, 'Equivalent Noise Charge' (expressed in e^- r.m.s) analysis gives

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

parallel noise series noise 1/f noise

τ is signal 'shaping time'

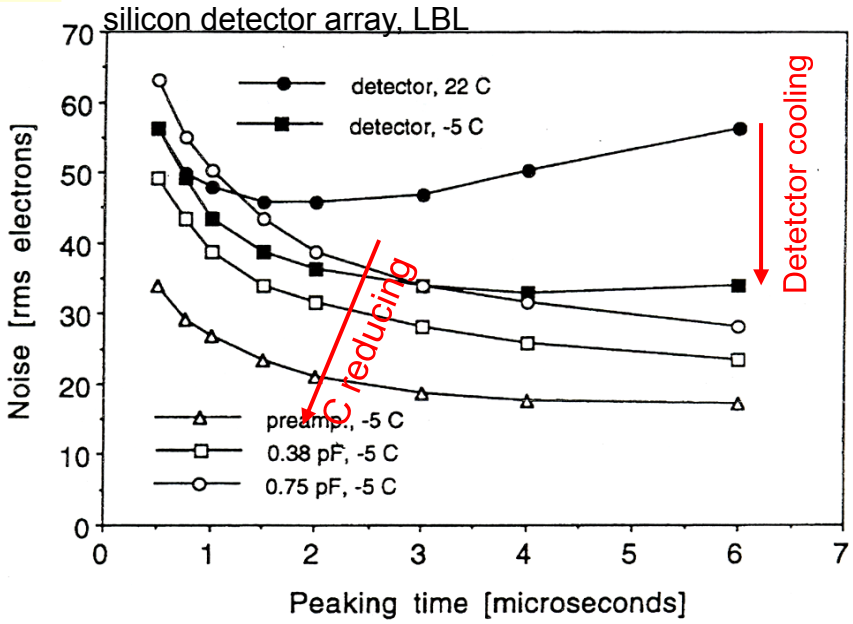


To reduce noise: $ENC \approx \sqrt{\left(\frac{kT}{2R_P}+\frac{eI_D}{4}\right)\tau+\left(\frac{kTC^2}{2g_m}\right)\frac{1}{\tau}+AC^2}$

-maximize R_P
OK, $R_P \rightarrow \infty$ by using a 'pulse restore'

-minimize detector 'leakage current' I_D
reduce temperature, detector material bulk *and surface,* design tricks

-minimize kT
(OK, cool detector, but limits to this…)



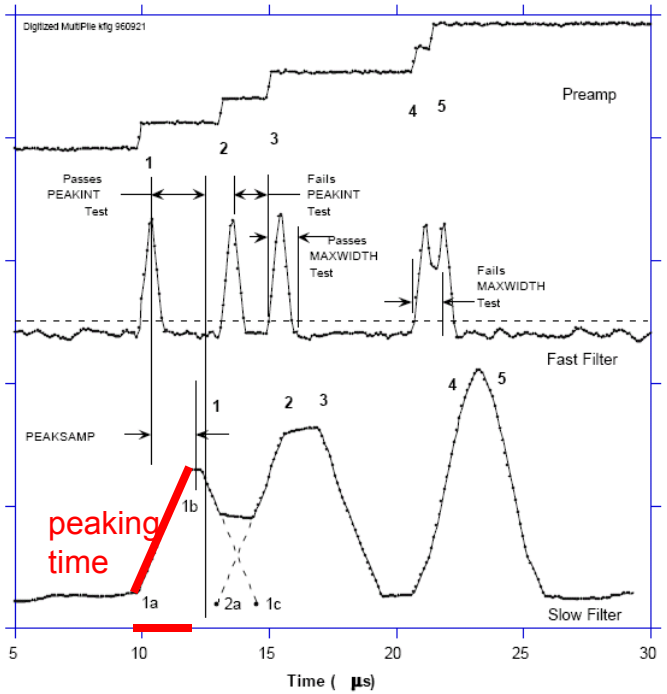
-minimize C
(crystal geometry, 'drift diode' designs, FET type/integration)

-*optimize choice of* τ
pulse shaping (or peaking) time can be varied 'online' as needed by experiment…

…but need to count at high rates (≥ 1/10τ) limits the maximum τ *values*, i.e. problems of *pulse pile-up*…

Pulse processor (for spectroscopy, now almost always 'digital' systems) has several tasks:

- ⇨ minimize preamp noise contribution to resolution (filter peaking time and shape)
- ⇨ detect and reject *pulse pile-up* events (and detector preamp pulse restores…)
- ⇨ and record corresponding detector 'dead time':

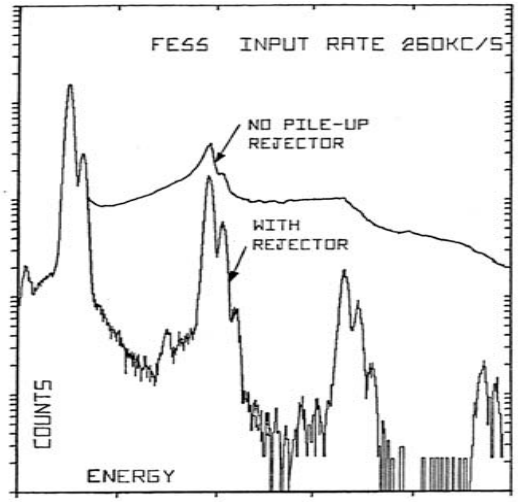


Fast channel ('time info')

'slow' channel (energy)

PUR

spectrum



Spectroscopy pulse processors are 'paralysable':

-'dead-time' T_P for processing each event

- any second event occurring within time T_P is rejected to avoid false 'pileup' peak in spectrum

For Poisson time-distributed X-ray events, measured spectrum output count rate can be obtained from

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

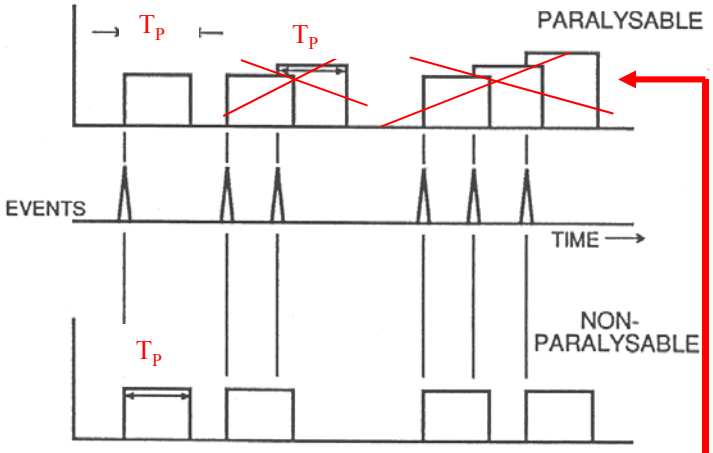
T_P is 'dead time per event'

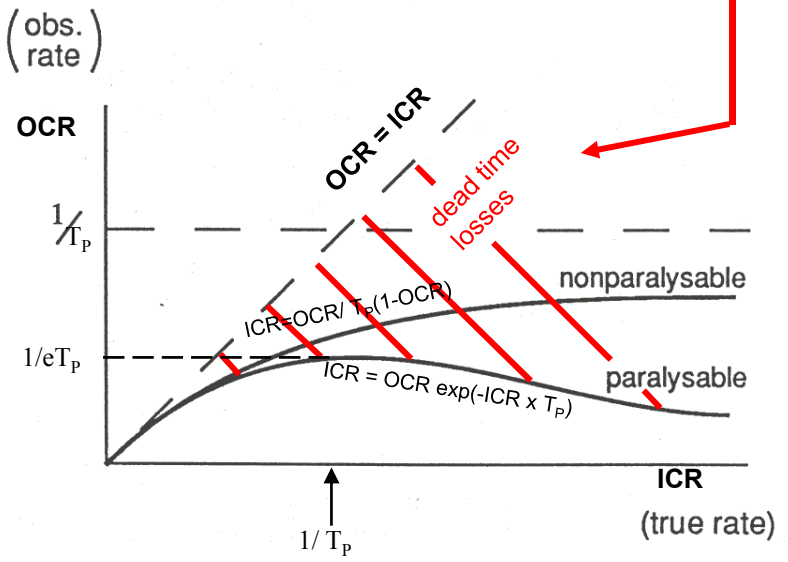
≈ 5x pulse 'shaping time' or ≈ 2x 'peaking time'

Limits to OCR usually:

Low counting rates:
insufficient detector size (solid angle)

High counting rates:
T_P cannot be reduced (energy resolution degrades!)





⟹ ***multielement detector systems***

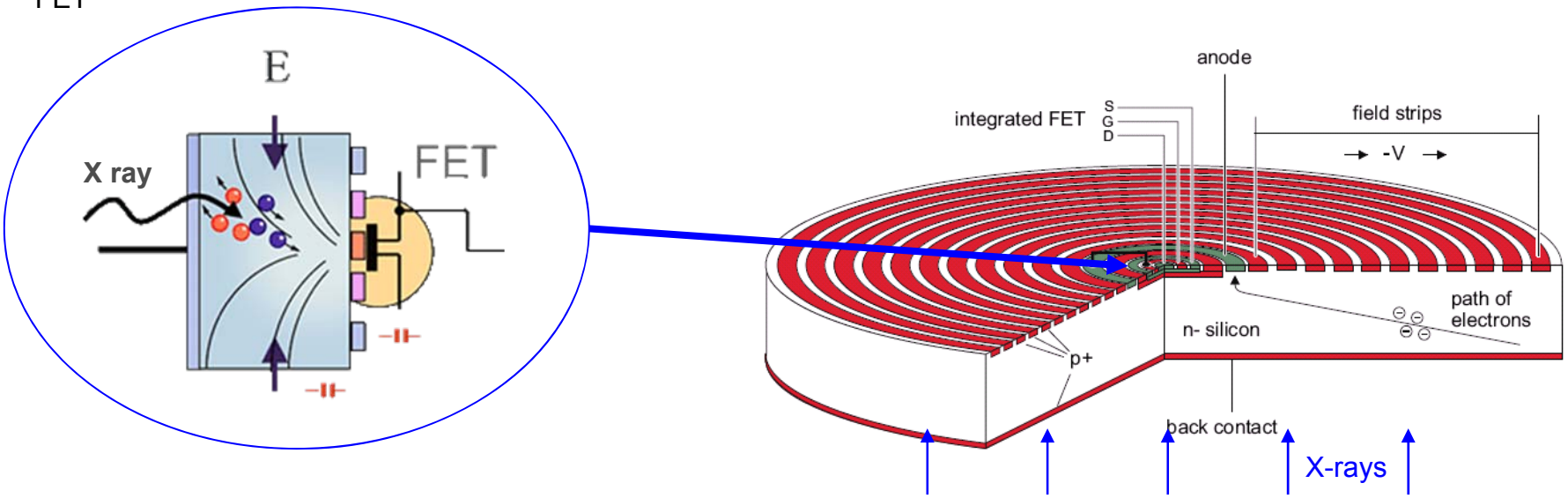
SDDs exploit the complex processing technologies available for planar processing of silicon:

Charge carriers collected at *low capacity* anode electrode contact

multielectrodes establish *transverse drift field*

charge is collected over large surface area (up to 1cm^2) without increasing anode capacity

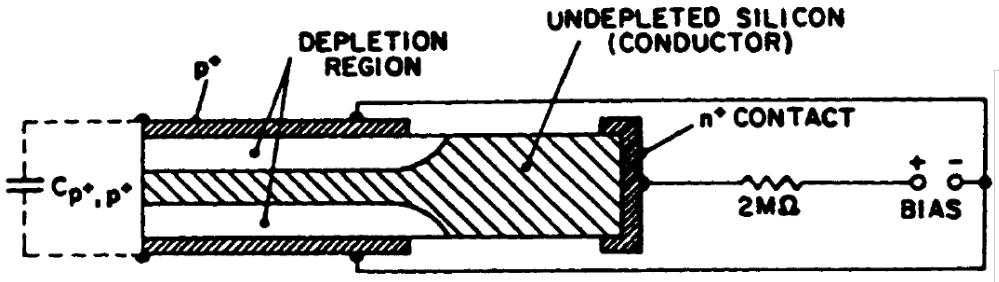
preamp' first stage ' FET' may be integrated into detector with C_{FET} <100fF

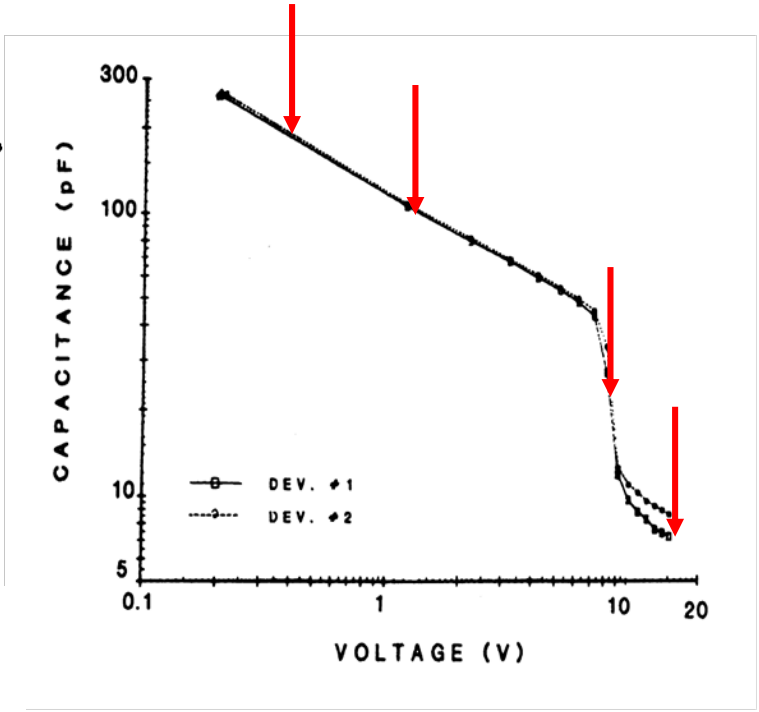


- high resistivity (i.e. low impurity) silicon → low bulk generation leakage current
- thermoelectric Peltier cooling -10ºC…-70ºC is sufficient for spectroscopy with pulse processor peaking times ~0.2 … ~10 µsec

→ compact and lightweight (~kgm) systems, insensitive to accoustics-vibrations

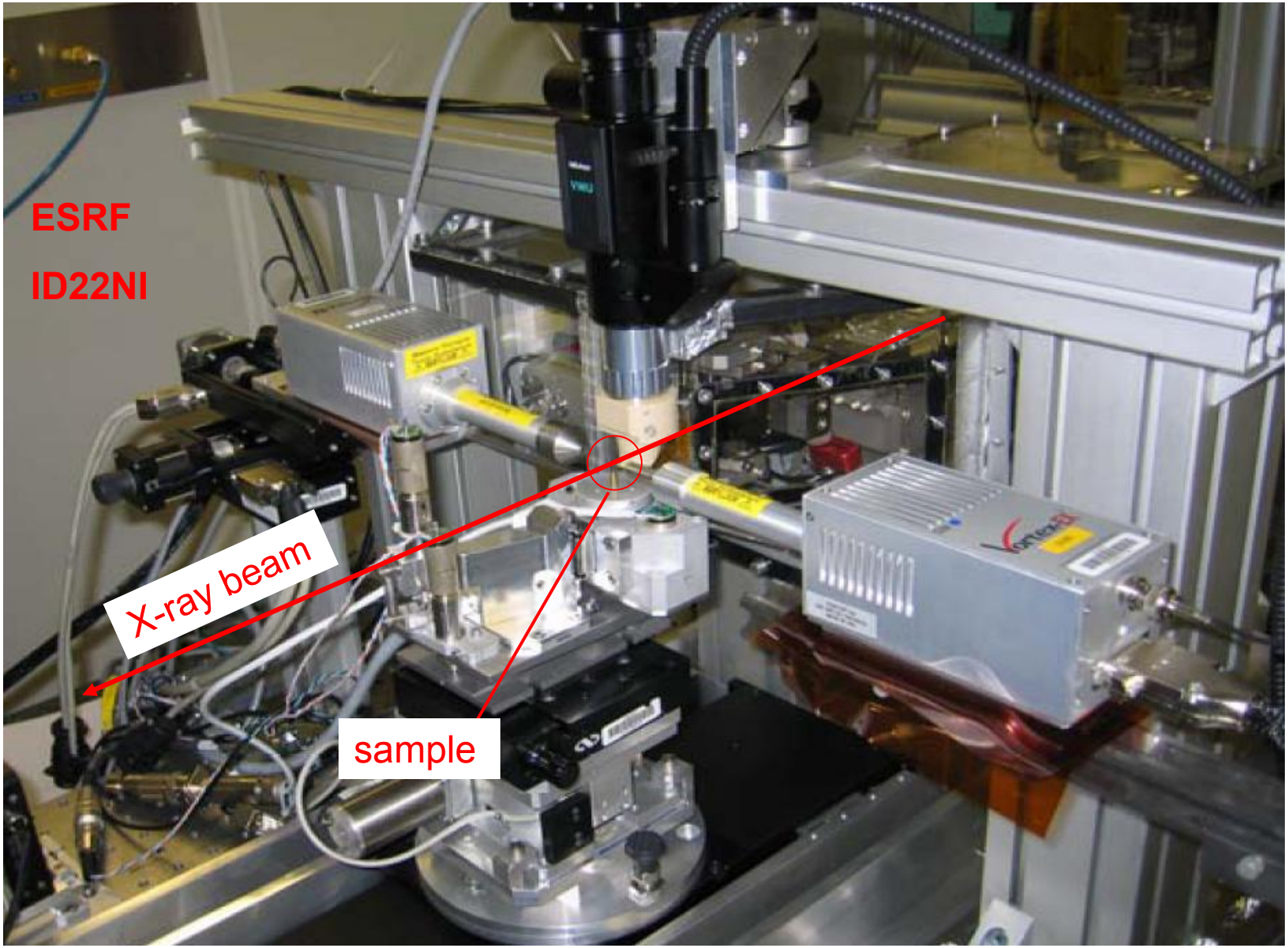
SDD: a clever trick to get low C



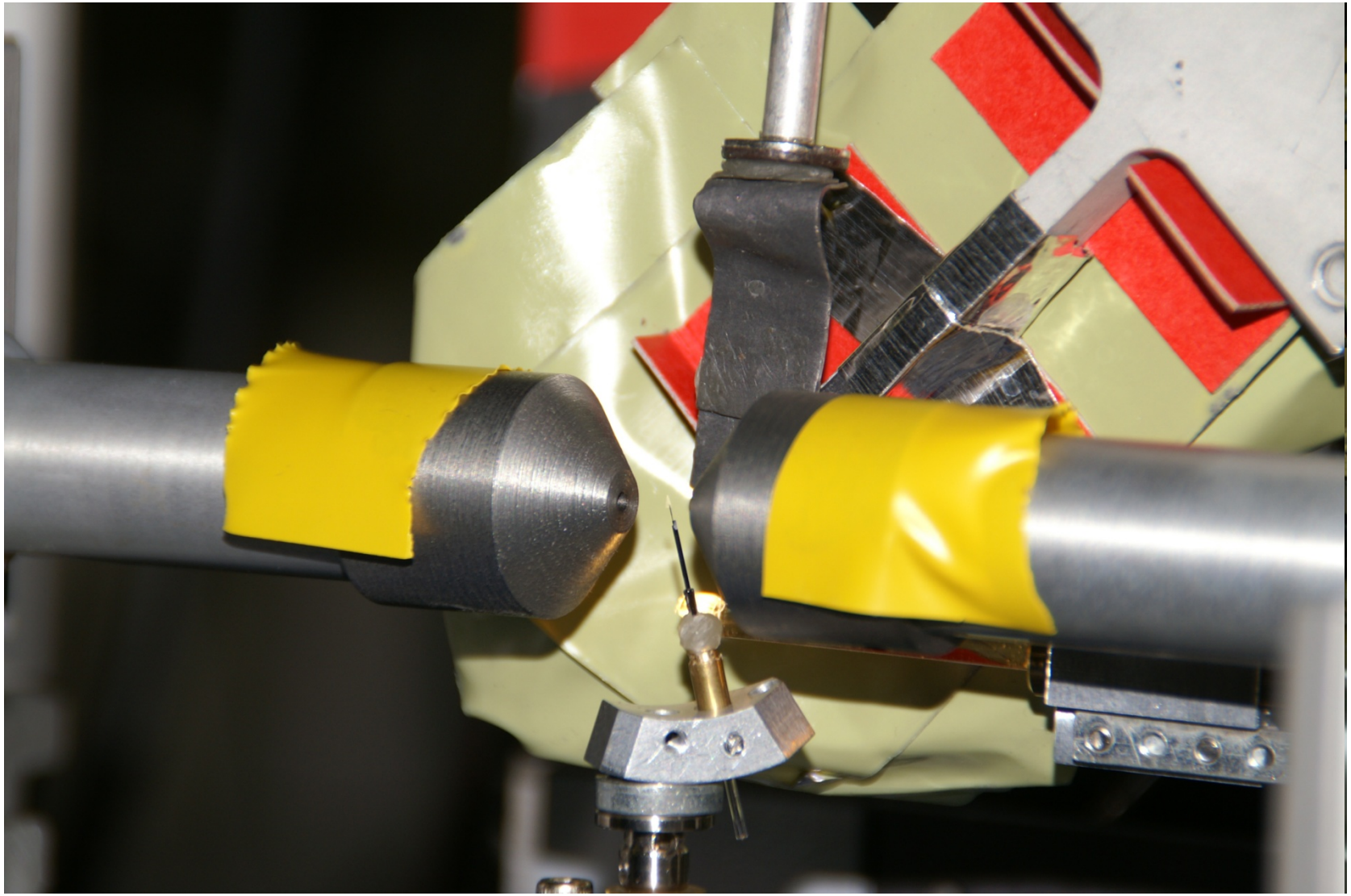


Gatti et al. IEEE Trans. Nucl. Sci. **NS-32** (1985) 1204)

