# EE 213, Microscopic Nanocharacterization of Materials
## Lecture 7. *Xray Microanalysis (cont.)*

Class website: https://ee213-winter16-01.courses.soe.ucsc.edu

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# EE213 Paper Notes

- See IEEE.jour for formatting notes. On class web site.
- Paper should be about 10 pages long including figures.
- Paper should include a 1 paragraph abstract
- Paper should have at least 10 references.
- For each reference, either a summary or the abstract of that reference attached as an Appendix to the paper.

EE213. W16
Homework 1
Due: 2.11.16
Max Score = 100
Note: if you take data from the literature, you need to reference the article.

1. (50 pts.) Consider a 150nm thick garnet sample irradiated by a focused 10nm diameter electron beam of 100KeV energy. The electron beam is normal to the sample surface and the point where the beam strikes the garnet is 20mm from the front of the detector which is the same Si(Li) XRay detector as in problem2. The detector axis is 45 degrees to the sample surface. From the table below, calculate the relative mass fraction ratios of the elements indicated. The counts represent the integrated counts under the Ka XRay peak after subtracting off the bremstrahlung XRay background. Note: you need to decide if you are able to use the thin film approximation.

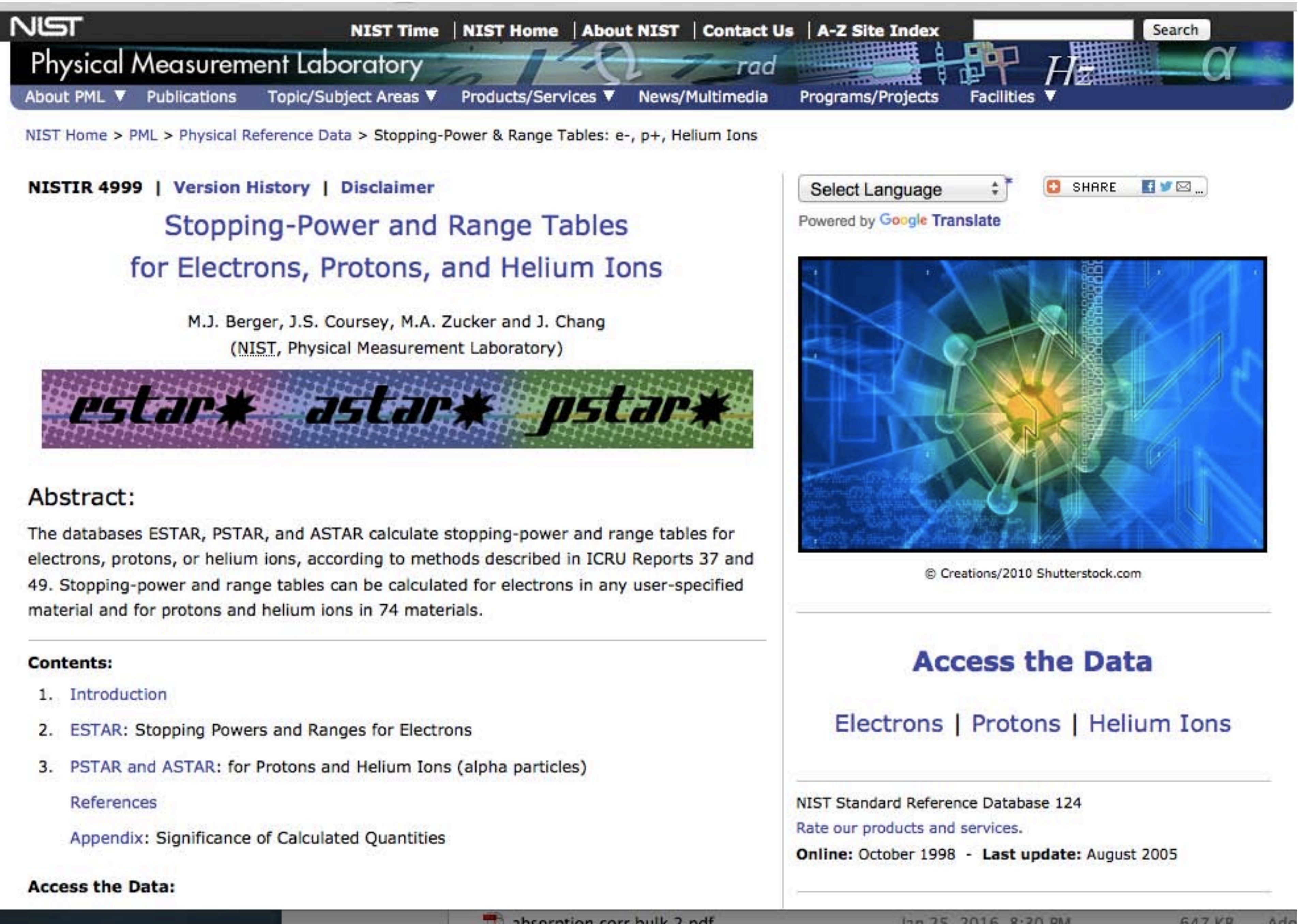
| Element | counts |
|---|---|
| Mg | 7600 |
| Al | 9,100 |
| Si | 20.600 |
| Ca | 4,400 |
| Ti | 900 |
| Cr | 1,200 |
| Fe | 8,100 |

