Detectors, data collection and other practical issues

Liquid Scattering X-ray School, November 2007

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X-ray detectors:

X-ray photons go in, electrons come out

Types of detectors, based on media used in conversion:

1. Gas detectors
2. Scintillators
3. Semiconductor detectors
4. Superconducting elements (tunneling junctions)
5. Photoemulsion

Detectors: X-ray Film, Geiger counter, Phosphor screen, Scintillators, wire 1D (linear) and 2D PSD, CCD, CMOS, avalanche photodiode, ionization chamber, etc.

Detectors:

Parameters important for detectors:

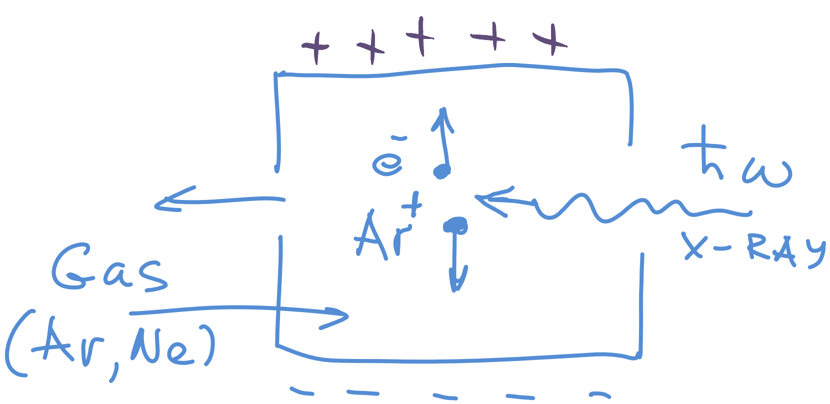
1. Efficiency
2. Spatial resolution (per element)
3. Solid angle acceptance (number of elements)
4. Dynamic range, dark noise
5. Readout Speed / Dead time
6. Energy resolution/discrimination
7. Distortions – spatial, uniformity of response, resistance to radiation damage, stability
8. Size, weight and cost

Gas Ionization Chambers

Einstein's photoelectric effect (Nobel Prize 1921)

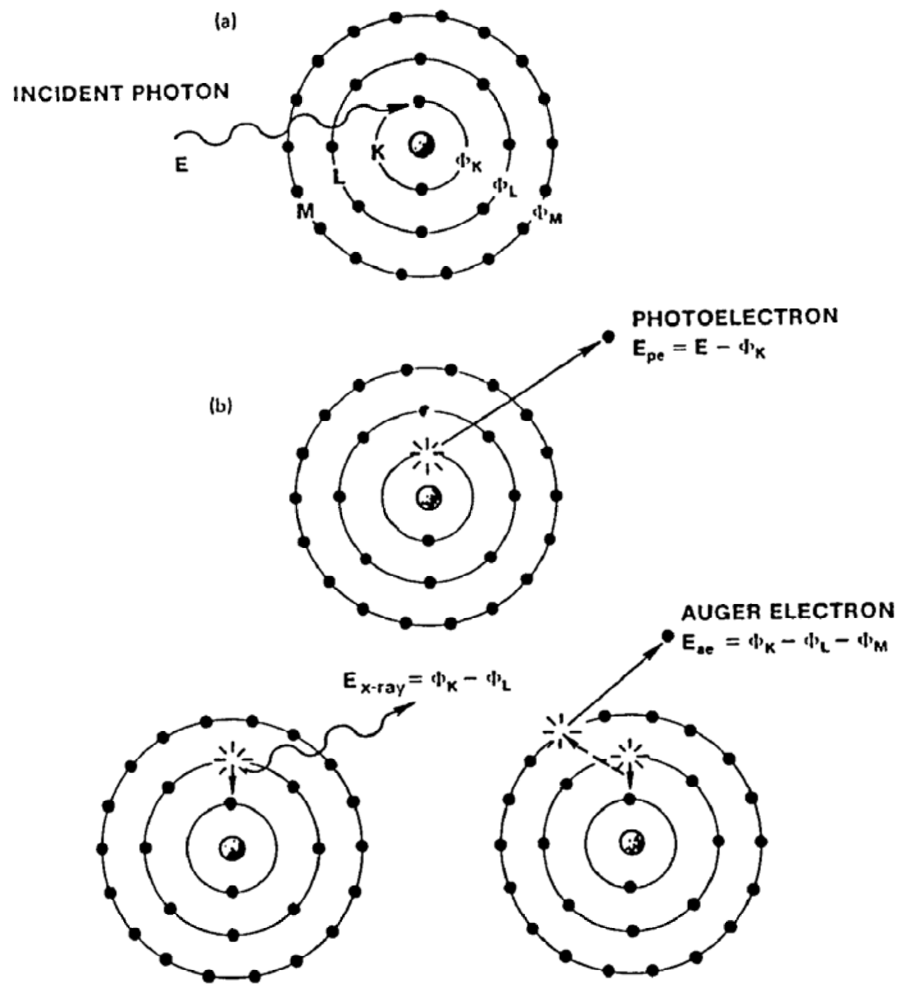
$$E_{\text{kin}} = h\nu - W$$

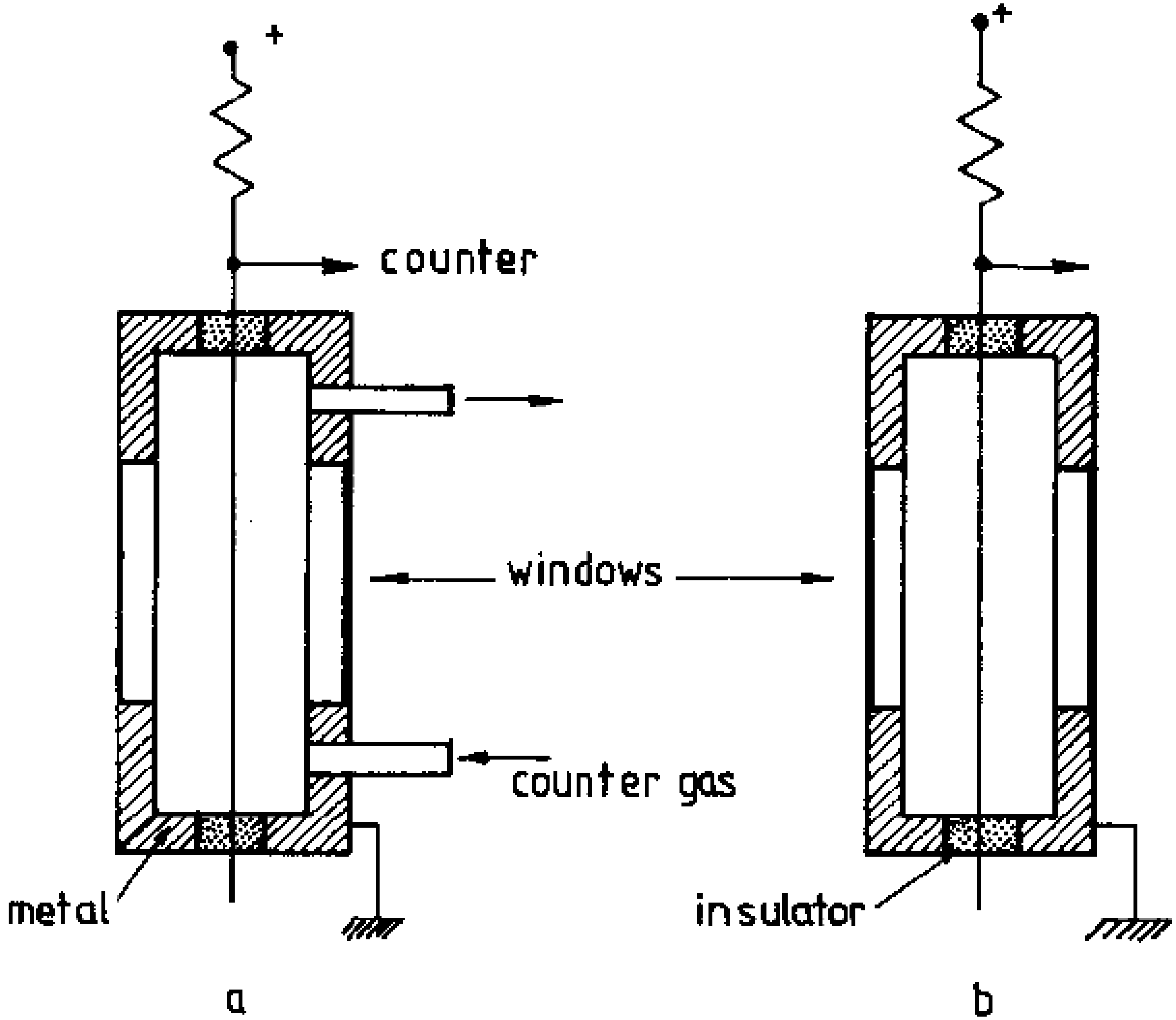
$$\text{Ar} \xrightarrow{h\nu} \text{Ar}^+ + e^-$$