practical SDD detector for high count rates



NASA ‘stardust’ experiment: Comet 81P/Wild



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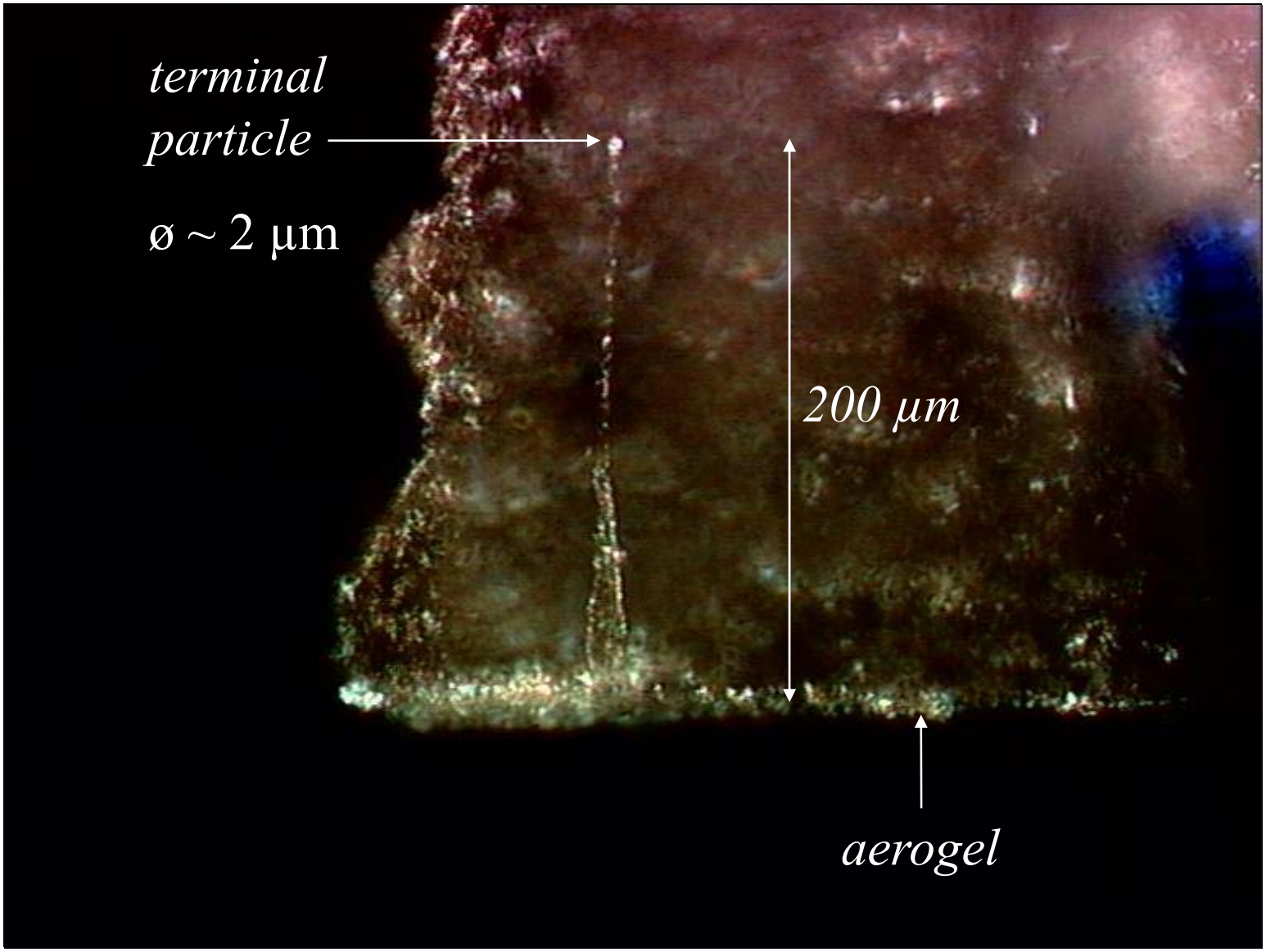
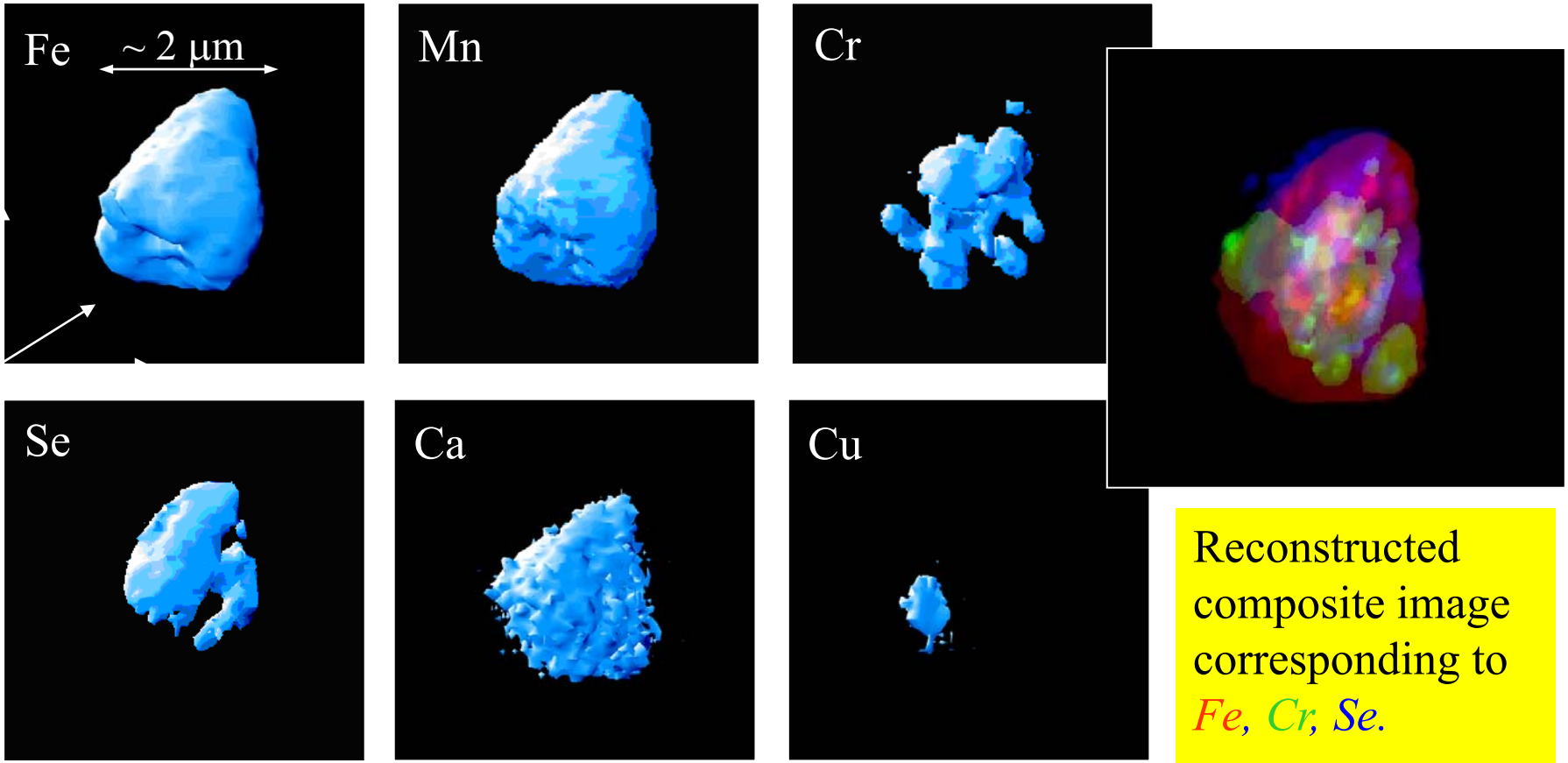


ESRF-ID13 0306

Low intensity isosurfaces (envelopes) of the detectable elements within the terminal particle:

Courtesy Laszlo Vincze, Univ. Gent

ESRF-ID13 0306

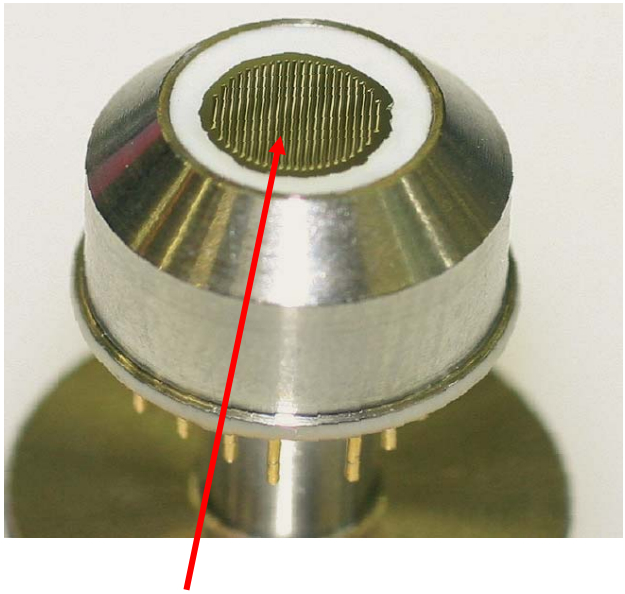


Reconstructed composite image corresponding to *Fe, Cr, Se.*

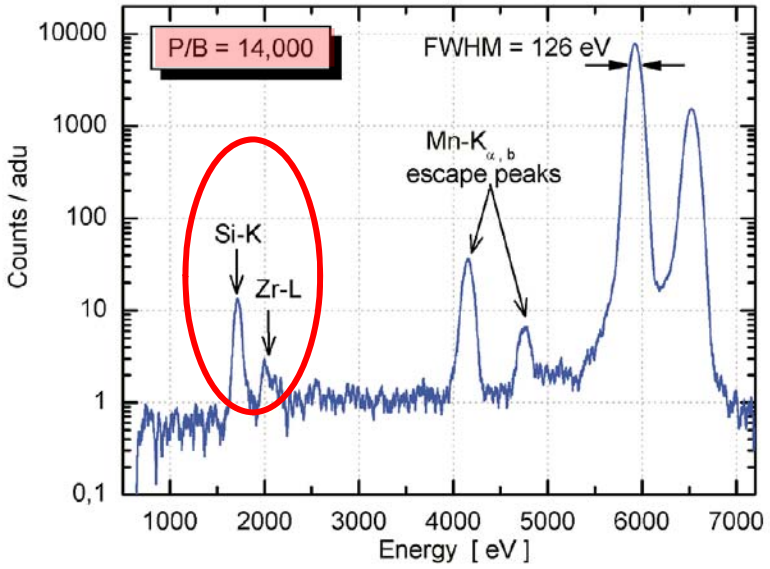
=> heterogeneous on the submicron level, main Fe-rich phase: olivine