2. (50 pts) A manufacturer of a Si(Li) XRay detector claims that the detector is more than 90% efficient at detecting any K$\alpha$ XRay from Na to Nd. The specification of the detector is shown below:

3mm thick active region
10 micron thick Be vacuum window
35 nm thick Au front contact layer (ie, facing the XRays)
300nm thick Si dead layer on the front surface
20 square mm active area

A) (25 pts) Are the claims true? Explain.
B) (25 pts) Could this detector be used to detect oxygen K$\alpha$ Xrays? Explain.

http://www.nist.gov/pml/data/star/



NIST Home > PML > Physical Reference Data > Stopping-Power & Range Tables: e-, p+, Helium Ions

**NISTIR 4999** | **Version History** | **Disclaimer**

## Stopping-Power and Range Tables for Electrons, Protons, and Helium Ions

M.J. Berger, J.S. Coursey, M.A. Zucker and J. Chang
(NIST, Physical Measurement Laboratory)

### Abstract:

The databases ESTAR, PSTAR, and ASTAR calculate stopping-power and range tables for electrons, protons, or helium ions, according to methods described in ICRU Reports 37 and 49. Stopping-power and range tables can be calculated for electrons in any user-specified material and for protons and helium ions in 74 materials.

**Contents:**

1. Introduction
2. ESTAR: Stopping Powers and Ranges for Electrons
3. PSTAR and ASTAR: for Protons and Helium Ions (alpha particles)