	(eV)
He	27.8
Ne	27.4
Ar	26.4
Kr	22.8
Xe	20.8
NaI (Tl)	50
Si (Li)	3.8
Ge	3

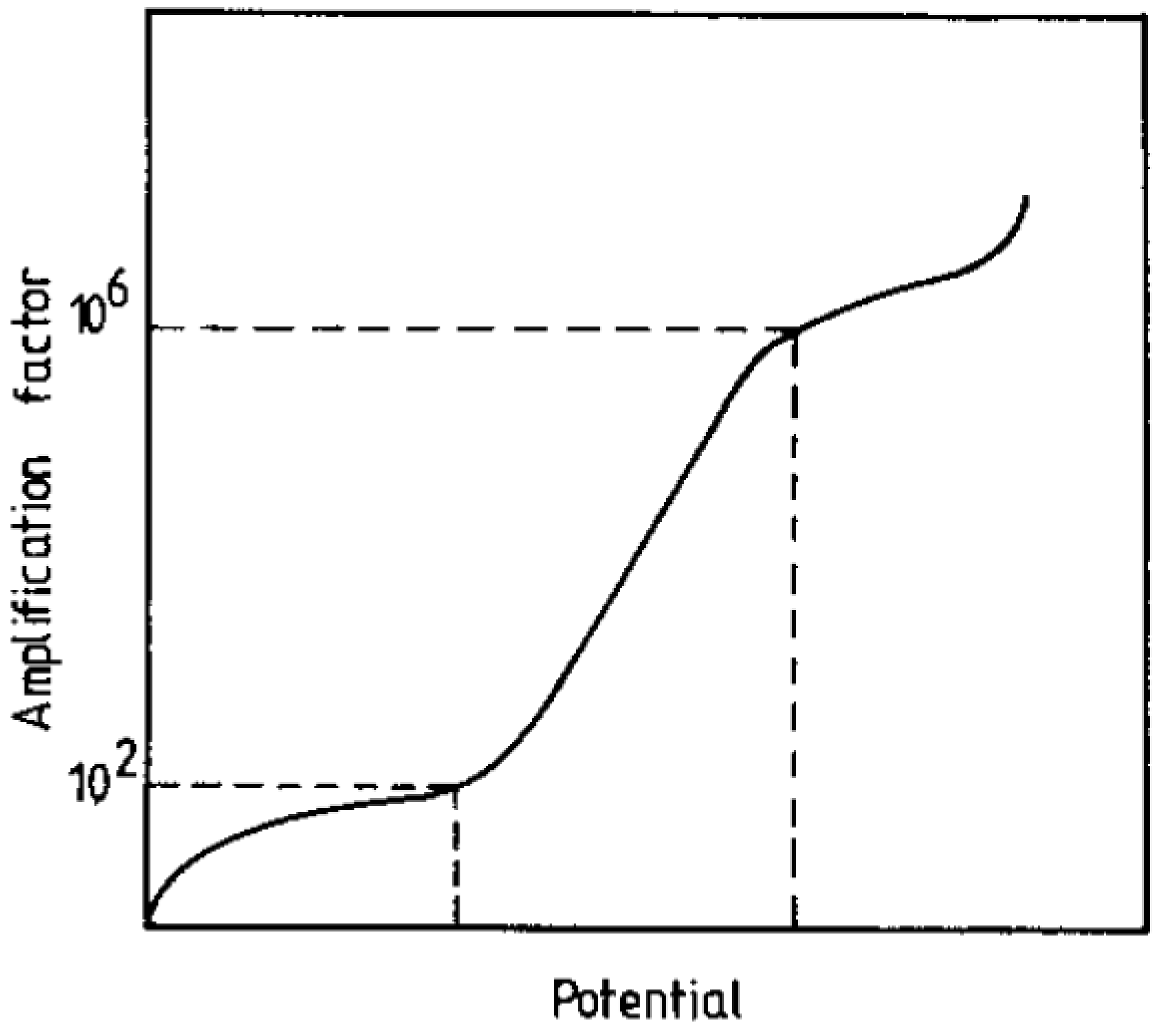
Auger and Photoelectric effect





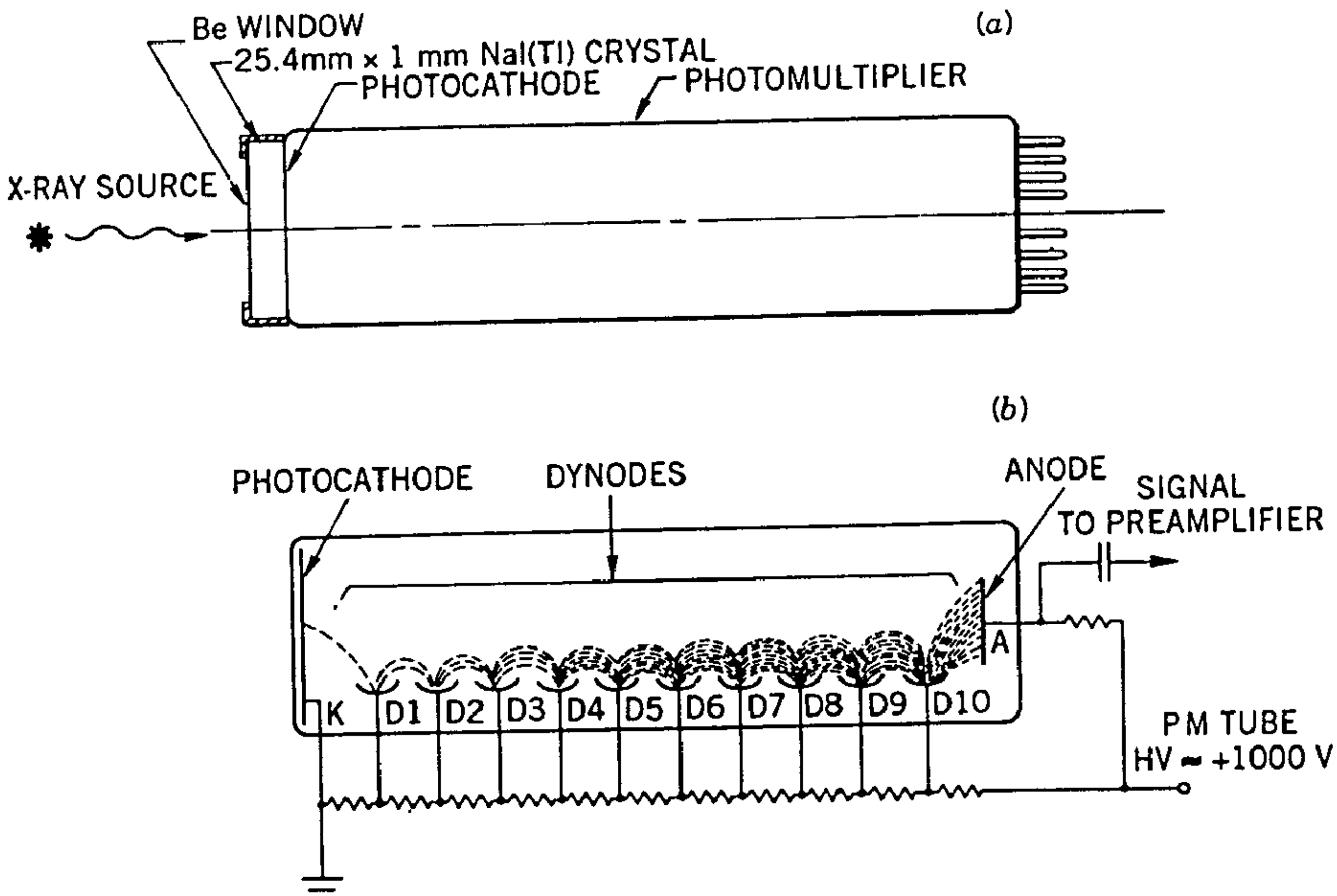
(a) Flow Gas Ion chamber and (b) sealed Gas Ion chamber
Gas used: He, Ne, Kr, He, Xe. Windows: mica, mylar, kapton etc.