SDDs with higher peak-valley performance

- very shallow, abrupt dopant-profile implant for front contact
- Zr collimator ring (avoids partial charge collection from X-rays at detector periphery

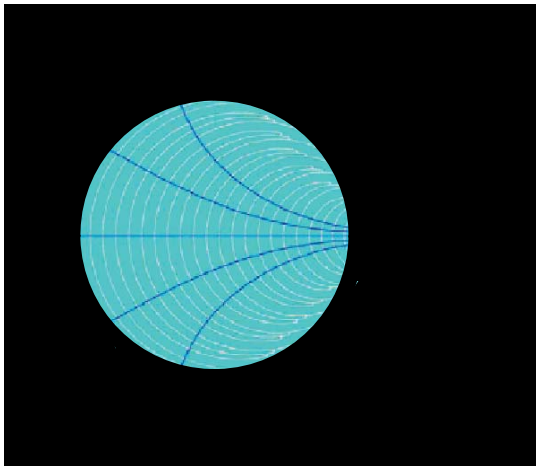


grid supported ultrathin
(~0.5µ) polymer window

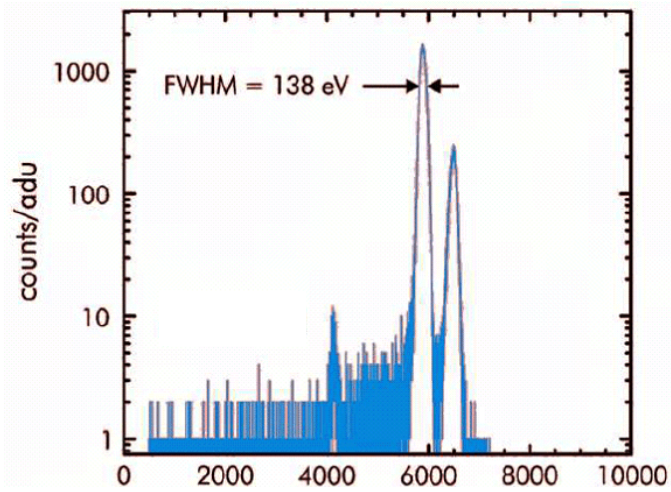


data from pndetector.de
(2µS pulse processor
peaking time)

integrated FET structure → near Fano-limited resolution at low count rates (peaking times >1μs)



Collimating mask e.g. Zr

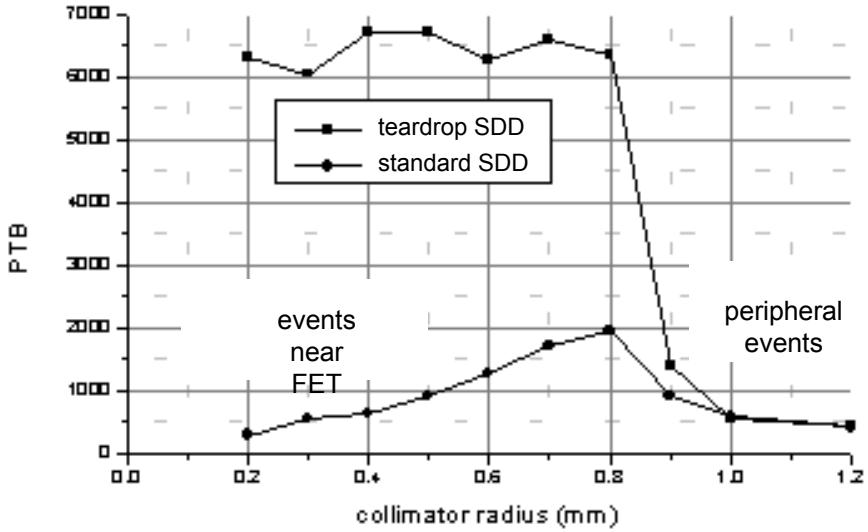


pnSensor GmbH

teardrop geometry + *metal collimator*

→peak / valley of 7000

→ *radiation protection of FET (hole-accumulation in surface oxide and trapping at Si- SiO2 interface)*

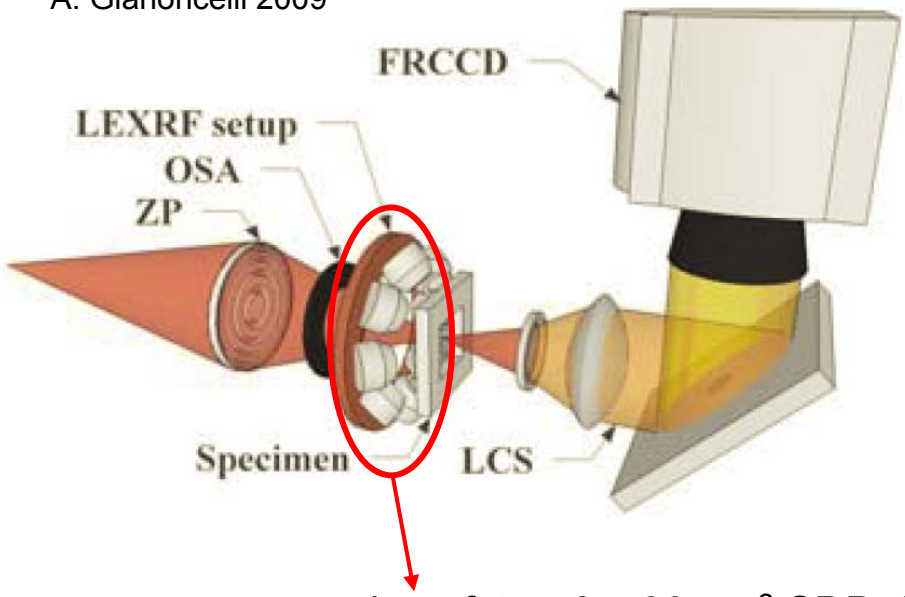


but

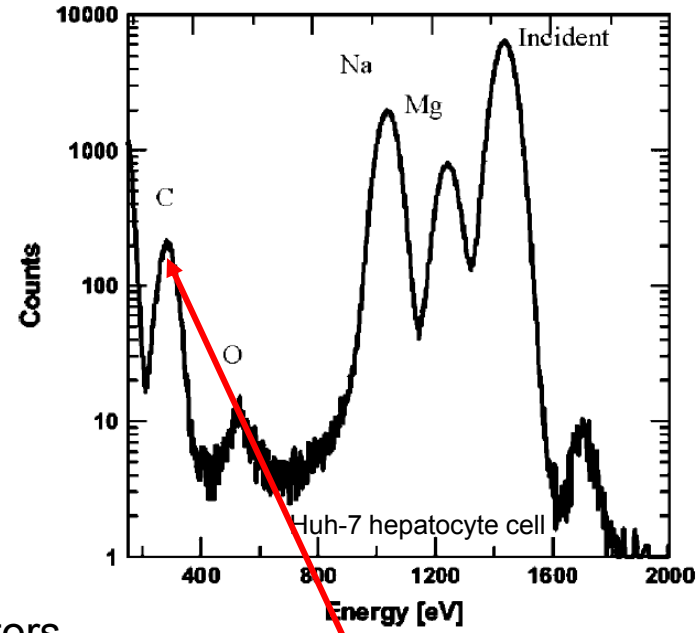
SDD Si thickness limit ~0.4mm *cf. 3mm for ‘conventional’ Si(Li) structure and >10mm for Ge*

TwinMic STXM at Elettra

A. Gianoncelli 2009



ring of 4 → 8 x 30mm^2 SDD detectors



1450 eV excitation,

77 eV fwhm C Kα line to count rate of 30 kcps
R Alberti 2009

!! Non-optimal geometry (beam scatter)
!! detector 'contamination' problems at low energies with 'wet' samples

Multielement detectors can offer higher overall count rates:
e.g. for N *independent* counting channels and a *uniform angular distribution of counts* we can expect a total count rate capability to be increased N-fold

multielement detectors (e.g. germanium 13 – 100 elements) are now commercialized

but

synchrotron undulator beams focused on sample are typically ~ 99% linear polarized
→ angular dependence of both Rayleigh (elastic) Compton (inelastic) scattering

an EDX detector measures *total* count rate (i.e. fluorescence and scatter)
→ in practice, *effective* count rate gain from an N channel detector is <N and often with degraded spectrum quality:
depends on:

- *the experiment-detector geometry*
- *the sample under investigation (concentration, Z of matrix, crystallinity…)*
- *energy of excitation beam…*

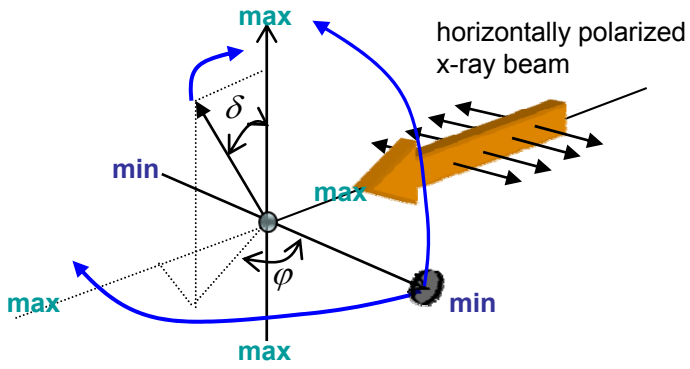
Polarization dependent elastic scattering cross section:

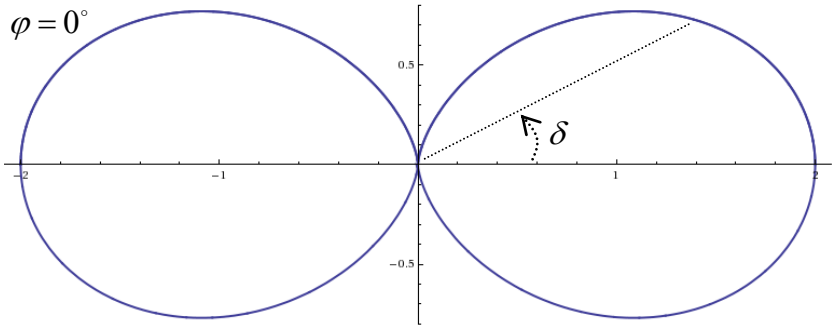
$$\left(\frac{d\sigma}{d\Omega}\right)_{\delta,\varphi} = \frac{r_e}{2}\left(1+\sin^2\delta\cdot\sin^2\varphi + P_0\left[\sin^2\delta\cdot\cos^2\varphi - \cos^2\delta\right]\right)$$

Compton scattering ignored here ('low energy' case)

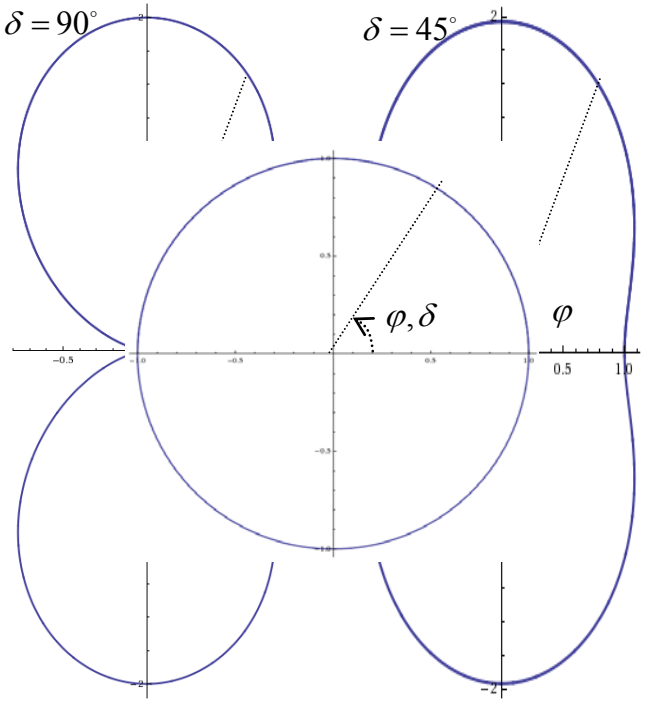
R. E. van Grieken, A. A. Markowicz, Handbook of X-ray Spectrometry (2002)

following graphics courtesy of J Szlachetko-ID21.