References

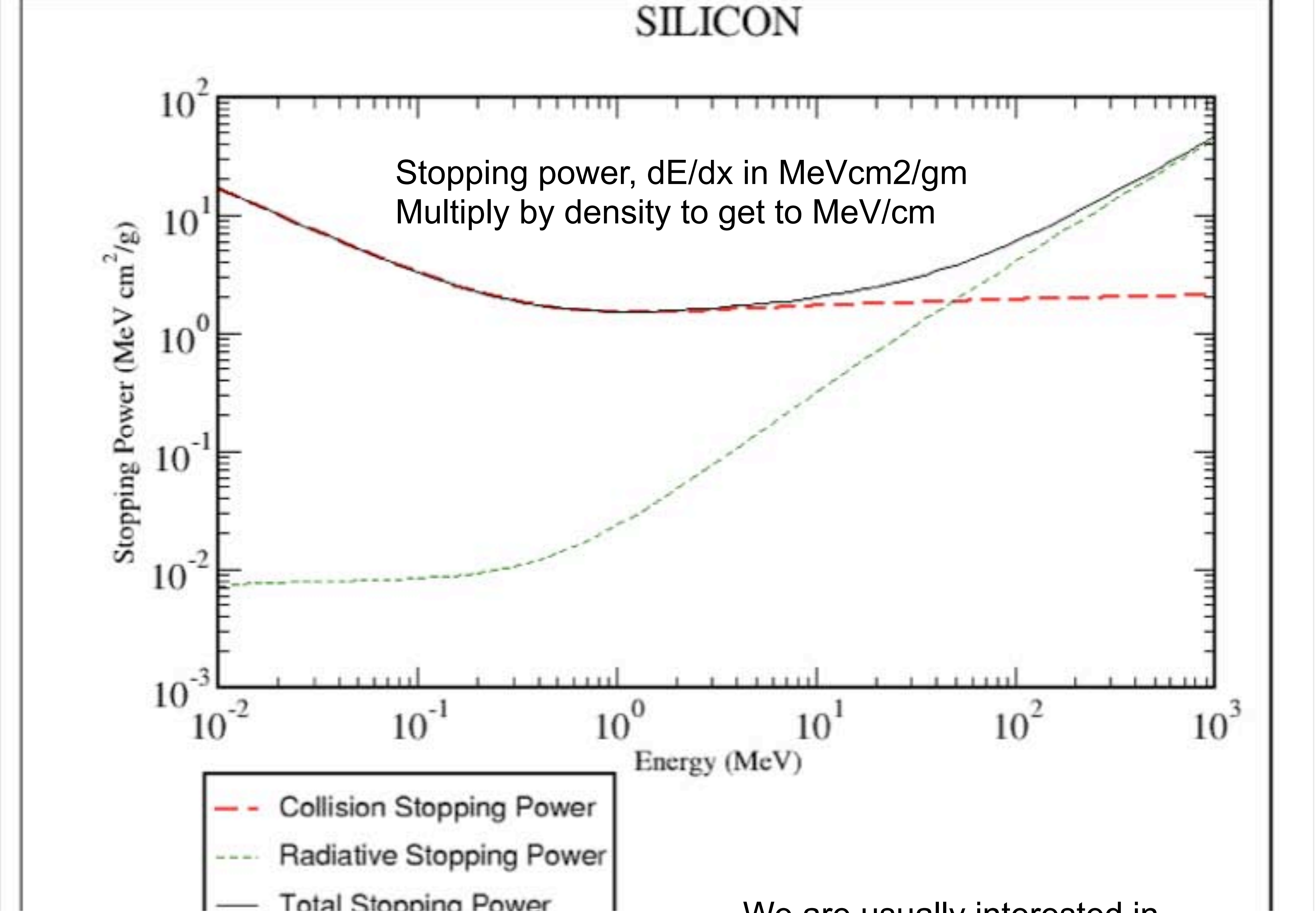
Appendix: Significance of Calculated Quantities

**Access the Data:**

## Access the Data

Electrons | Protons | Helium Ions

NIST Standard Reference Database 124
Rate our products and services.
**Online:** October 1998 - **Last update:** August 2005