Gas detectors

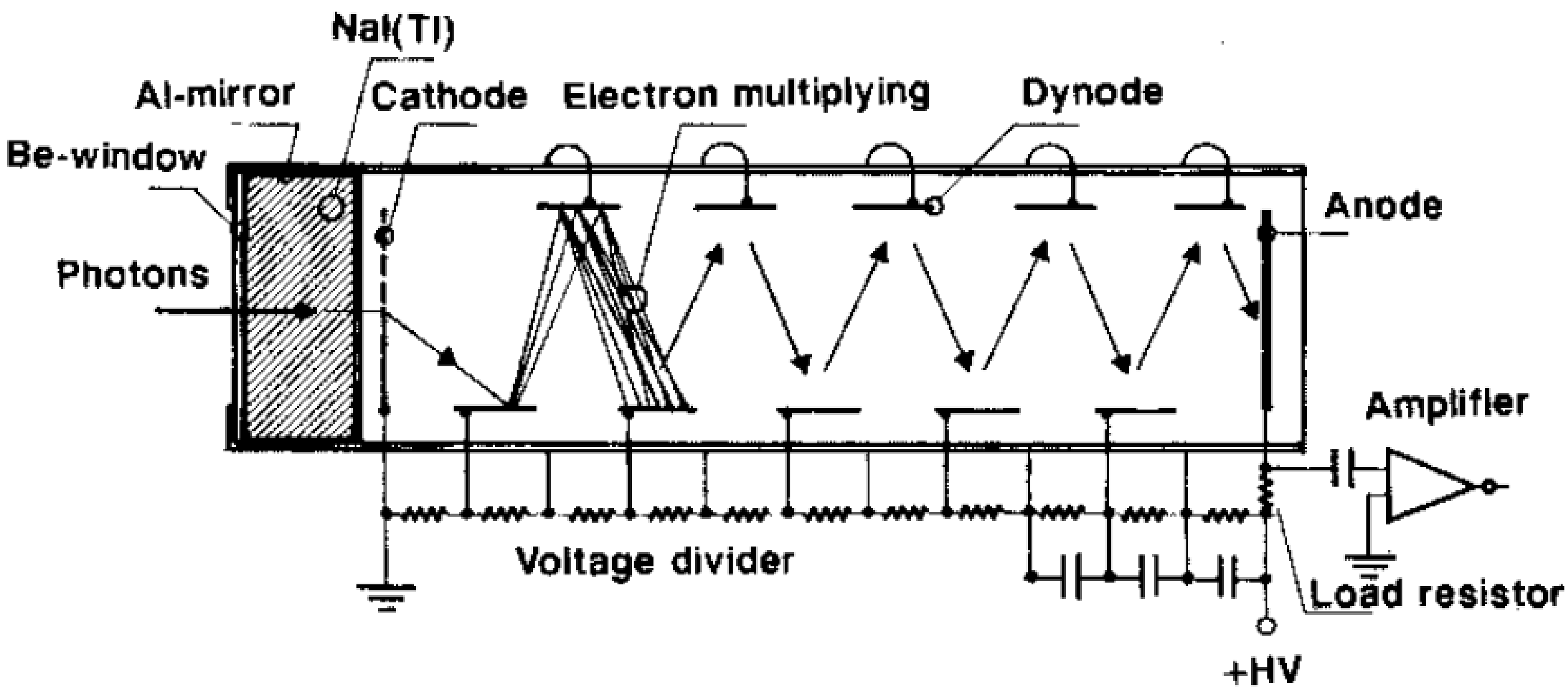


Region of proportionality for gas detectors

Scintillation Counters

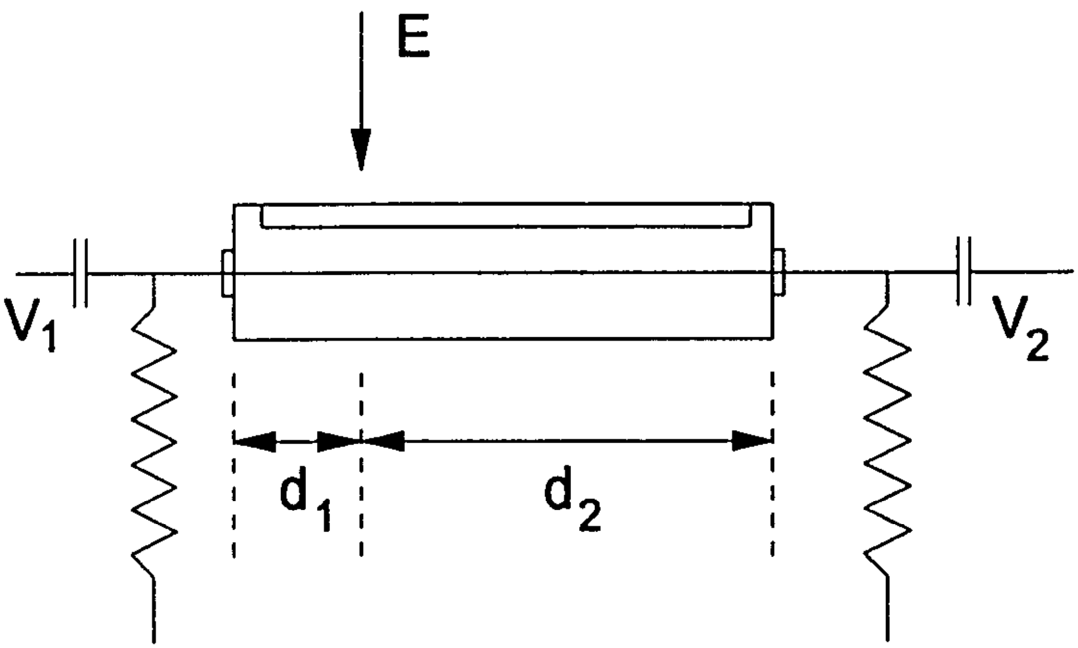


Layout of Scintillation Counter



qw

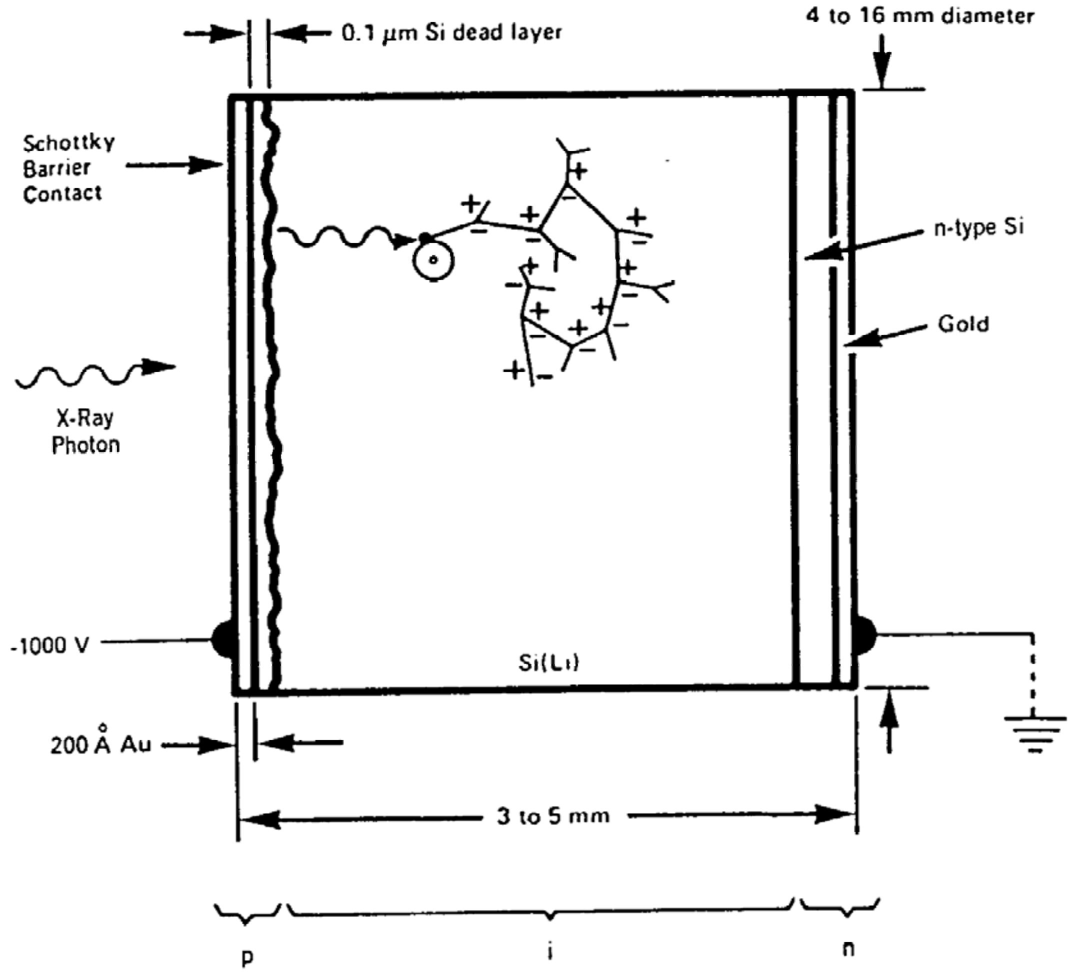
Position Sensitive detector



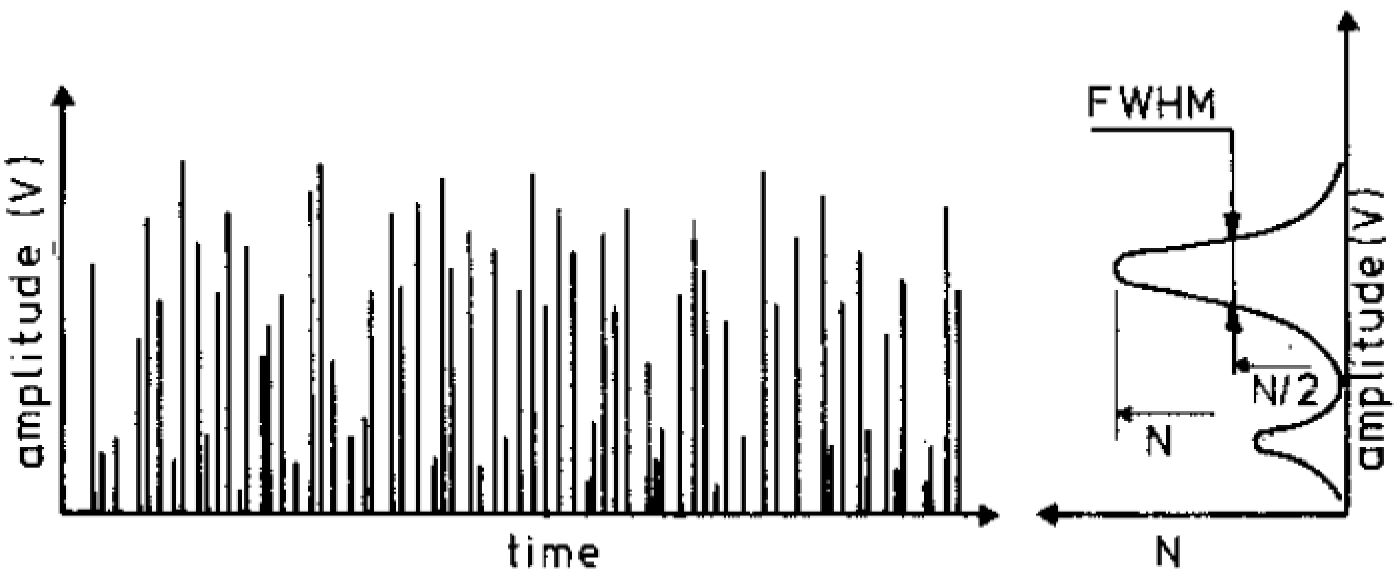
Idea: Proportional Counter (Ion Chamber) with high resistivity anode - to slow down electron pulses

Measure d1 and d2 based on how quickly the signal travels along the anode wire ("rise time").

Solid State Detectors: Si, Ge



Pulse height analysis and discrimination



Pulse amplitude (related to x-ray energy)
FWHM is 130-150 eV for Si detectors, 900 eV for gas detectors, ~3500 eV for scintillators

Energy discrimination

Low-energy contributions – primarily fluorescence (inner shell electronic excitations) – can be eliminated by adsorbers, pulse height discrimination

High-energy contributions – higher harmonics – can easily survive adsorbers. Not a problem for *grazing incidence* measurements.
Can be eliminated by pulse height discrimination.

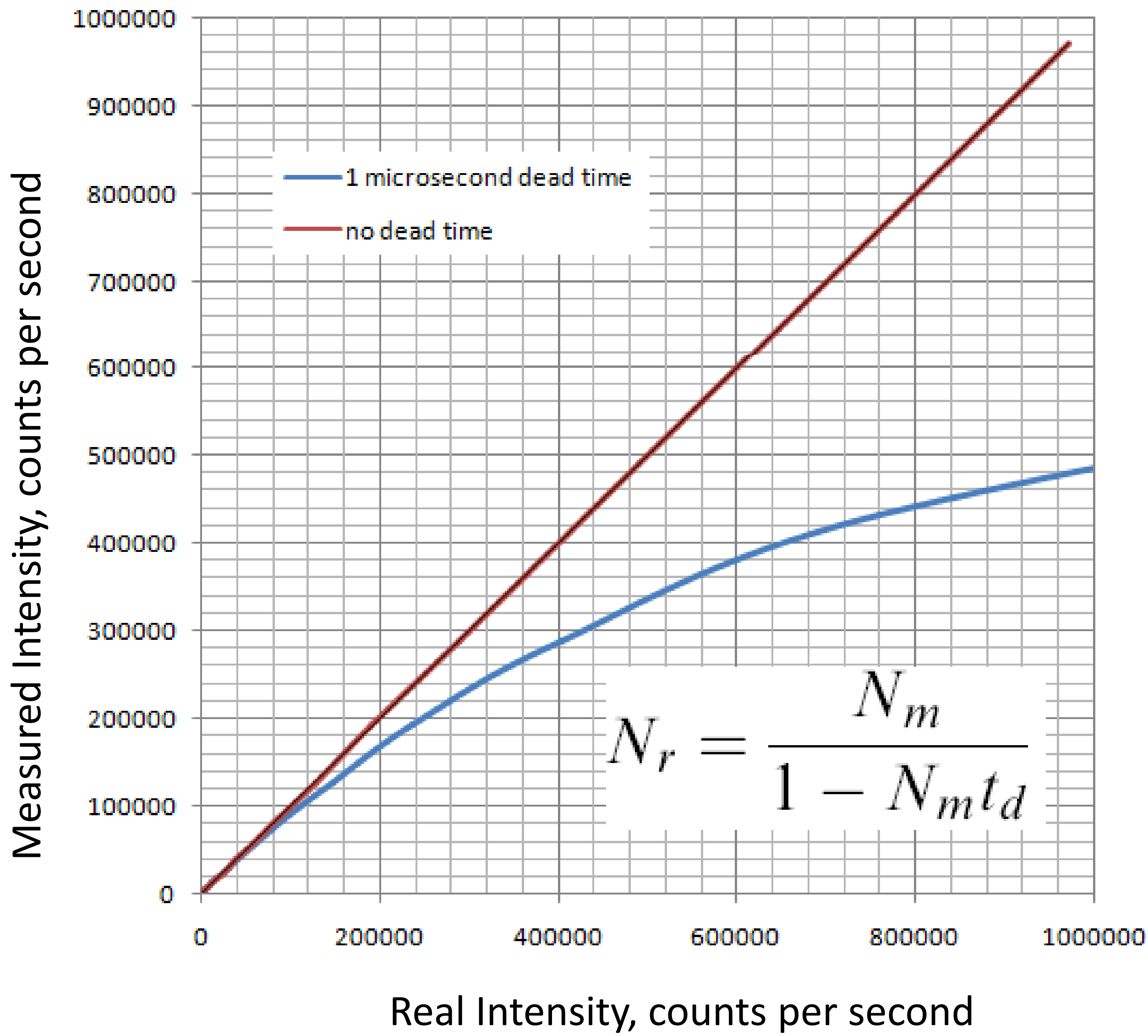
“Dead time” corrections

For a short time t_d after a pulse the detector is “dead” (unable to detect any new pulses). All pulses coming within dead time of each other will register as a single pulse.

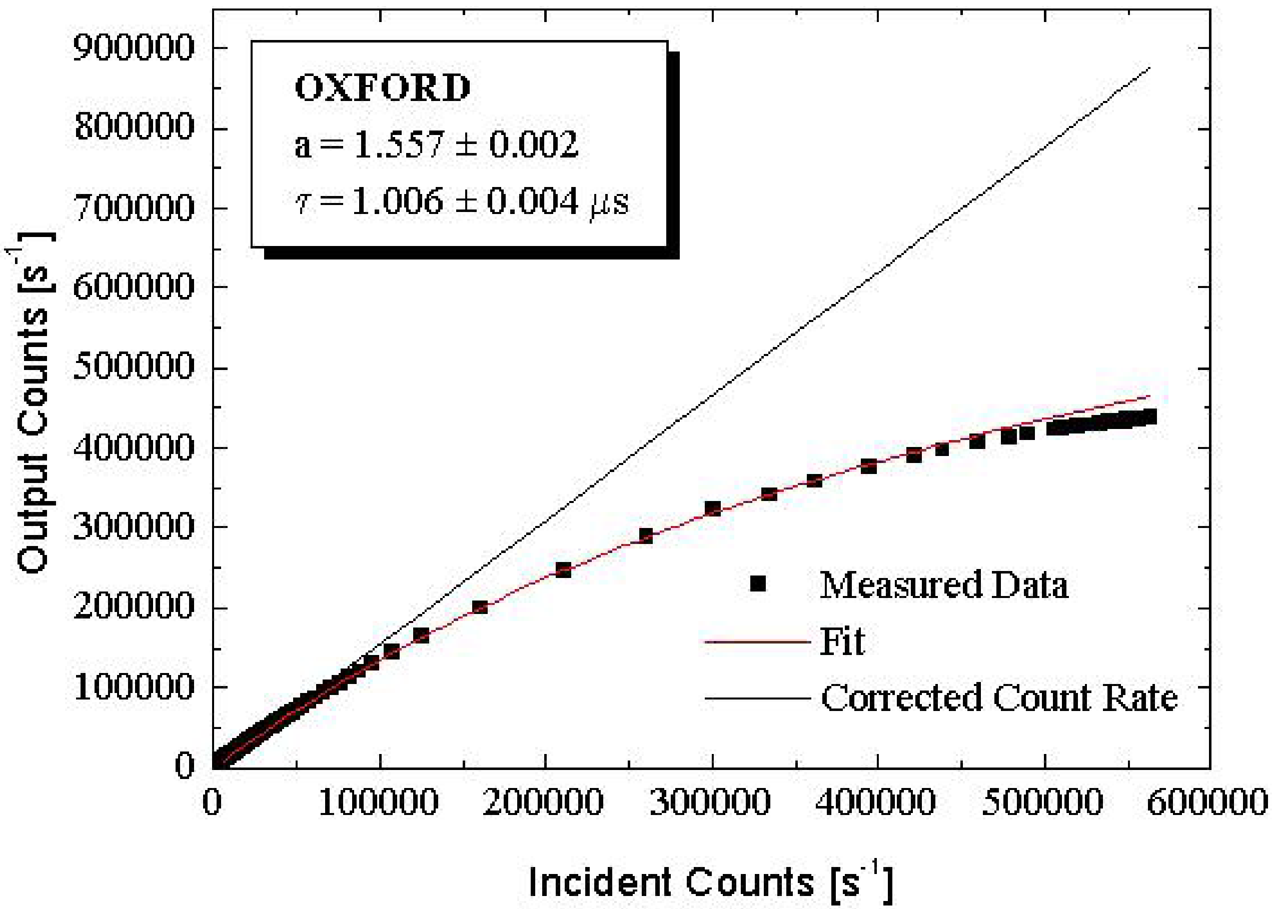
Typical values of t_d are 1-2 μs for scintillators, 0.2 μs for proportional counters.

$$N_r = \frac{N_m}{1 - N_m t_d}$$

Relationship between real and measured count rates (N_r and N_m), and dead time t_d.



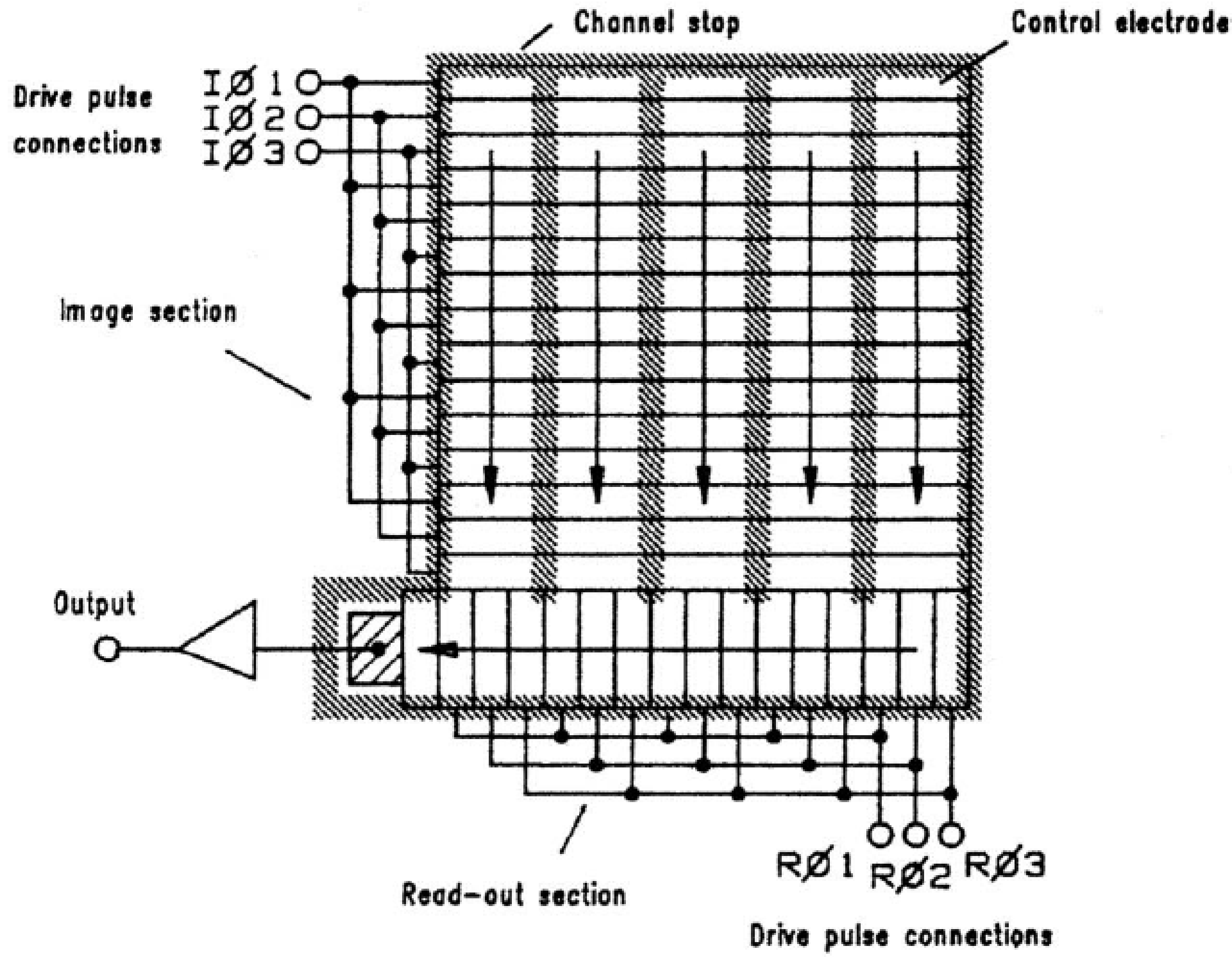
Relationship between real and measured count rates (N_r and N_m), and dead time t_d.