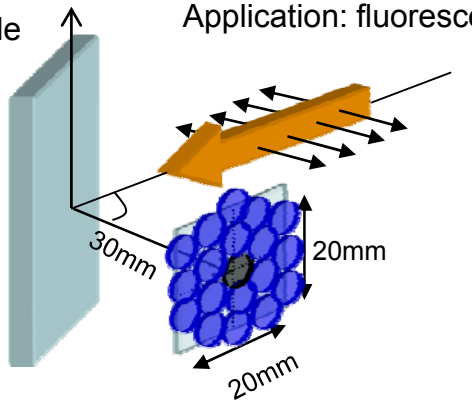
non isotropic process



Cross section for fluorescence radiation is isotropic (~independent of φ, δ)

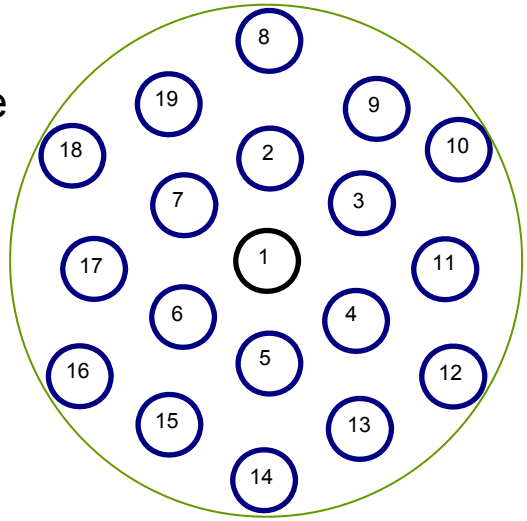
Sample

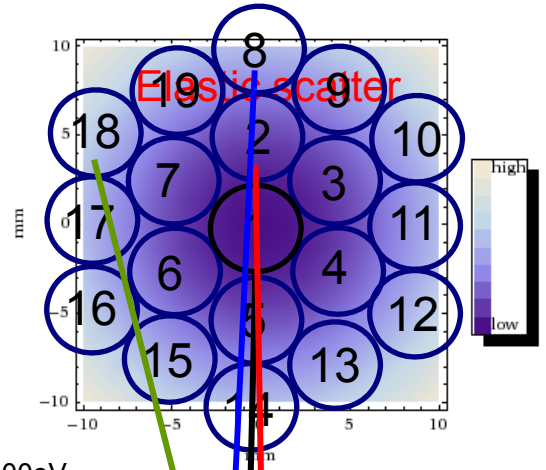
Application: fluorescence from 'dilute' samples

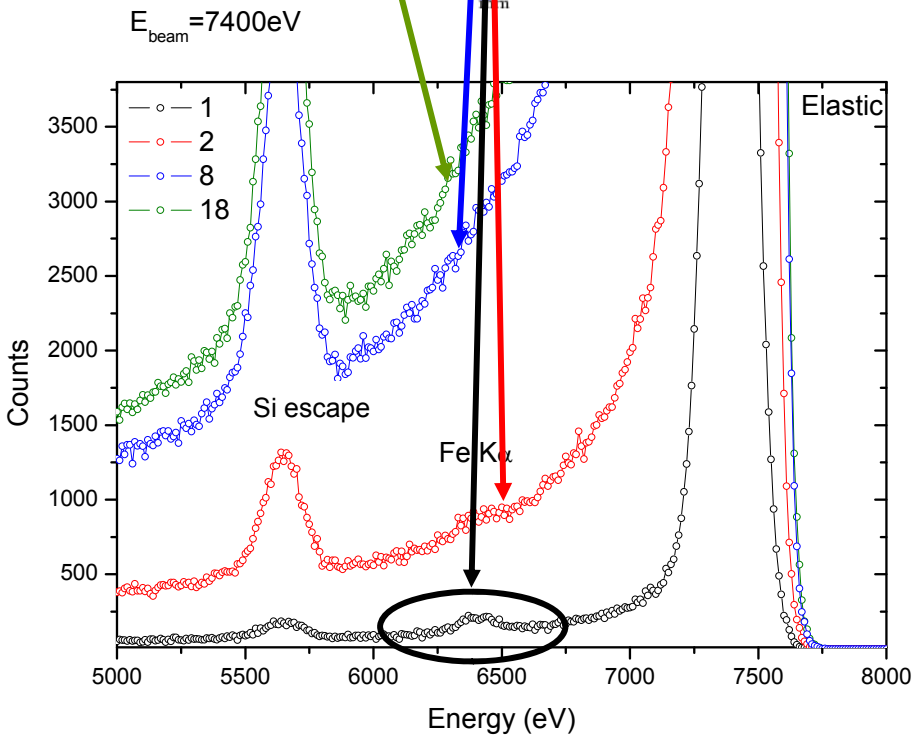


→ importance of multielement 'packing factor'
i.e. inter-element dead spaces

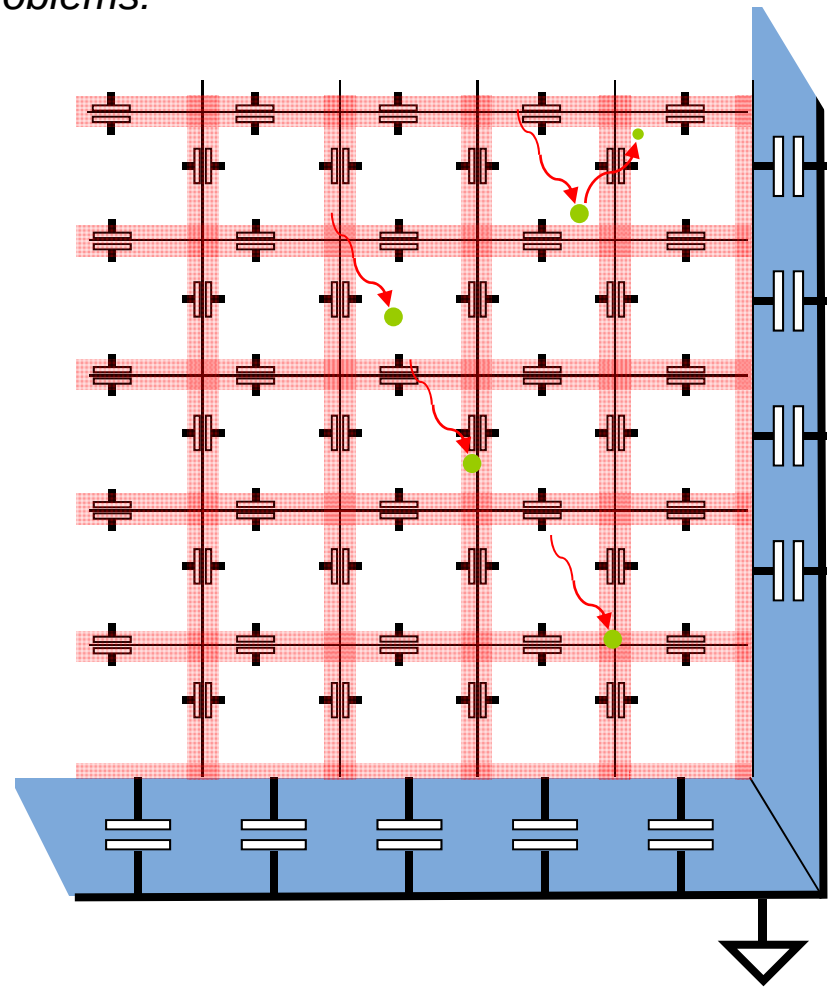
usual case for 'discrete elements'







a semiconductor can be electrically segmented by lithographic mask doping of contacts to create an x, y matrix of individual sensing areas. This gives a 100% sensitive area *but there are problems:*



After an X-ray is absorbed, diffusion creates a 'cloud' of electric charge which may be split the signal between bordering sensing areas.

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

These *physical crosstalk* effects clearly become more serious as the individual detector areas are reduced in size. Possible solution is use of a grid collimator to cover border areas.

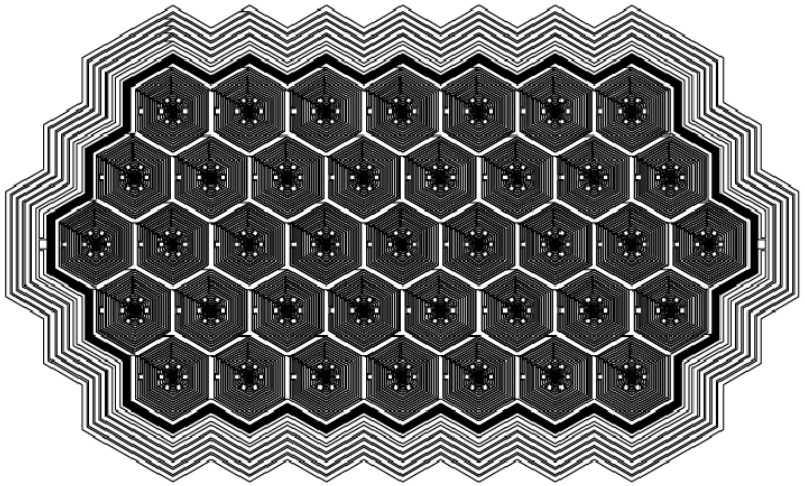
As well as its shunt capacity to a common rear electrode contact, each sensing area is capacitively coupled to its neighbours. *Electronic crosstalk* from individual fet preamplifier restore switching generates false spectral peaks. Problem is worst for short pulse processor peaking times.