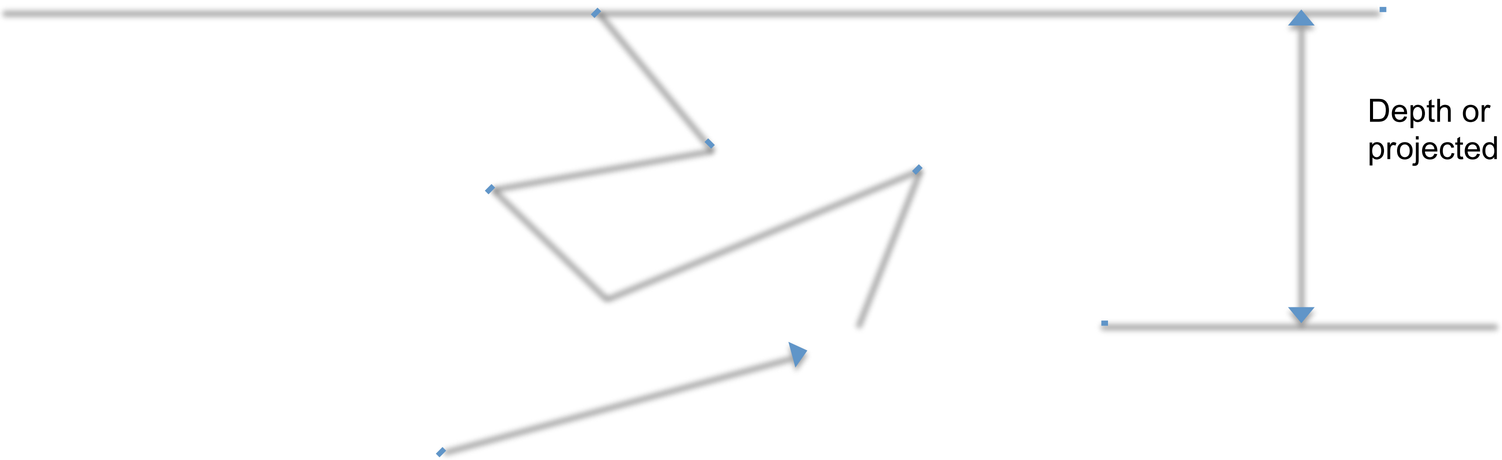


Stopping power, dE/dx in MeVcm2/gm
Multiply by density to get to MeV/cm

We are usually interested in
region less than 1 MeV

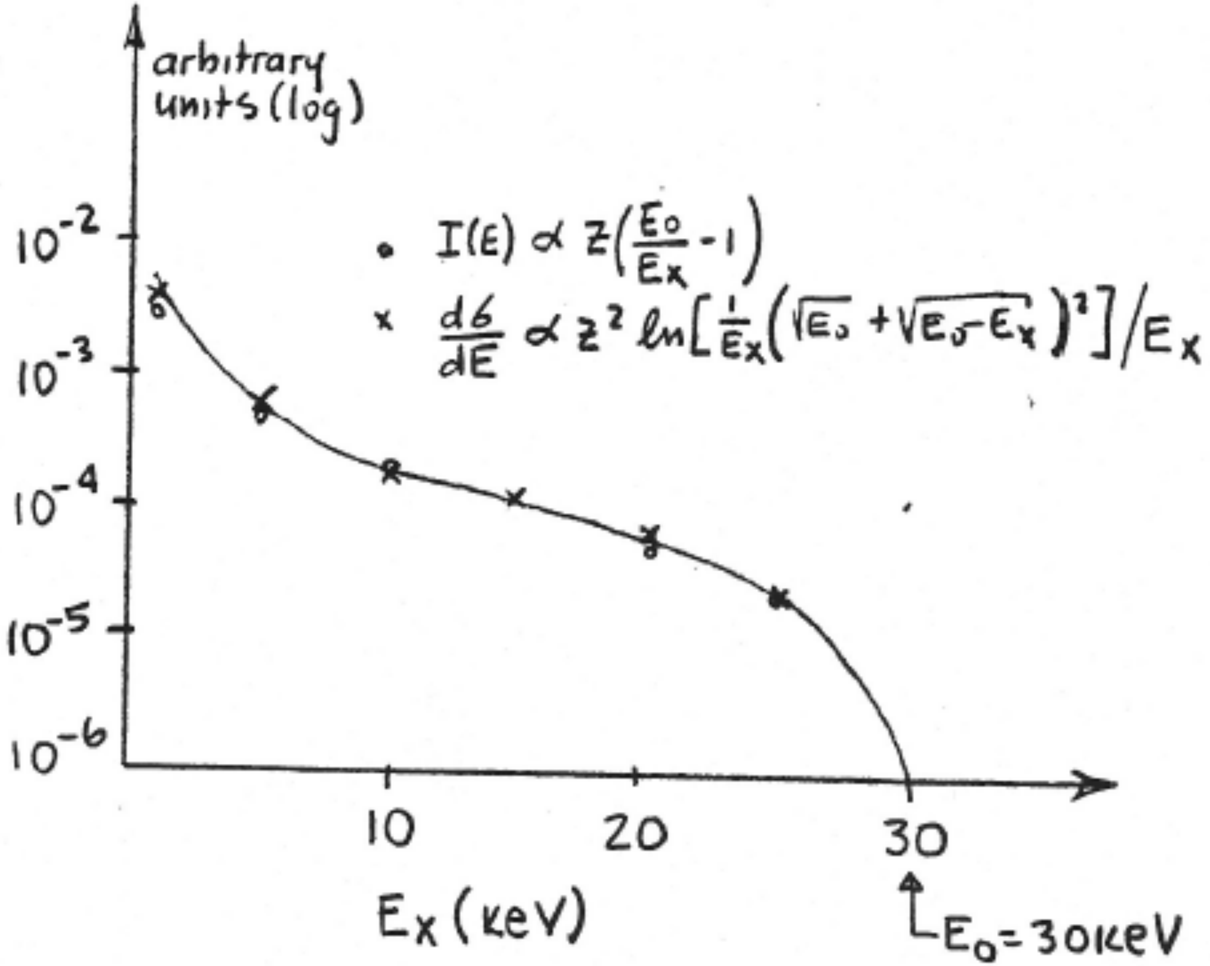
# Stopping power and range

- Note that $dE/dx = \langle E \rangle_{avg} / \Lambda_{in}$ approx.
- $\langle E \rangle_{avg} = 13.5 Z^{1/2}$ eV