Typical dataset for Oxford Cyberstar X1000 Scintillator detector (from T. Gog et al. CMC NewsBlip 01-02-01).

Max attainable count rate ~400,000 cps. Above 350,000 is no longer described by deadtime formula

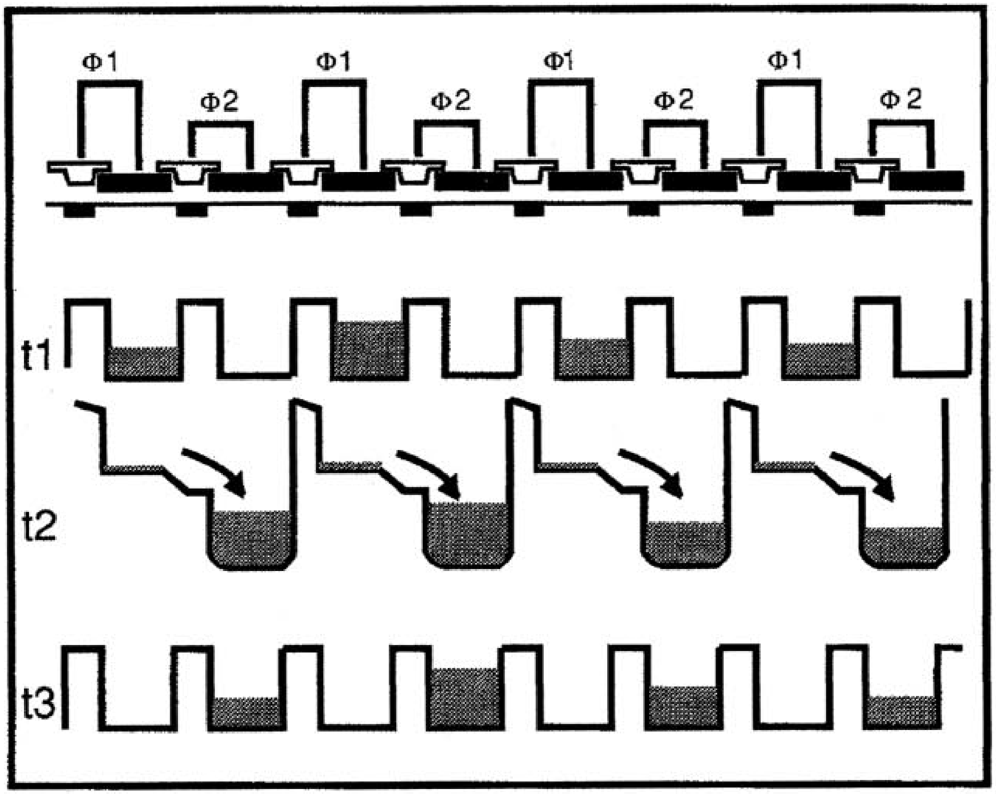
CCD Readout

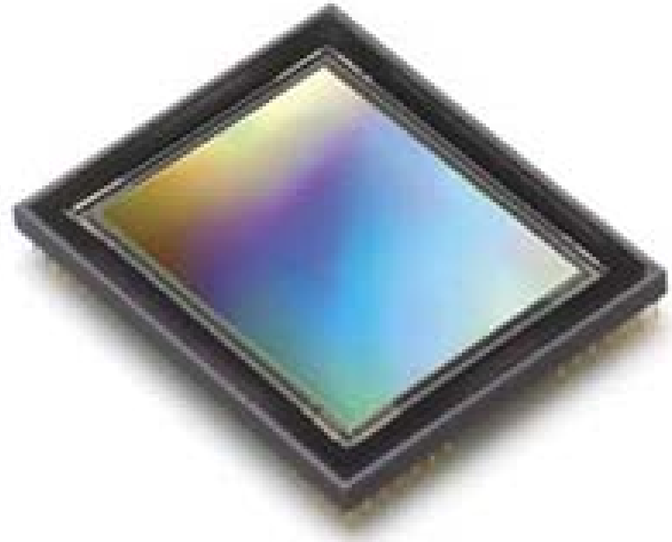


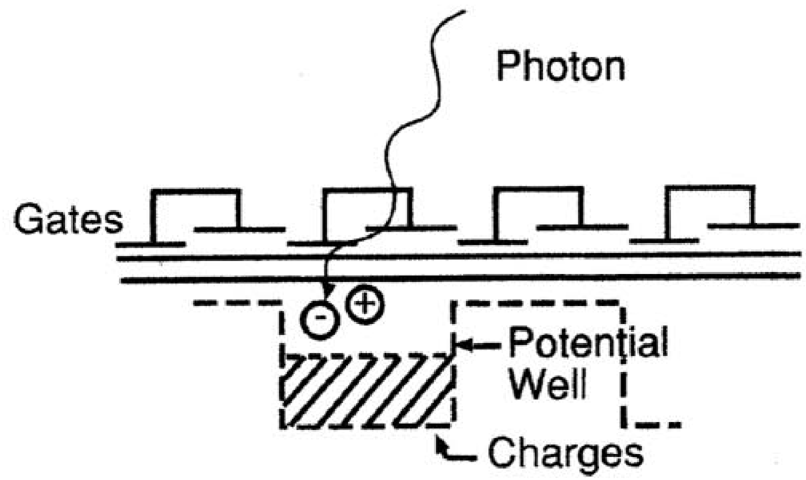
CCD Image Sensor

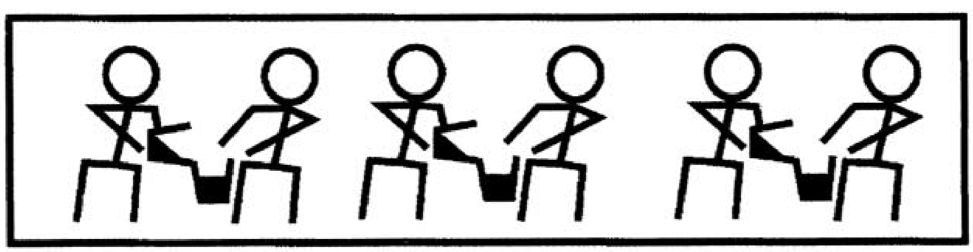
CCD

Charge Couple Device (CCD)









Data collection

Problem: intensity variations
by 10^8-10^{10} in reflectivity measurements
by 10^3-10^5 in GIDX, diffuse scattering

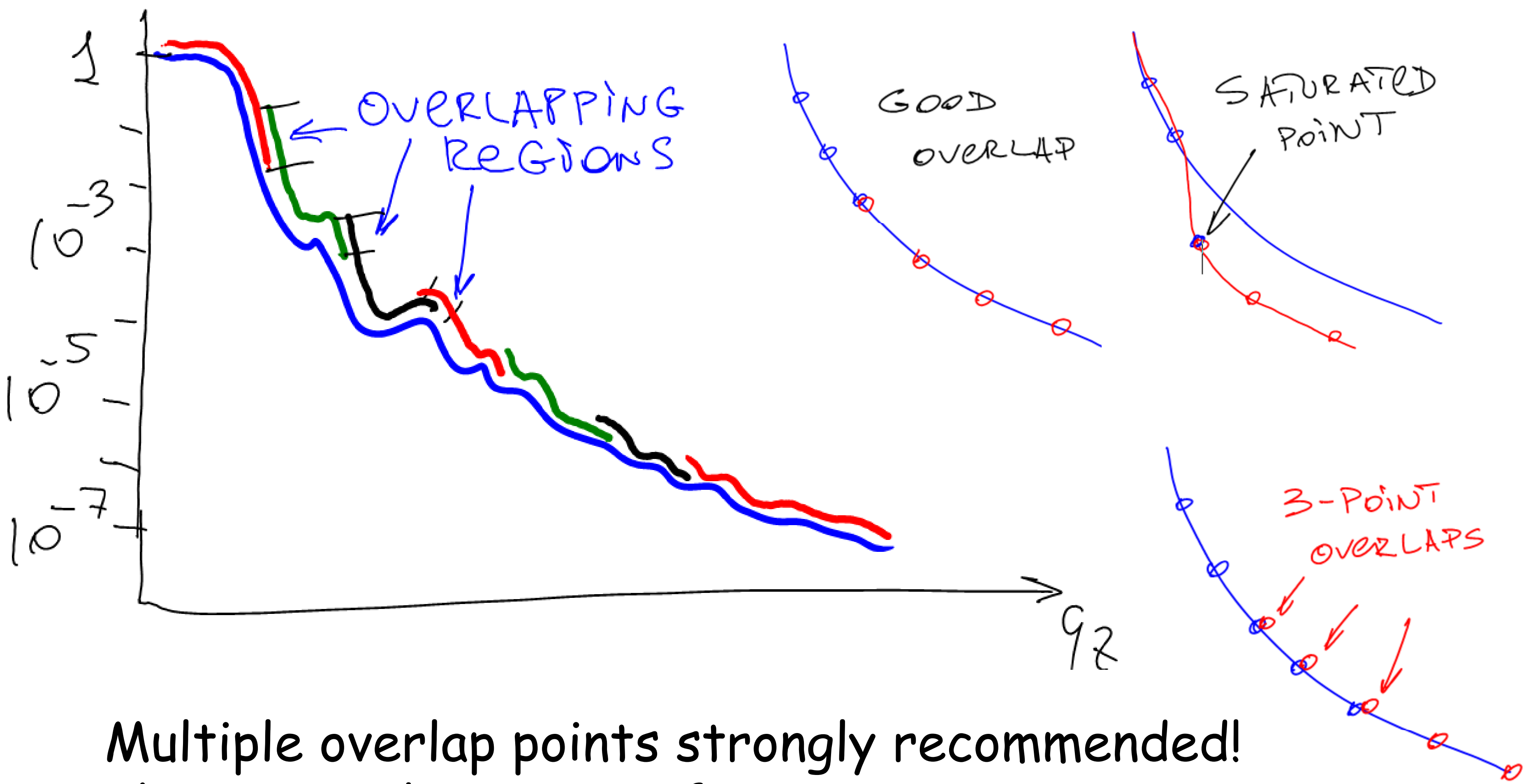
Solution: use attenuators for high intensity beams,
then "stitch" the data together

Attenuator – set of mounted films on a motorized linear translator

If 1 film attenuates by a factor of 2, 2 films attenuate by 4, 3 films by 8, etc. Actual attenuation per film depends on energy, material, thickness.

Use "binary" attenuator system.