- partial solution is 'synchronous' FET restore

multielement silicon drift diode arrays

'near wafer-scale' lithographic processing

→large, tightly-packed arrays possible



39 cell detector with on-chip FETs,

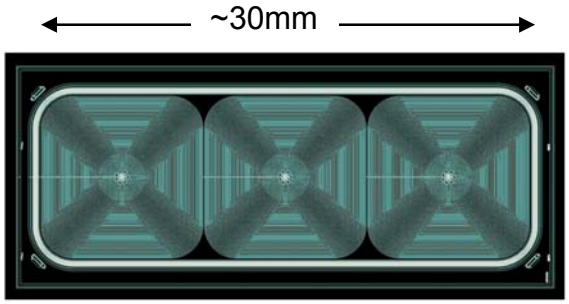
total active area 195mm²

(after L Strüder, MPI-Garching)

practical challenges of large cell counts:

→yield issues (bad cell and cell-to-cell variability, especially on-chip FET parameters)
→power dissipation (cooling!)
→need for multi channel pulse processors
→overall system fabrication complexity / cost

ASIC preamplifier-readout electronics



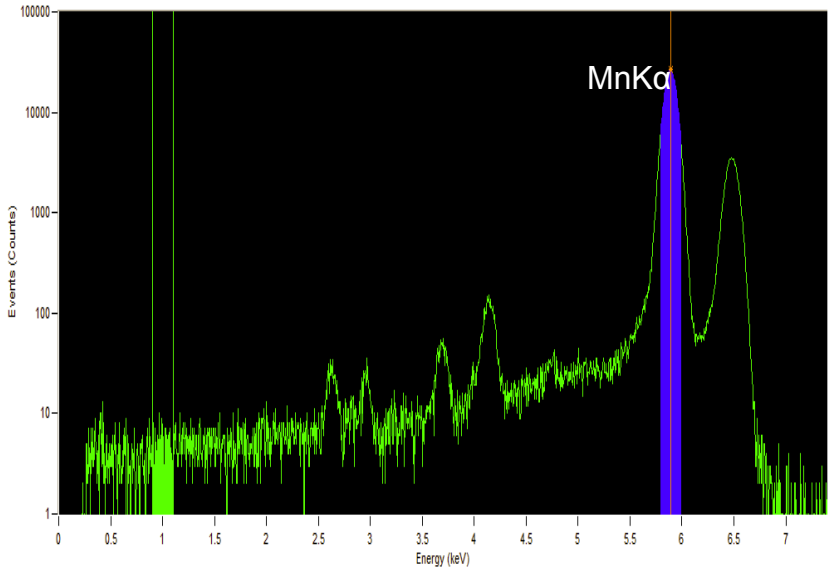
3-element monolithic array x 2

PNSensor / PNDetector

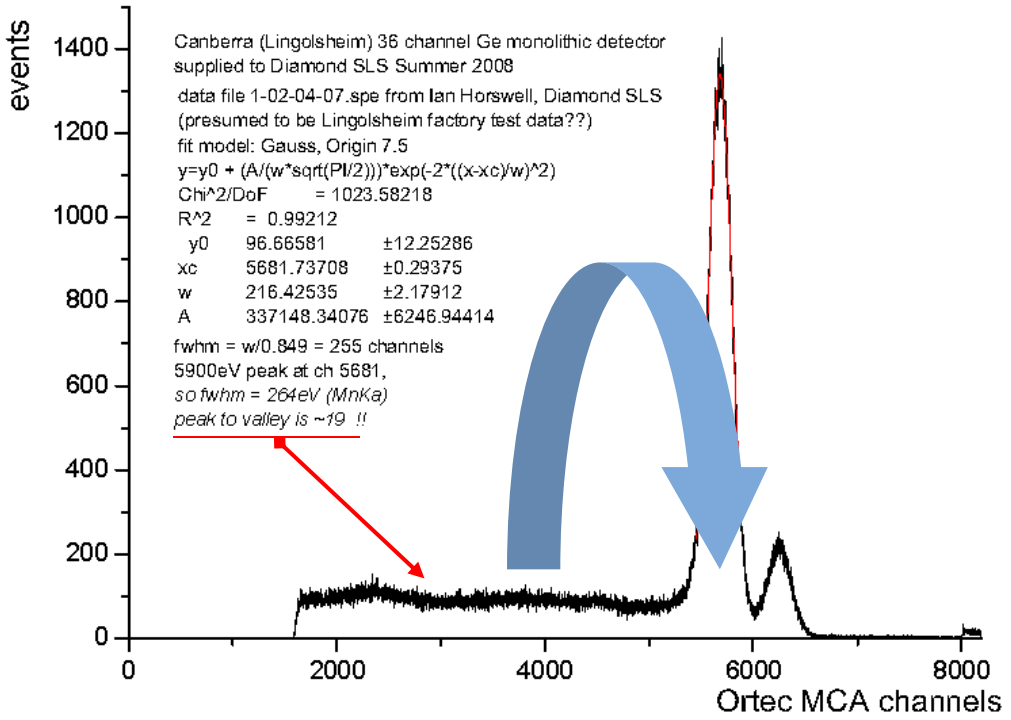
Pulse processor peaking time (μS) vs. resolution

Ch	0.1	0.2	0.3	0.5	0.72	1
1	203.8	176.2	164.3	150.0	144.1	137.9
2	207.2	178.9	163.7	149.5	143.9	139
3	208.6	177.1	163.3	149.4	142.3	139.6
1	202.2	178.2	163.6	150.9	143.1	139.9
2	202.3	173.3	162.3	149.9	142.8	138.6
3	204.3	177.4	163.6	151.1	143.6	138.5

~5Mcps total

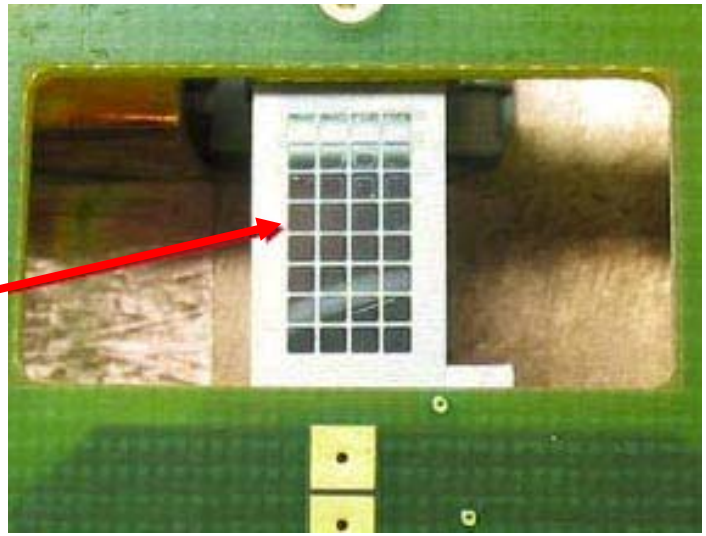


low count rate spectrum at optimum peaking time
but what does high rate crosstalk look like??



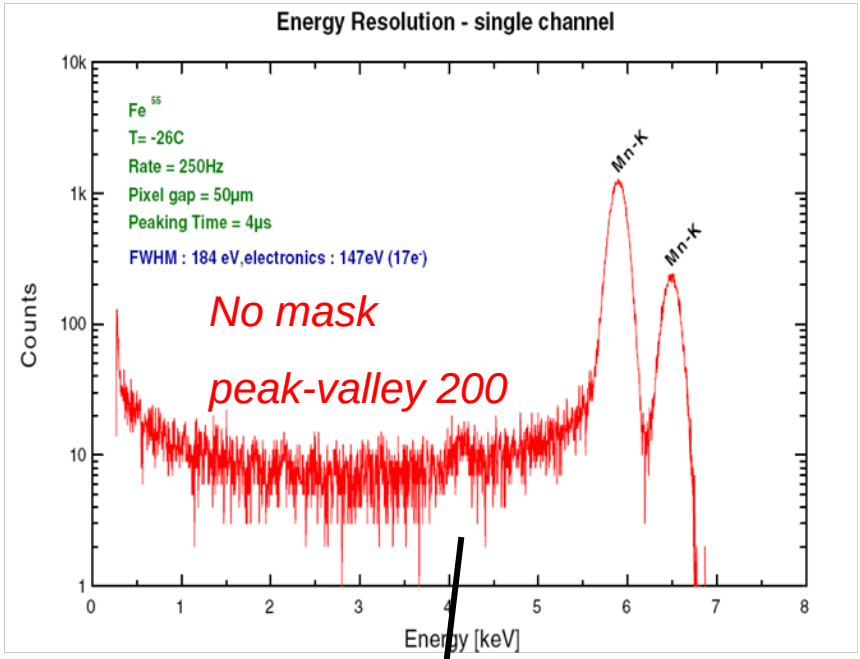
'split charge' tail events move into photopeak with time and count-rate dependency…

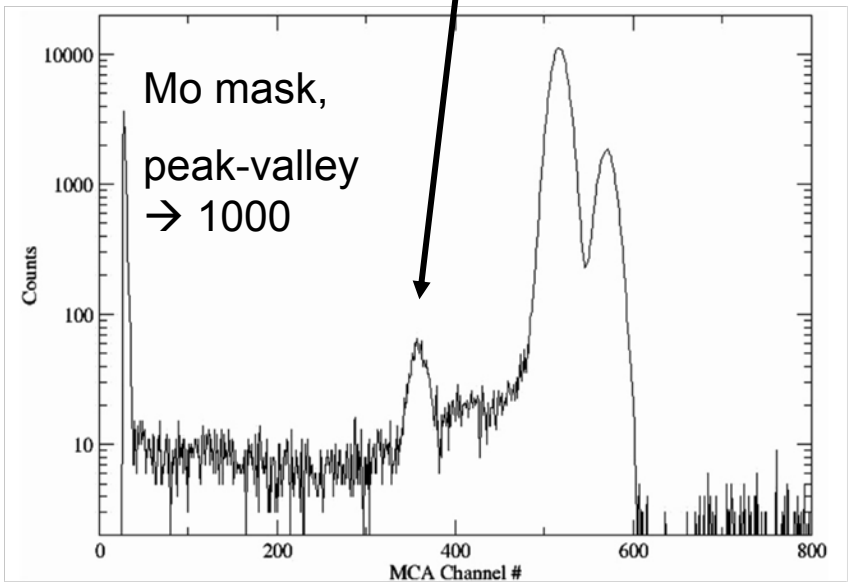
Molybdenum mask on planar silicon detector developed at NSLS-BNL