Depth or projected range

Total path = range or Bethe range



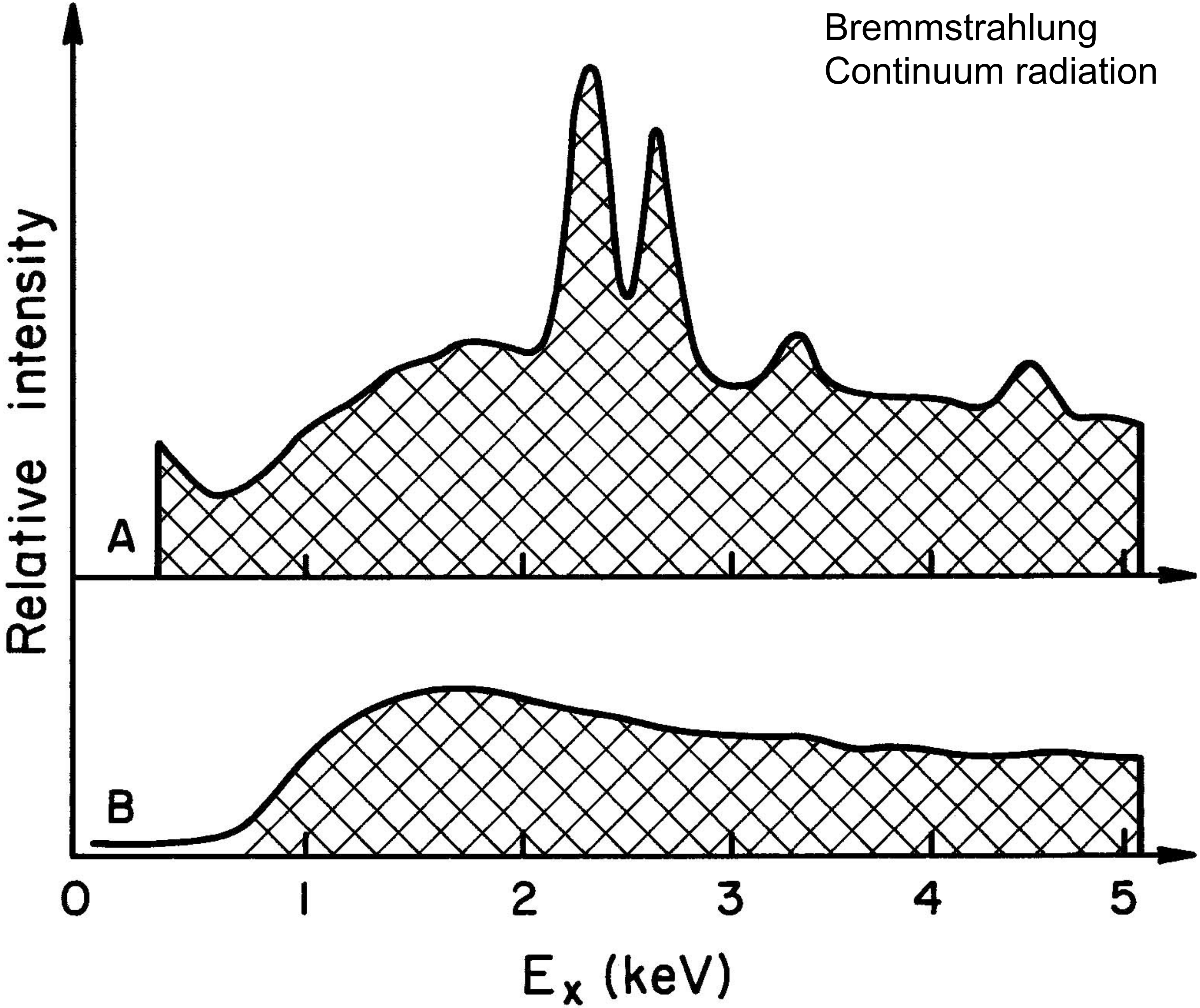