Stitch together overlapping regions

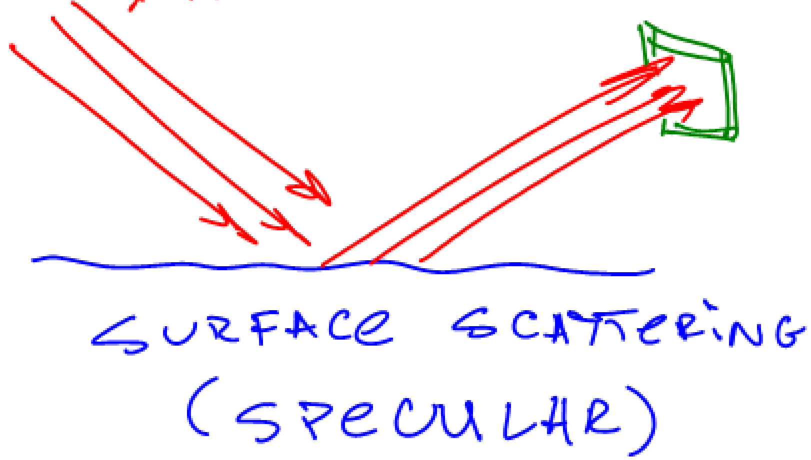


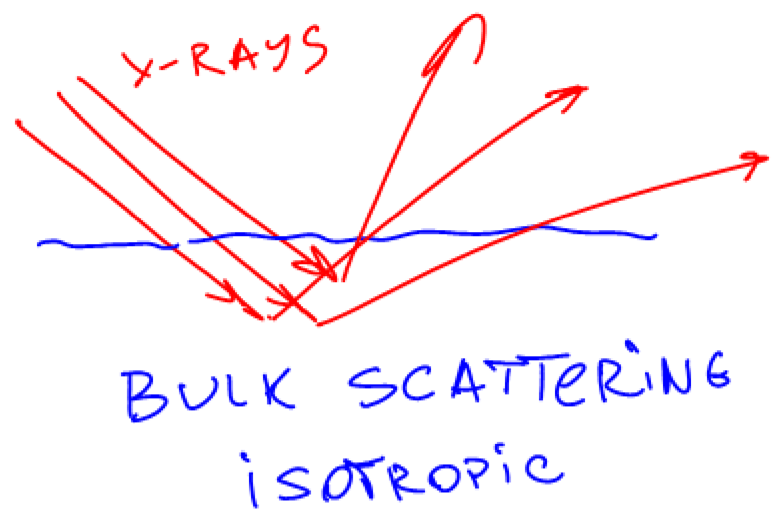
Multiple overlap points strongly recommended!
Change overlap regions for consistency

Data collection

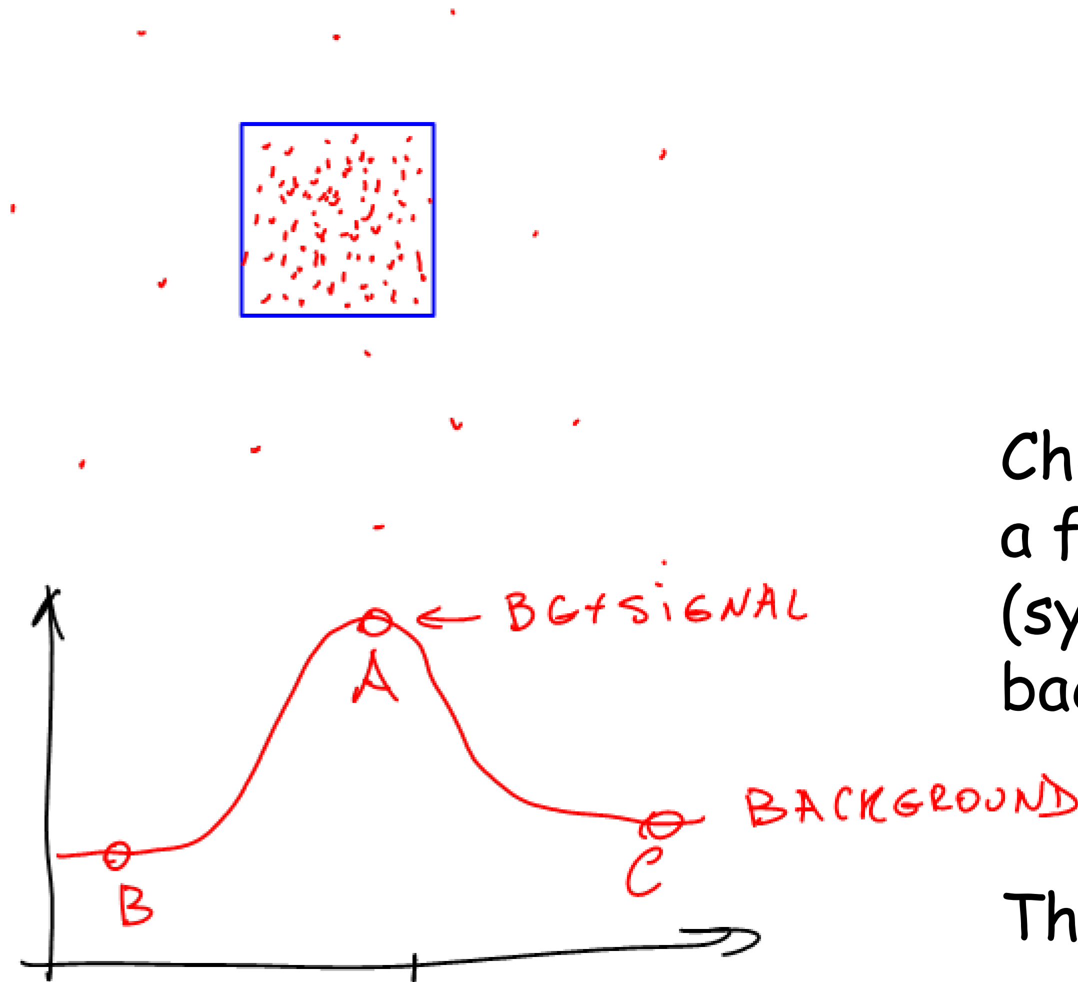
Problem: How to separate bulk contribution from surface scattering

Solution: Since bulk scattering is isotropic, subtract signal in off-specular geometry





Background subtraction:



$$\text{SIGNAL} = A - \frac{B+C}{2}$$

Check lineshape for a few locations (symmetry, alignment, background offset)

Then simply do 3-point measurement (saves time)

Data collection

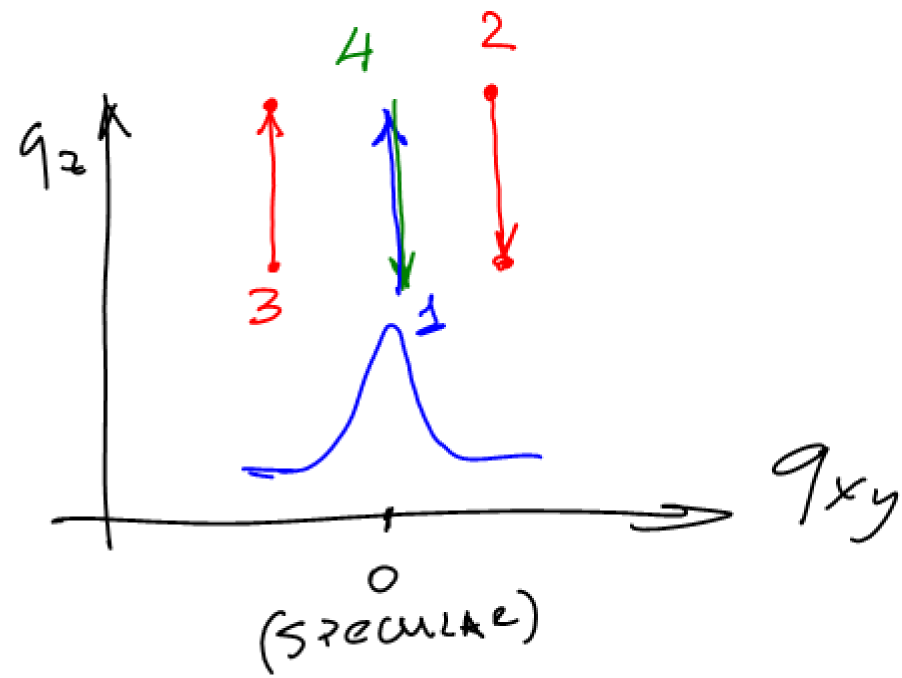
Problem: reproducibility of data

Solution: repeated measurements

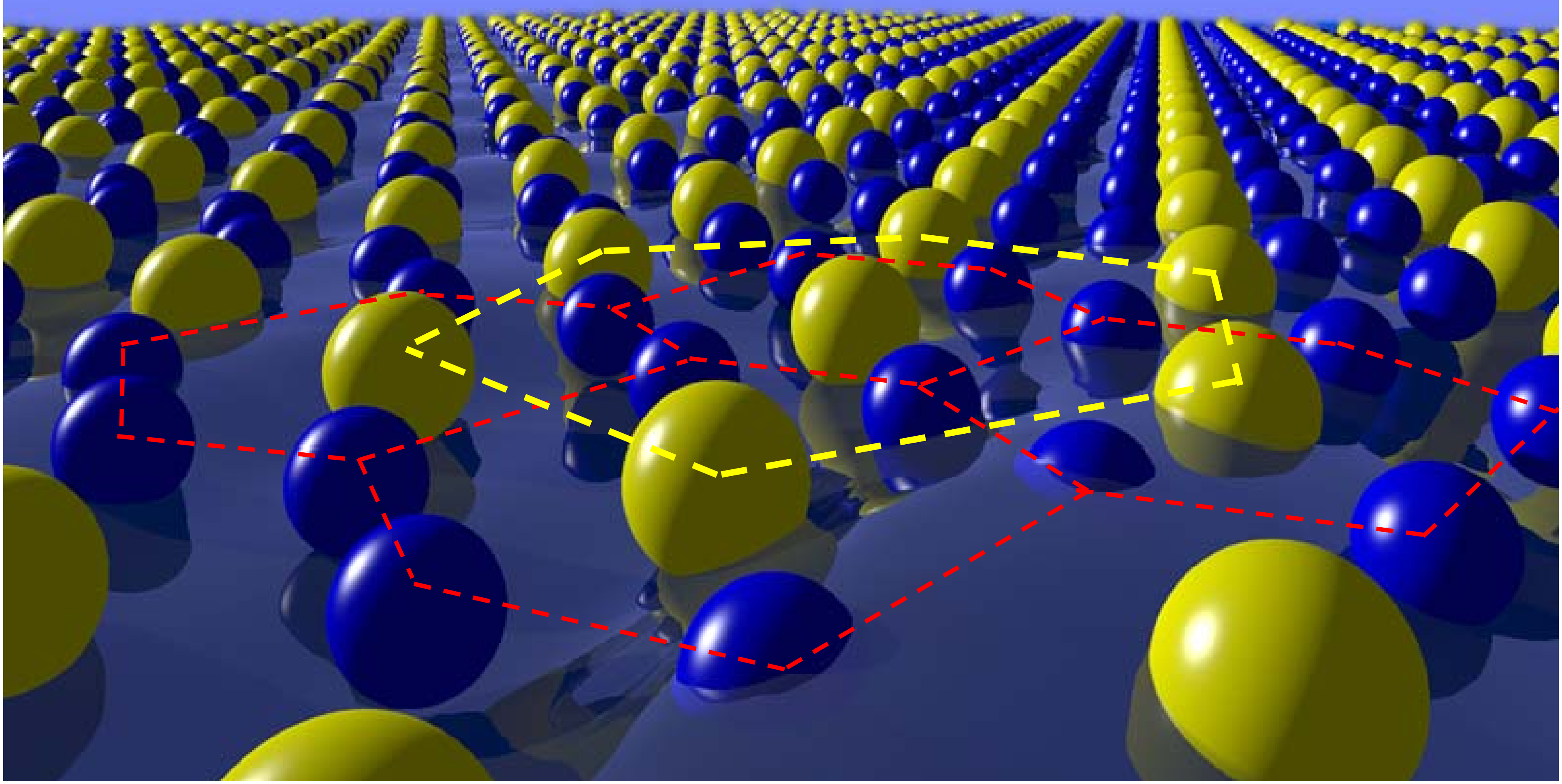
+ repeat everything "backwards"

Why?

- backlash effect
- beam damage
- sample history etc.