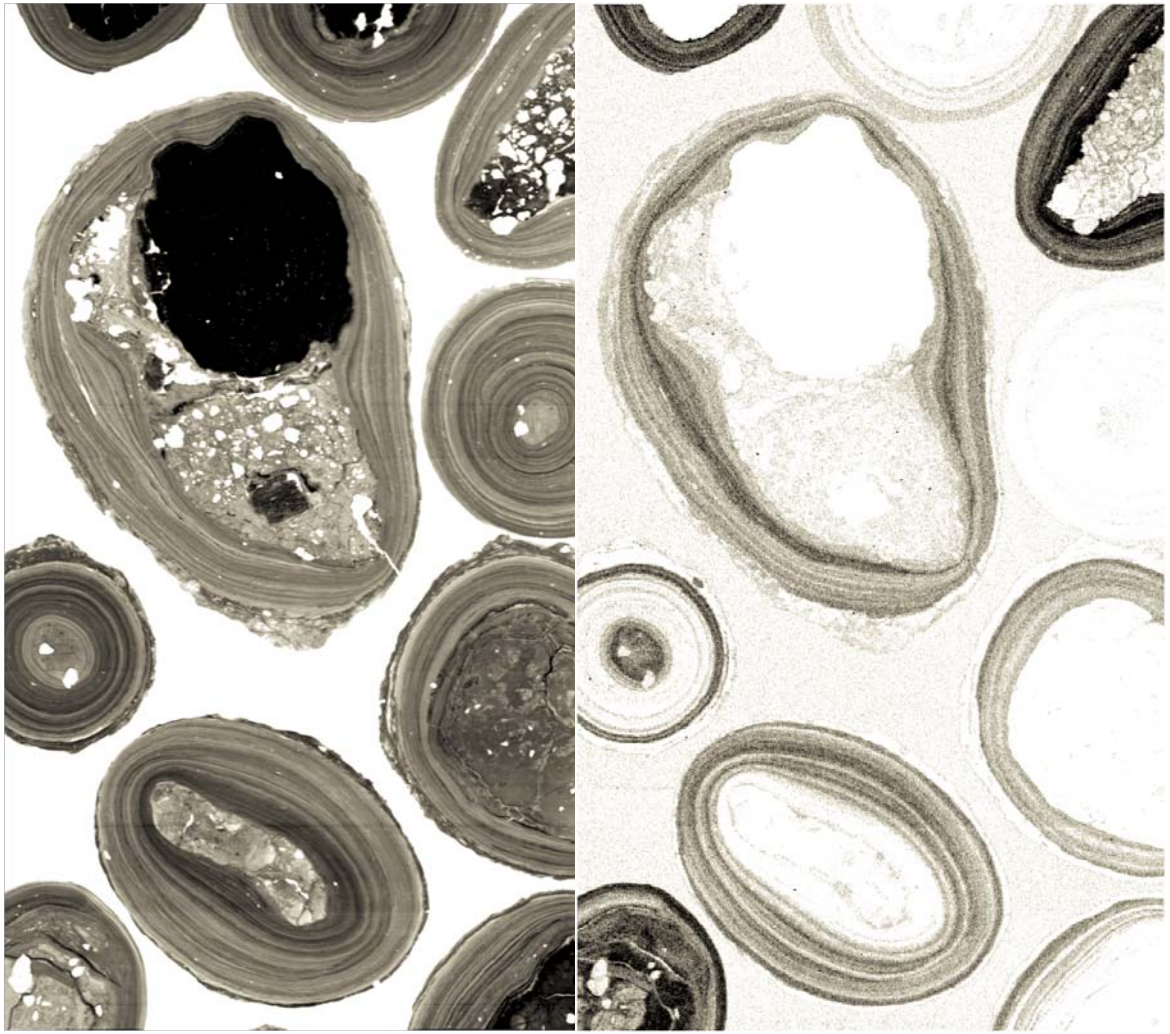


C.G. Ryan et al. /Nucl. Instr. and Meth. Phys. B 260 (2007) 1–7

‘Maia’ fluorescence detector now in development by BNL and CSIRO,
→ 384 x 1mm2 detector elements, 400µ thick Si



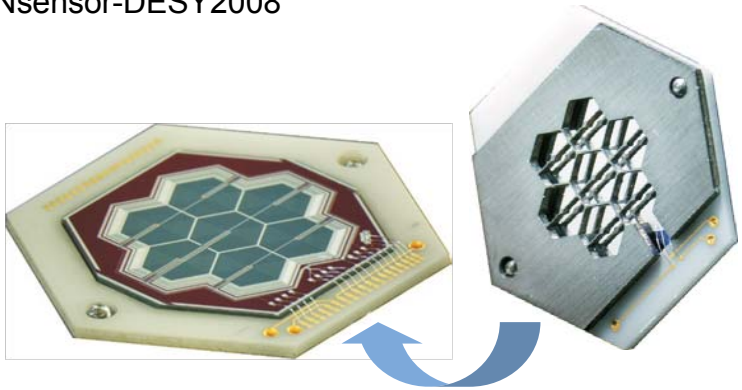


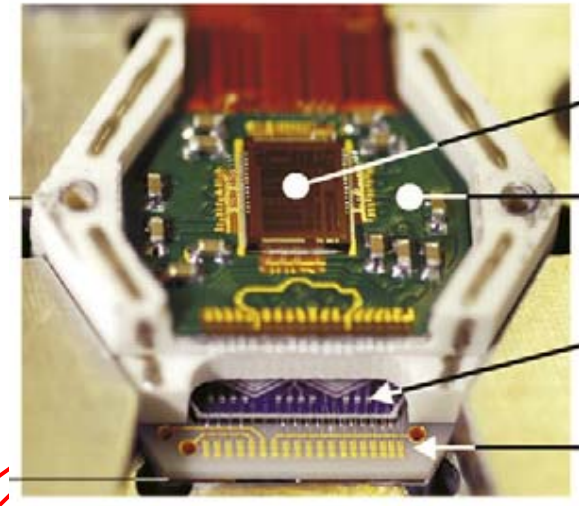


Fe (left) and trace 10-100ppm Y (right) images of ‘Rose Dam’ natural mineral iron-oxide nodules
1625 x 2625 pixels 5ms integration per 7.5 µm pixel, 17.2 keV excitation

multi-monolithic SDDs: first attempts

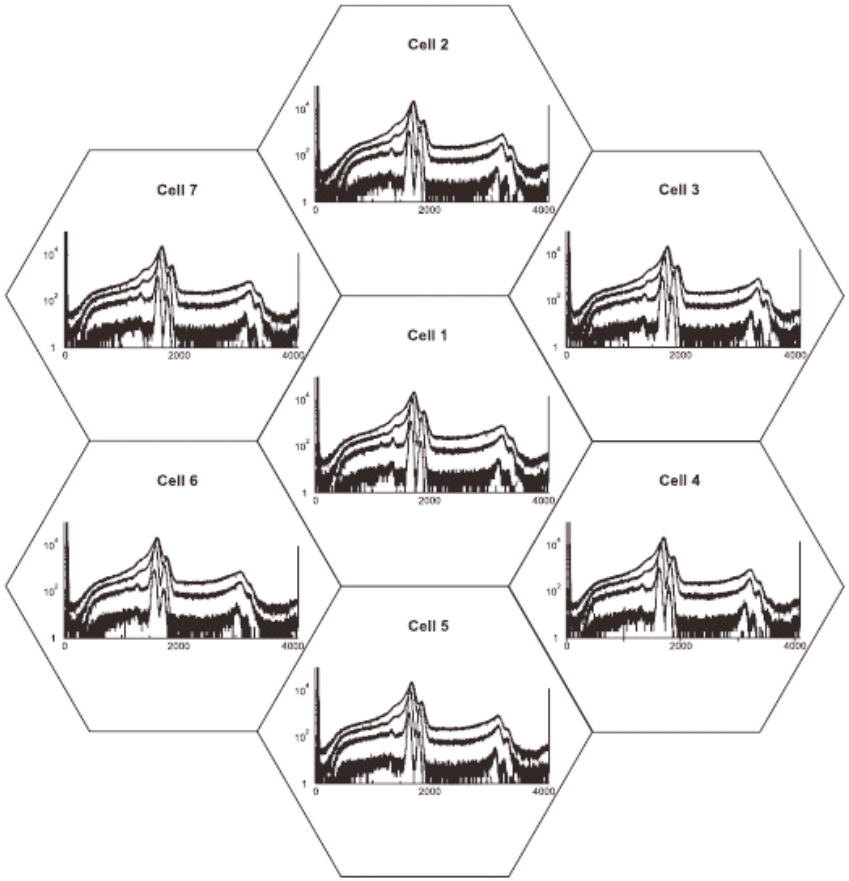
PNsensor-DESY2008





Hansen et al. DESY2008

DESY multi-monolithic SDD: performance

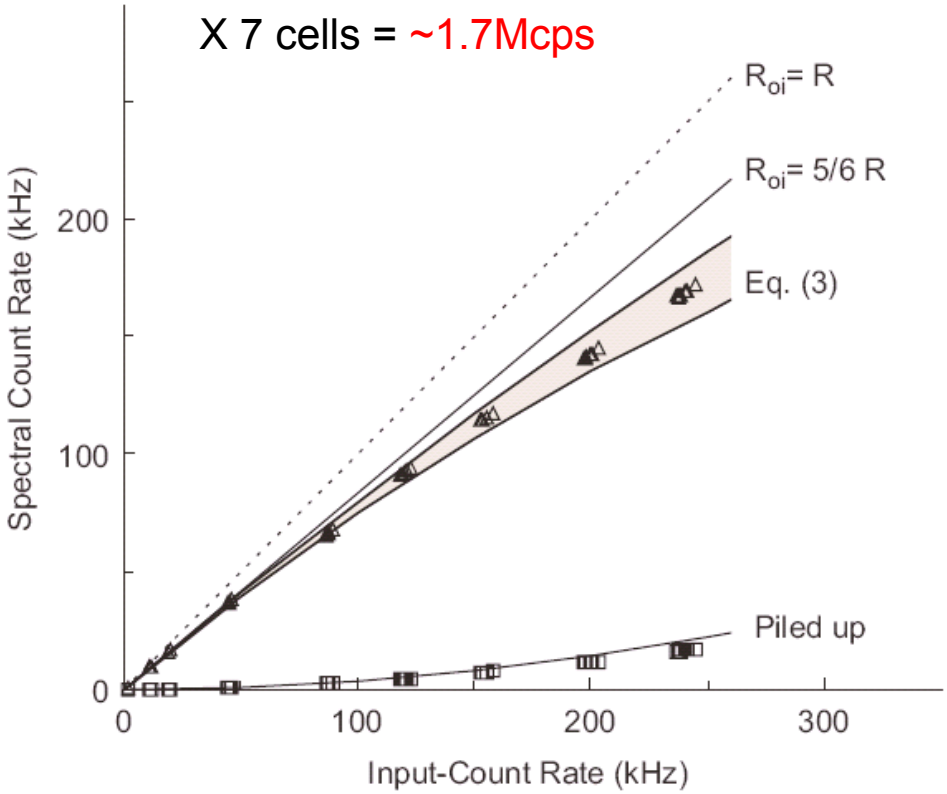


Cu K spectra from the 7 SDD channels, resolution 250~300ev fwhm (at +7ºC !)