results normalized at 15 keV

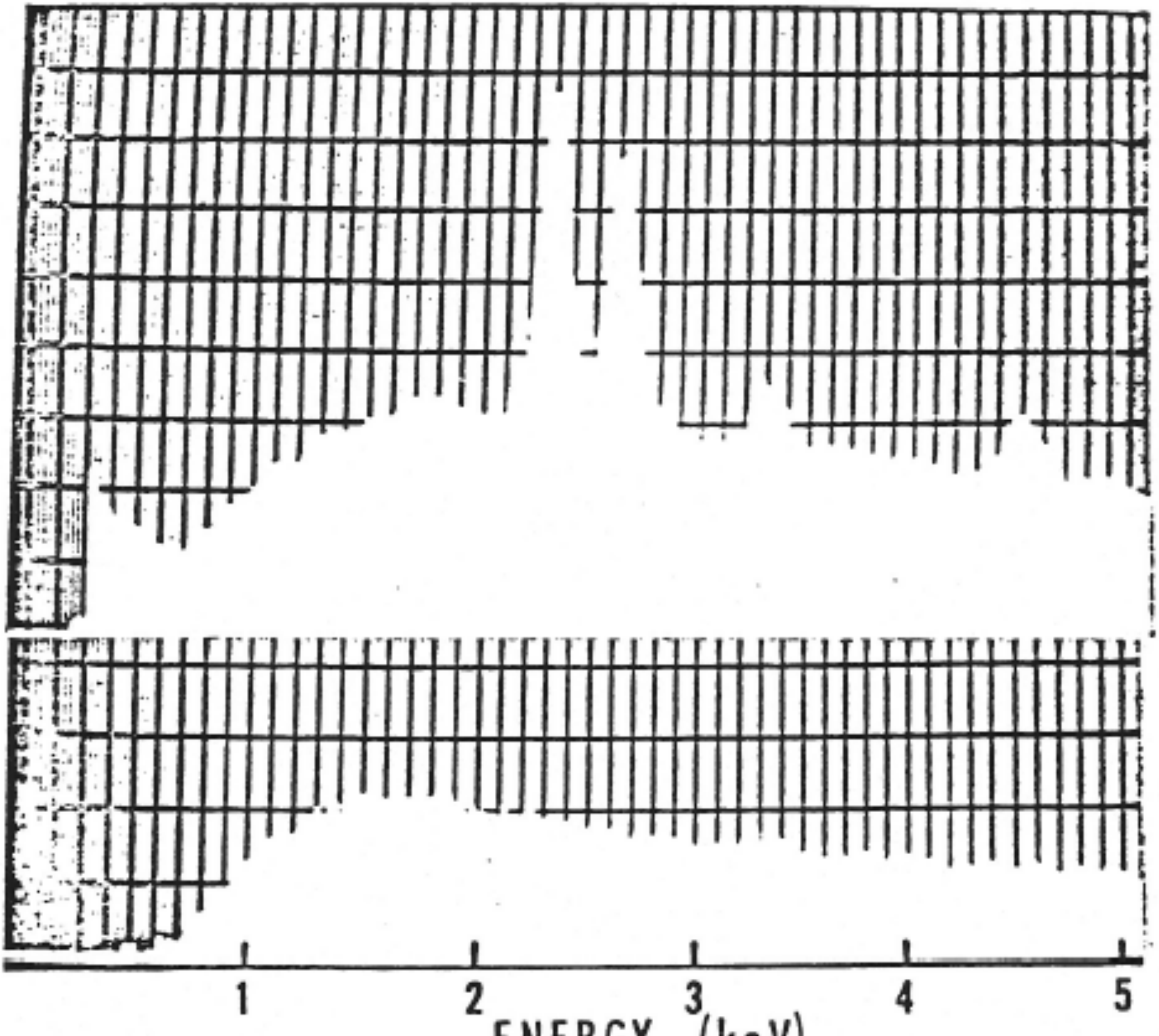
$I(E)$ → solid target emission (Kramers, 1923)