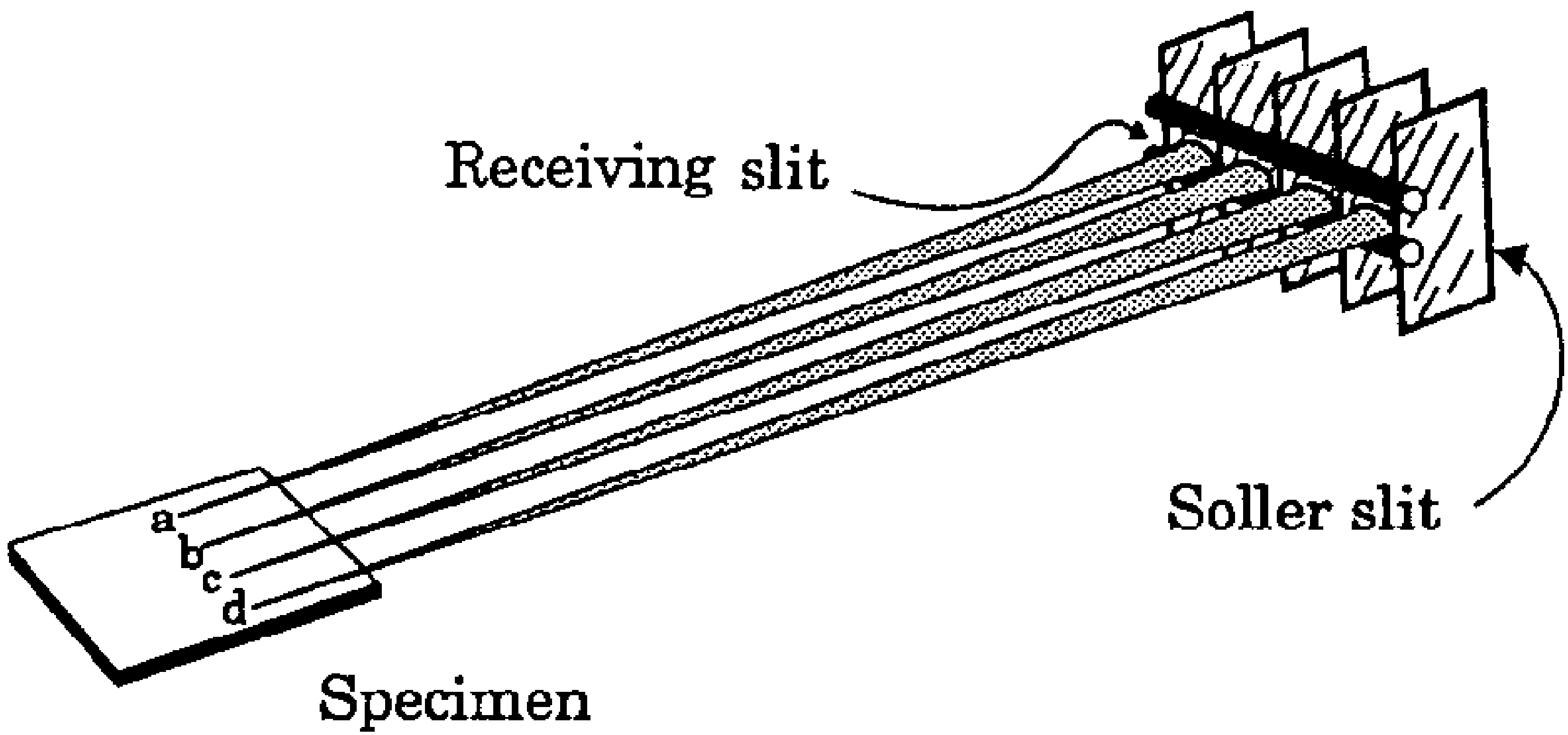


GIXD Example:
Surface Freezing in AuSi

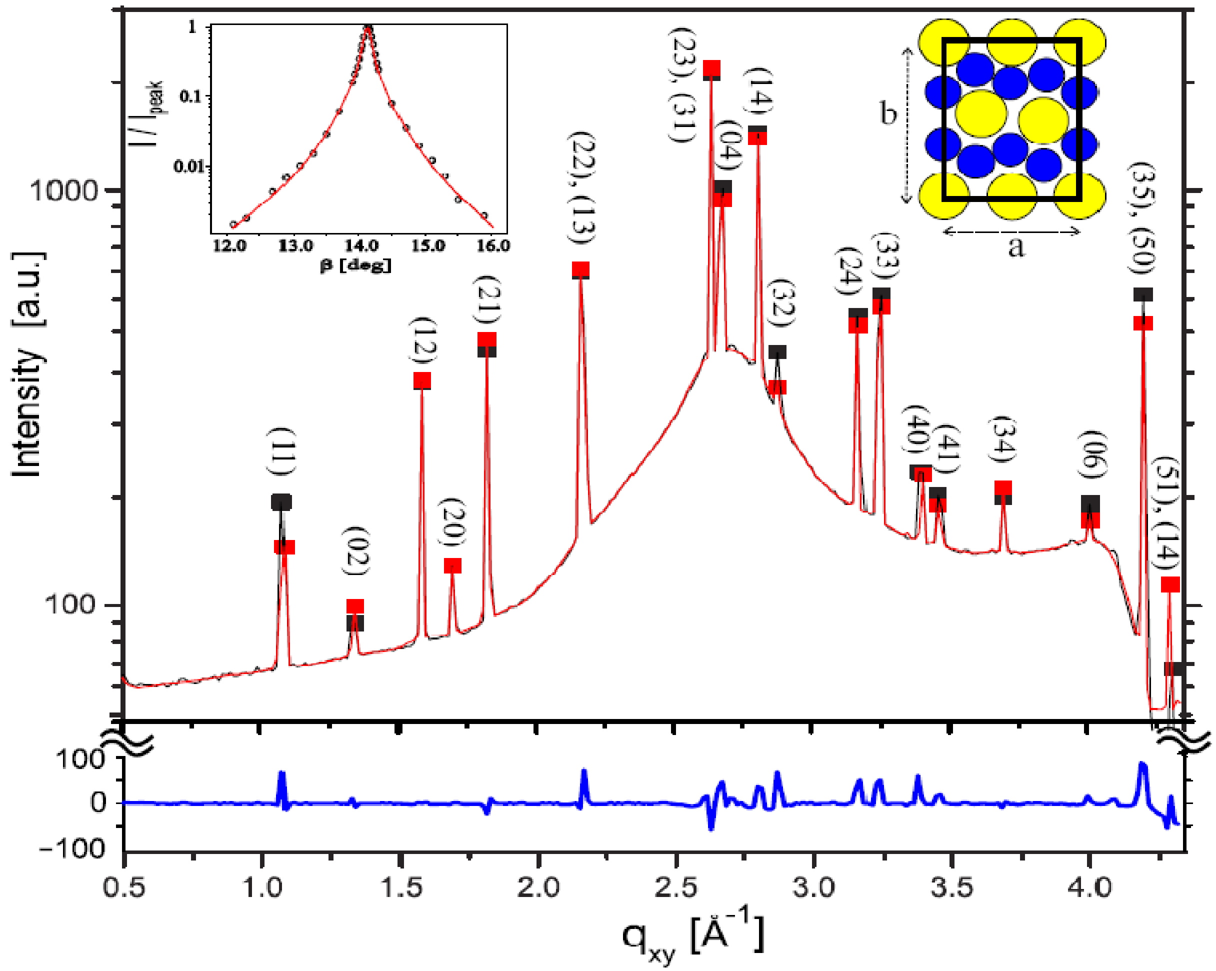


Soler Slits

resolution ~ 0.1 degree

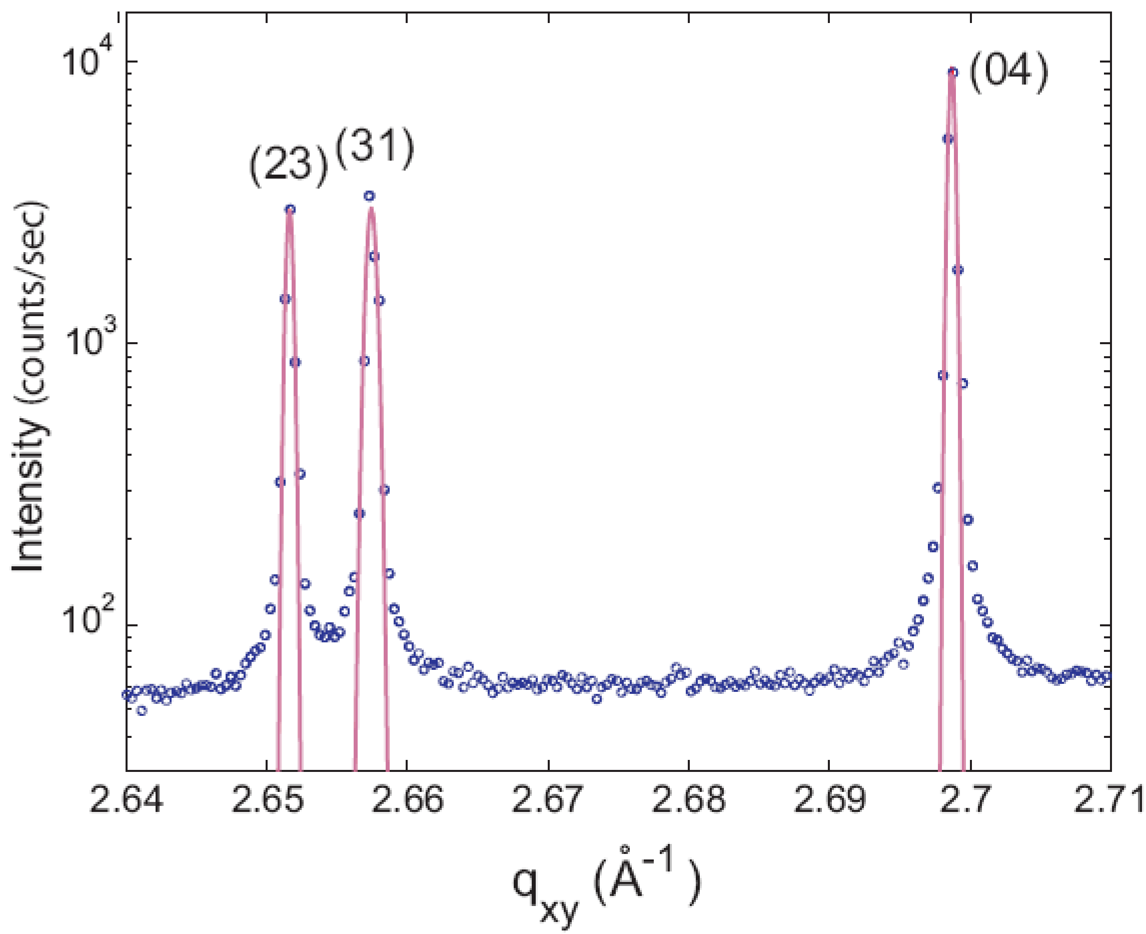


Grazing Incidence Diffraction w/ Soller Slits (penetration depth ~1.4nm, 5 atomic layers)



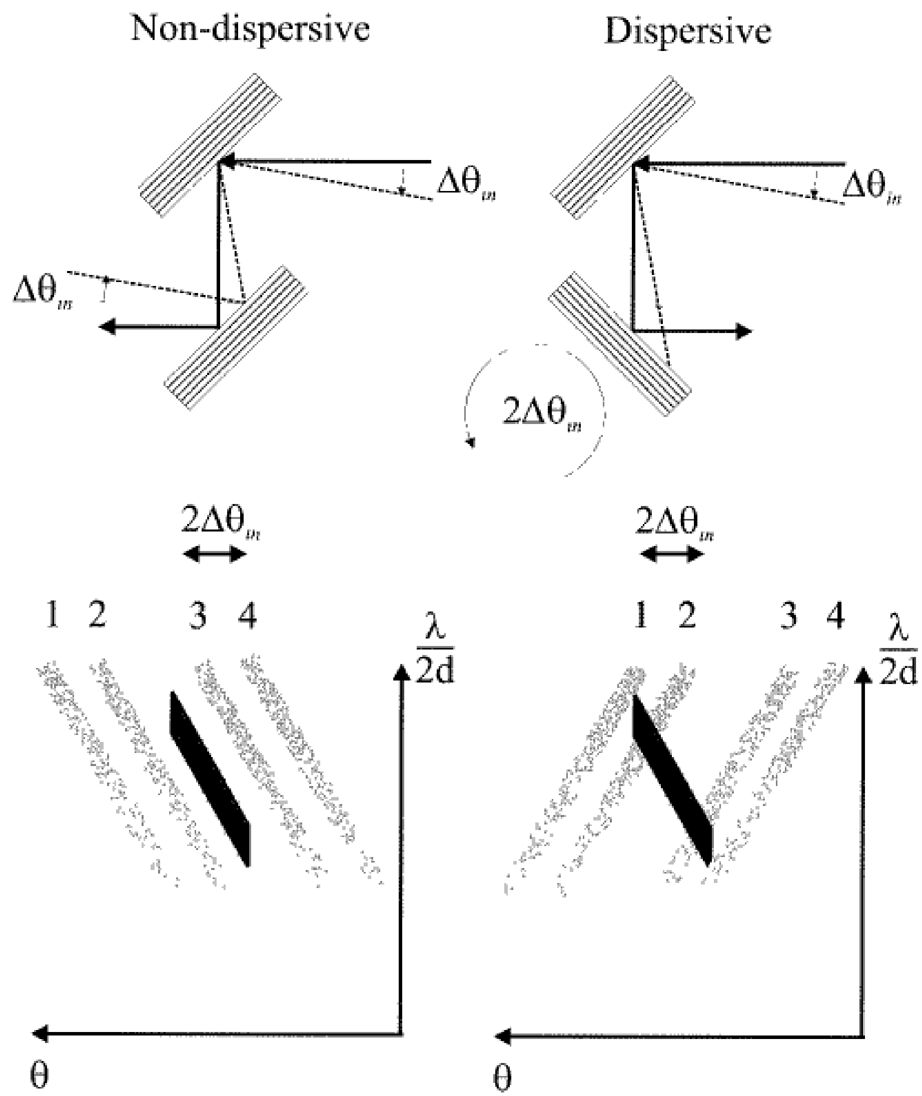
O.G. Shpyrko et al., Science 313, 77 (2006)