Total count rate

~ 240kcps (30% loss)

X 7 cells = ~1.7Mcps



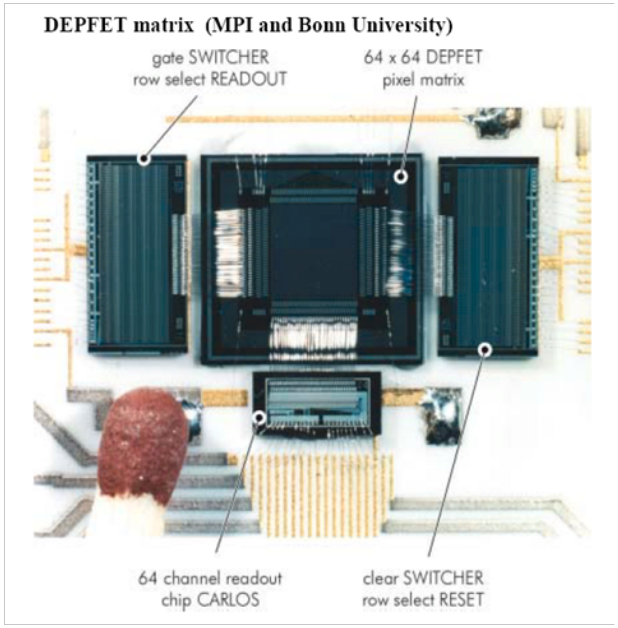
Hansen et al. DESY2008

DEPFET-macropad arrays

Matrix arrangement of DEPFET transistor amplifiers at centre of drift diode structure 'macropad' cells .

Various readout possibilities:

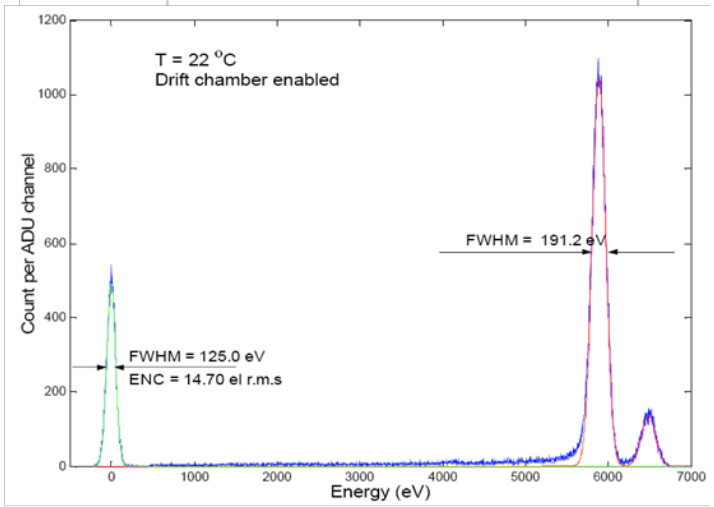
- direct macropad addressing
- row-by-row readout through single node,
- parallel readout of columns (fast)
- pixel bump bonding to CMOS ASIC (*XFEL project*)



'crosstalk' issues…

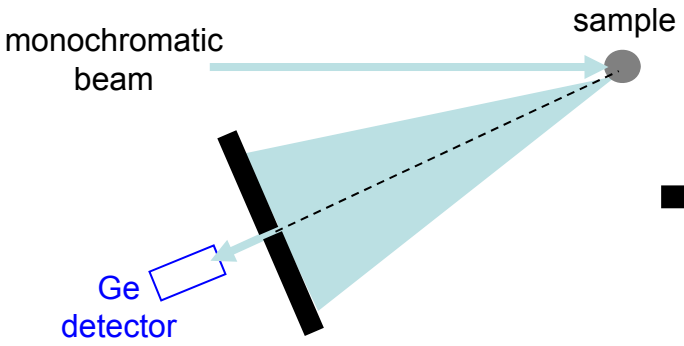
4 x 4 x 1mm^2 pixel prototype tests

at room temperature
191 eV resolution

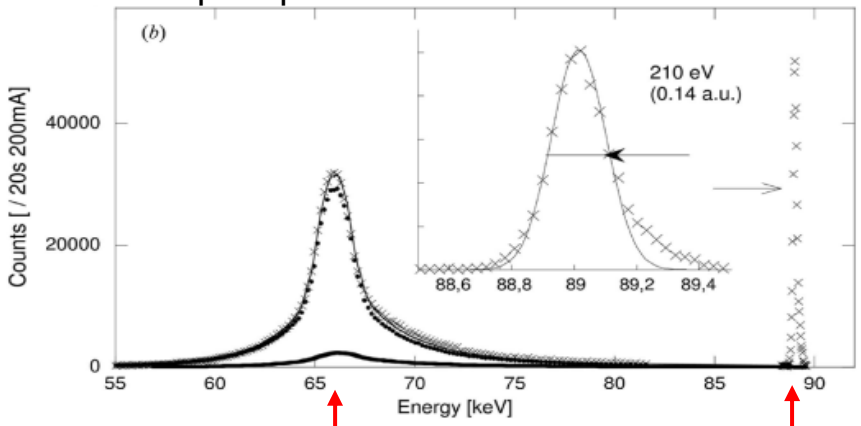


G Lutz, L Strüder MPI Garching

ID15: magnetic Compton scattering spectra (fixed, monochromatic beam energy ~50 … 150keV)
Slit selection of Compton backscatter angle



Compton profiles



Compton scattered photon peak

elastic scattered photon peak (beam energy)

At present, only Germanium detectors are adequate for this application:

- high Z for adequate absorption
(large detector volumes (>cm^3) *with negligible charge carrier trapping*)

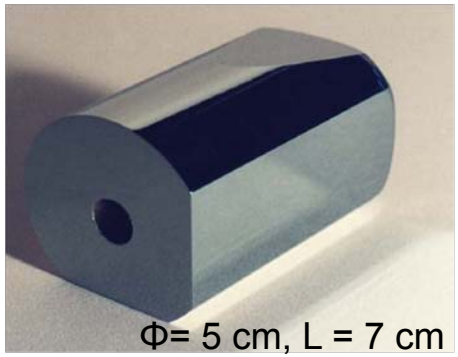
-high energy resolution (ΔE/E ~0.5% at 100keV) and *clean* Gaussian line shape

At high energies, Compton scattering is dominant interaction

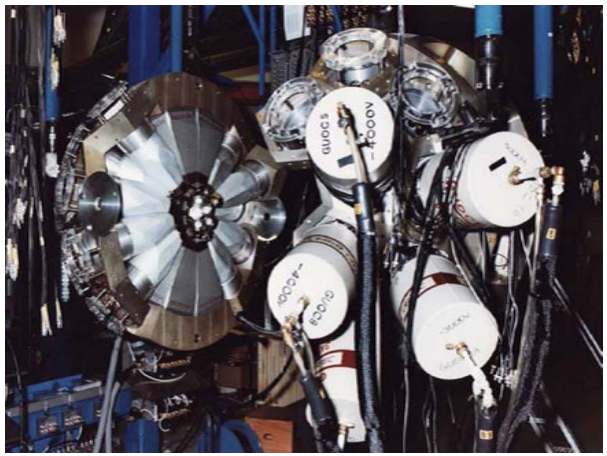
→large volume Germanium detectors required

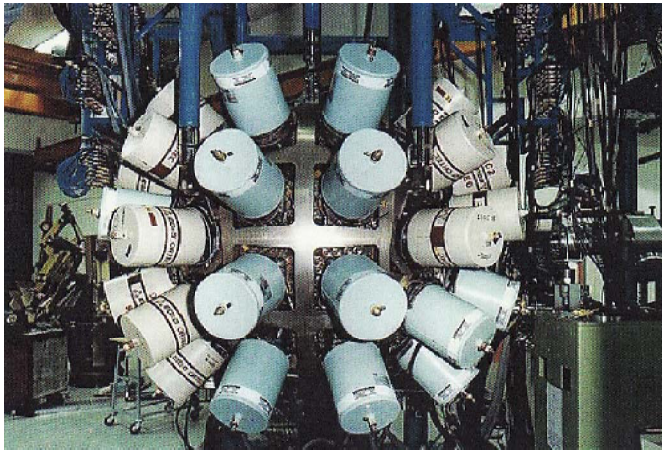
→spectroscopy may only possible by reconstructing photon interactions using timestamped information from multiple detectors

(partially absorbed events can be vetoed)



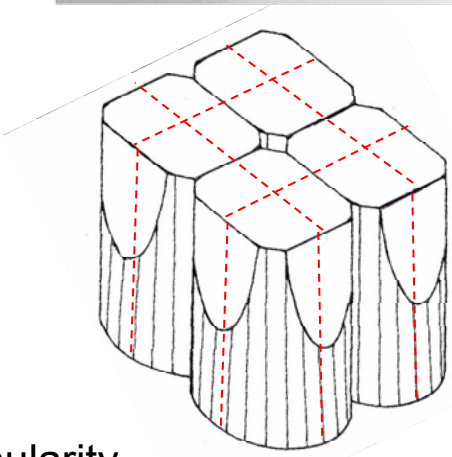
'Clover' detectors
shaped for close-packed geometry and near 4π solid angles in nuclear pyhsics experiments





eurogam2

P. Jones et al., Nucl. Instr. and Meth. **A 362** (1995) 556



Detectors can be electrically 'segmented' to give better tracking granularity

Summary

At low count rates (<< 1/peaking time) Silicon and Germanium approach theoretical performance limits (Fano statistics) over the large range of X-ray energies used at 3rd generation synchrotrons.

Higher ΔE/E resolutions, or better X-ray absorption, can theoretically be obtained with compound semiconductor materials. For precise, quantitative spectroscopy, there are today no competitors to Silicon and Germanium due to a lack of large, pure-and-perfect crystals of binary or ternary compounds.

For low energies (<20keV), Silicon is a near-ideal detector material offering advanced processing technologies including the fabrication of on-detector low-noise electronics. Higher energies require Germanium, but detectors made from this material are unlikely ever to reach the sophistication of silicon devices due to the lack of a large scale market (i.e. the electronics industry!) to spur the needed developments.

Pulse processor pile-up effects degrade spectral quality and counting efficiency at high count rates (≥ 1/peaking time). Multi-element detectors may attain higher count rates but the gain is limited by geometric considerations and the practical challenges of making individual detector channels operate in a true, independent manner free of crosstalk.

some bibliography

Radiation Detectors: general principles:

G Knoll ‘Radiation Detection and Measurement’, Wiley , 2000

C Delaney, E Finch ‘Radiation Detectors: Physical Principles and Applications’, OUP 1992

Semiconductor Detectors, physics and practical application issues:

H Spieler, ‘Semiconductor Detector Systems’, OUP, 2005

G Lutz, ‘Semiconductor Radiation Detectors: Device Physics’, Springer Berlin 1999

above are in ESRF-ILL Joint Library (in fact, permanently on my office shelf)