(23) and (31) peaks cannot be resolved with soller slits (previous slide) but can be resolved with crystal analyzer



O.G. Shpyrko et al., Phys. Rev. B (to appear) 2007

Double-bounce monochromator



Contrast mechanisms of chemical species:

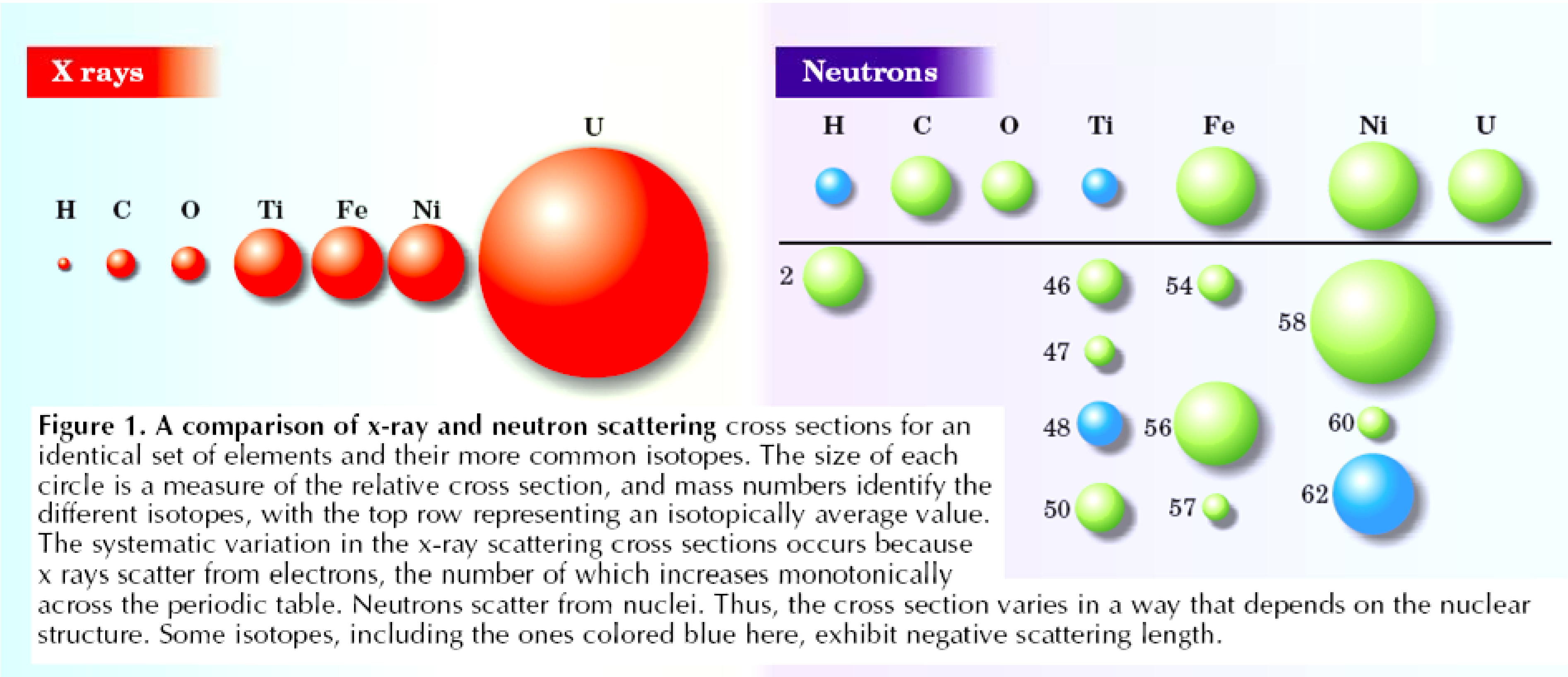
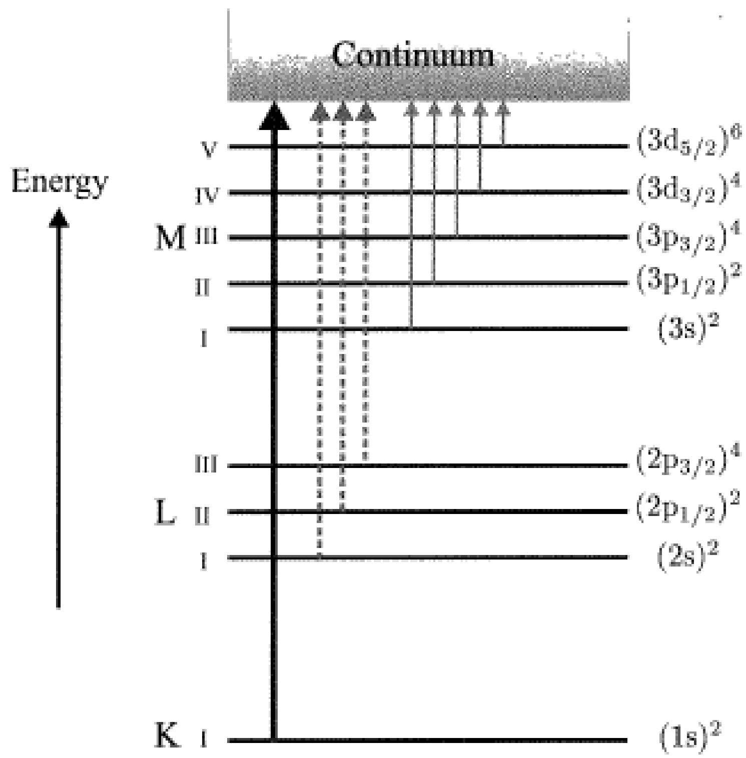
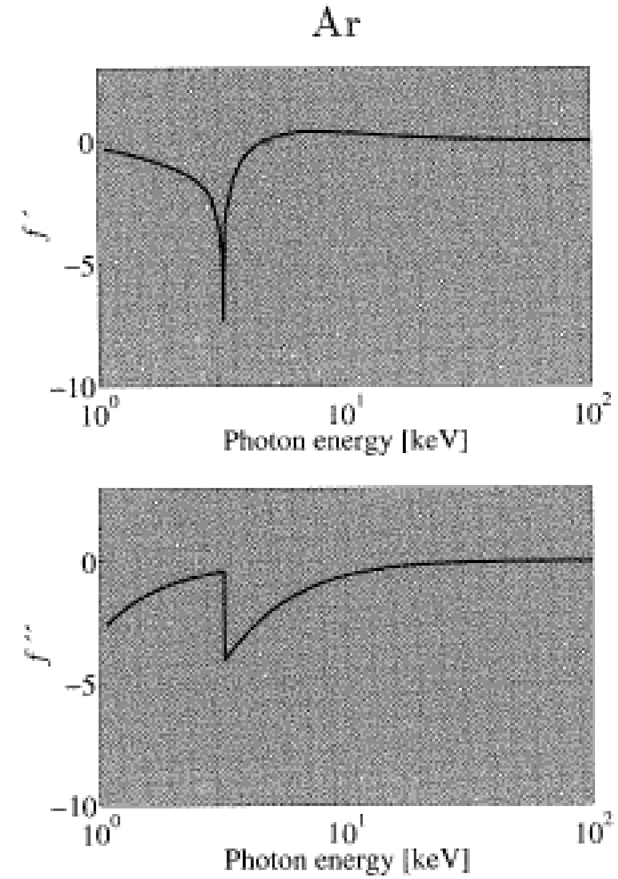


Figure 1. A comparison of x-ray and neutron scattering cross sections for an identical set of elements and their more common isotopes. The size of each circle is a measure of the relative cross section, and mass numbers identify the different isotopes, with the top row representing an isotopically average value. The systematic variation in the x-ray scattering cross sections occurs because x rays scatter from electrons, the number of which increases monotonically across the periodic table. Neutrons scatter from nuclei. Thus, the cross section varies in a way that depends on the nuclear structure. Some isotopes, including the ones colored blue here, exhibit negative scattering length.

Thomas E. Mason, Physics Today 59, 44-49 (2006)

Other contrast mechanisms: absorption edges





Basic idea: when tuned to resonant edge of an element, the element “loses” a few electrons. Near K-edge Ar effective electron density varies from Z+f’ of 18 to 11.

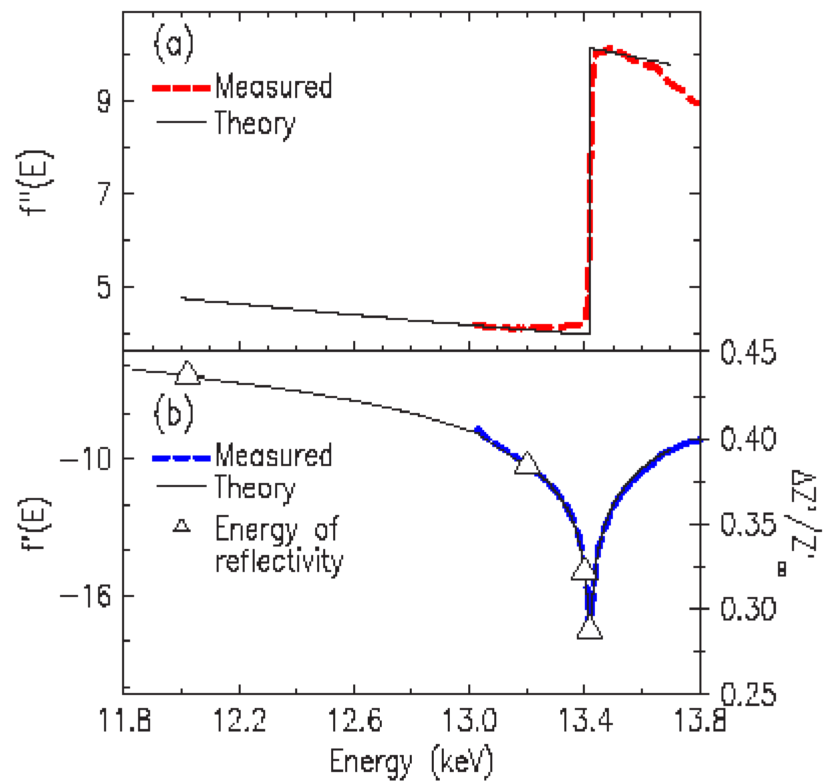
Energy changes near adsorption edge serve as “tuning knob” to vary contrast between various species

Example: Gibbs Adsorption in liquid BiSn

Tuning Knob:
X-ray Energy Resonance

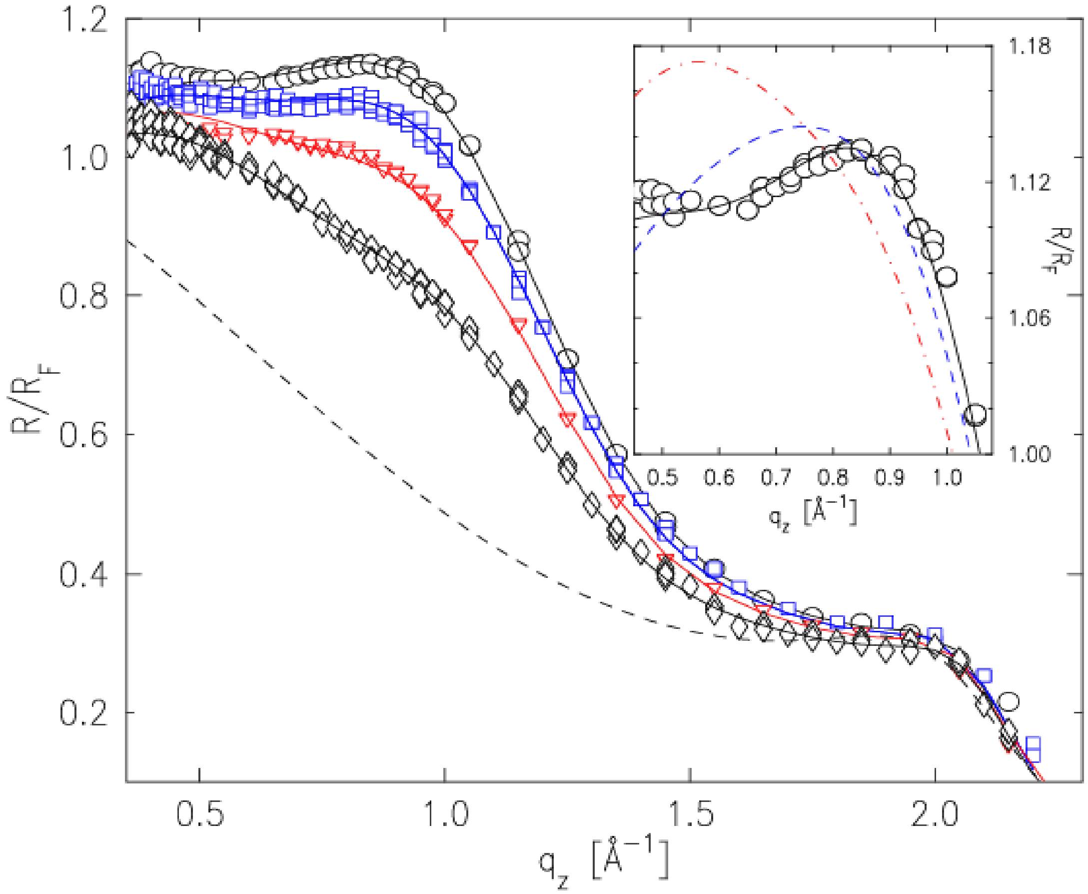
When scanned through Bi L3-edge, Bi-Sn electron contrast gets reduced by ~30% (!)

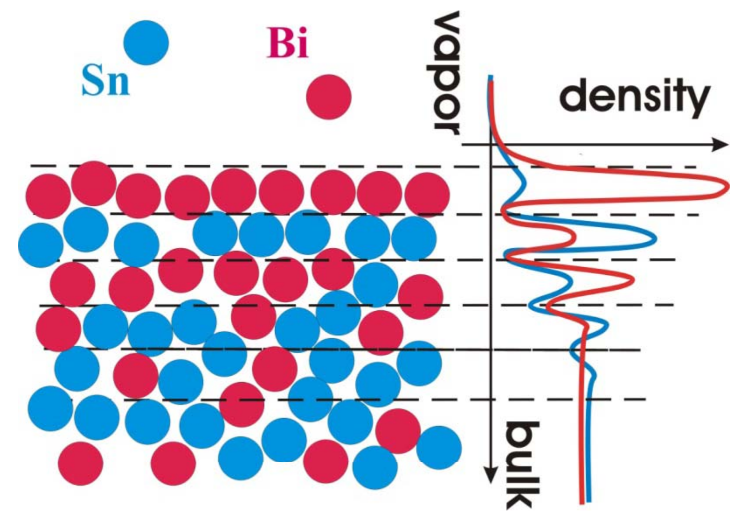
Comparing reflectivity on-edge and off-edge will tell you where Bi atoms are hiding



O.G. Shpyrko et al., Phys. Rev. Lett. **95**, 106103 (2005)

BiSn: Miscible (eutectic) Binary alloy





O.G. Shpyrko et al., Phys. Rev. Lett. **95**, 106103 (2005)

Think about other contrast mechanisms, new (and old) techniques

- Standing Wave
- Surface Extended Edge Adsorption (SEXAFS)
- Fluorescence (e.g. in grazing incidence)
- X-ray Microscopy, Phase Contrast Imaging
- Use of X-ray coherence – dynamics with X-ray Photon Correlation Spectroscopy (XPCS)
- New detectors, new approaches in data analysis (model-independent density profiles)
- Anything possible in visible light optics is probably doable with x-rays!

Other useful tricks and tips for aspiring young x-ray ninjas

$$\lambda\,[\text{Å}] = \frac{hc}{\mathcal{E}} = \frac{12.398}{\mathcal{E}\,[\text{keV}]}$$

Note that 12.398 is almost 4π
2k in $Å^{-1}$ = Energy in keV

To convert from degrees to radians divide by 60.
sin(x)=x for small x<<1

Example: Energy is 6.5 keV, $\alpha=\beta$=6 deg.
What is q_z ?
q_z=2k sin(α)=6.5/10=0.65 $Å^{-1}$

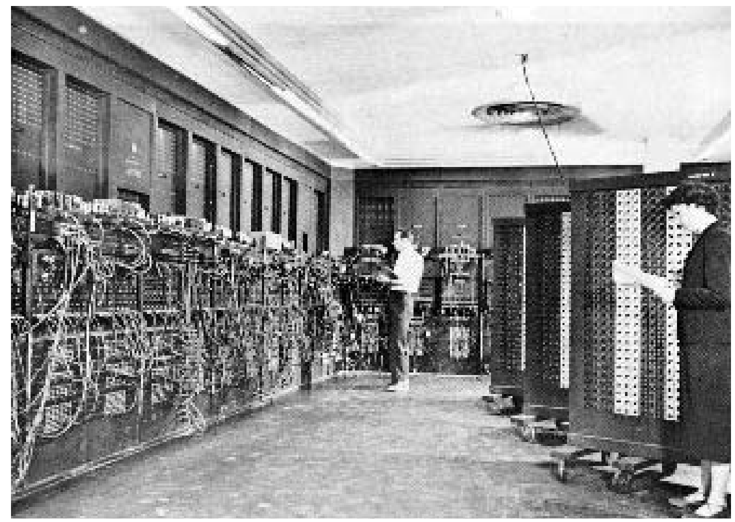
Learning geometry of scattering (names of motors, angles, distances) will help you do quick "back of the envelope" calculations

Example: How far to move detector in q_x to avoid specular reflection (for background subtraction?)

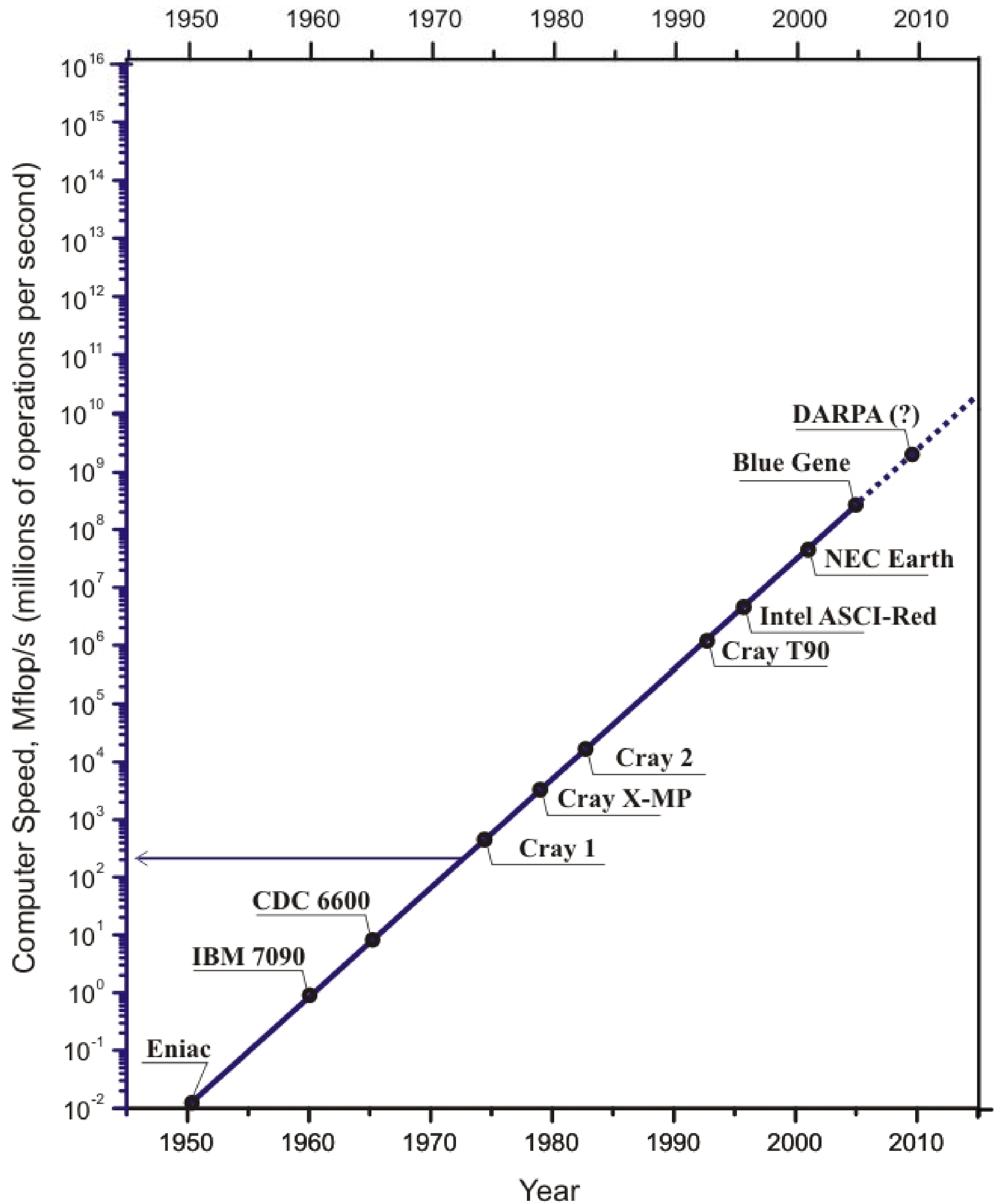
Back of the envelope: 4mm horizontal slits, 630mm from the sample
2θ ~ 1/300 or 3mrad (need to move only half of the width)
q_x=k sin(2θ)=6.5 /300/2=0.01 $Å^{-1}$ (approx.)

First Synchrotron Light:
General Electric, 1947

First Computer:
ENIAC, 1943



12 orders
of magnitude
In 6 decades



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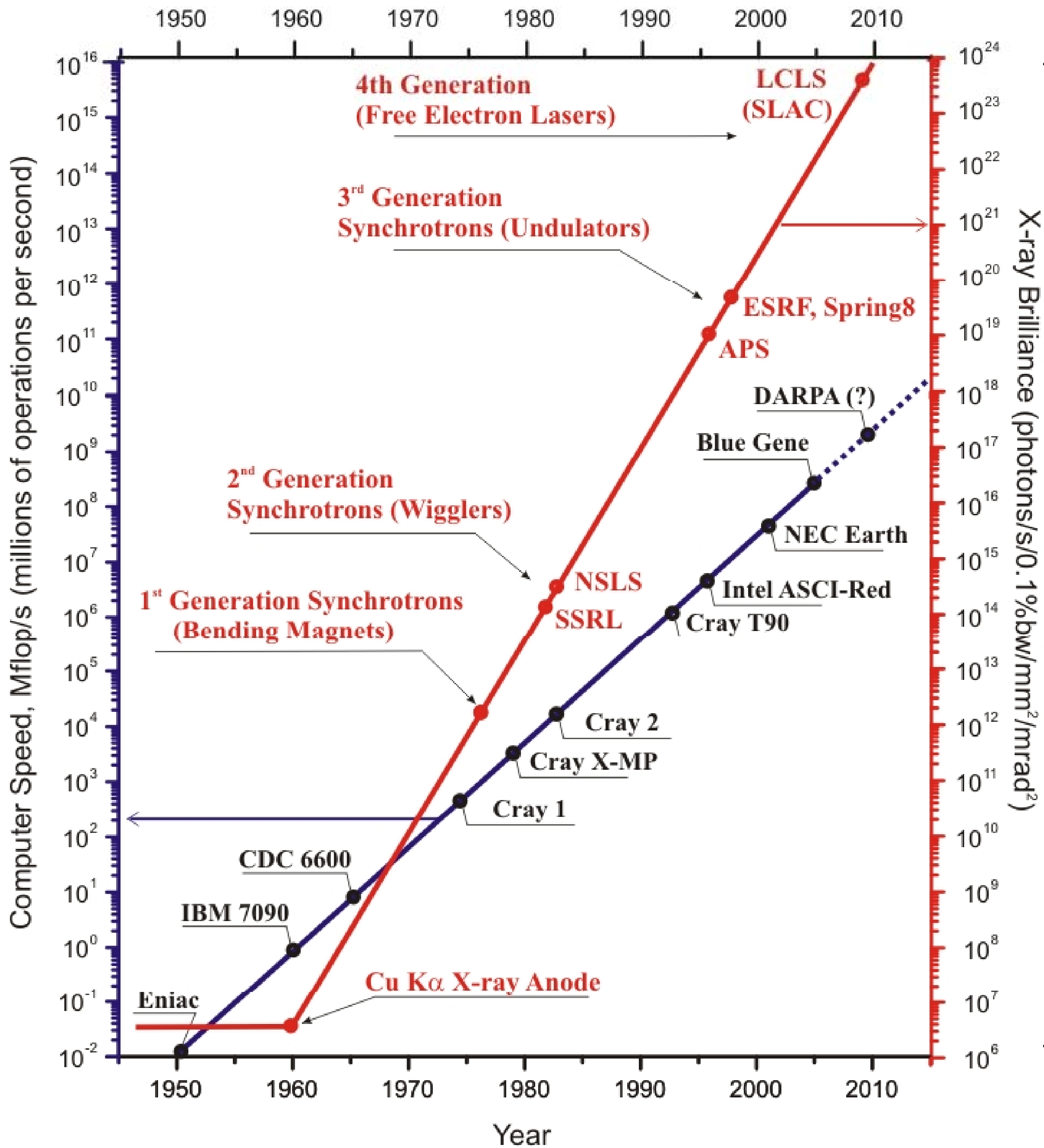

18 orders
of magnitude
In 5 decades!

Further reading material

General information on detectors:

Handbook on Synchrotron Radiation
(ed. D. E. Eastman and Y. Farge)

X-ray Position sensitive detectors:

U. Arndt J. Appl. Cryst. 19, 145-163 (1986)
A. Gabriel, Rev. Sci. Inst. 48, 1303 (1977)

CCD:

S. M. Gruner et al., Rev. Sci. Inst. 73, 2815 (2002)

Synchrotron Radiation:

H. Winick et al., Ann. Rev. Nucl. Part. Sci..28, 33 (1978)