$\frac{d\sigma}{dE}$ → semi-classical electron slowing down (Jackson, 1962)

— Figure 2. —

Bremmstrahlung
Continuum radiation





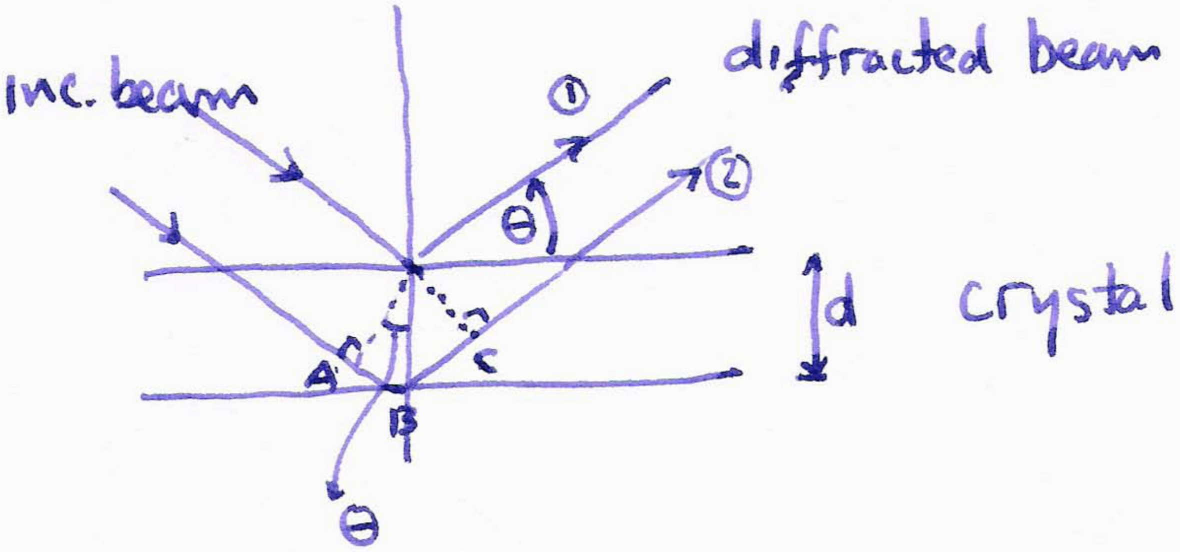
raw spectr
of biologica
section 10
thick take
with Si(Li
detector a
100 keV elec

— low energy
is due to d
transmissio
function

Bremstrahlung
spectrum aft
subtraction of
characteristic
and extraneo
background

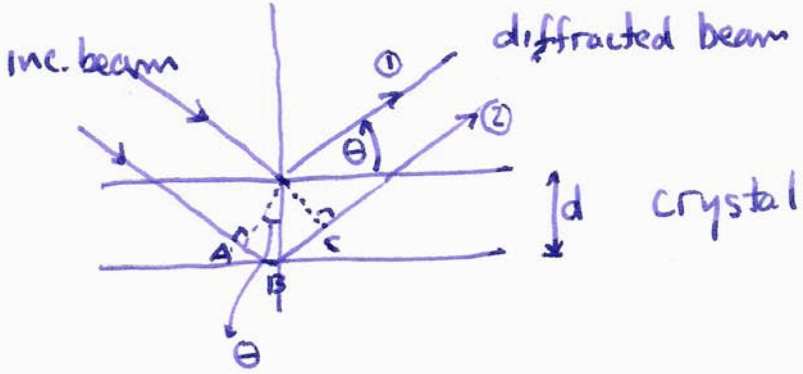
# How to Detect X Rays?

1. by wavelength (WDS)



# How to Detect X Rays?

1. by wavelength (WDS)



path difference between ① and ② is:

$$\overline{AB}+\overline{BC}=2d\sin\theta$$

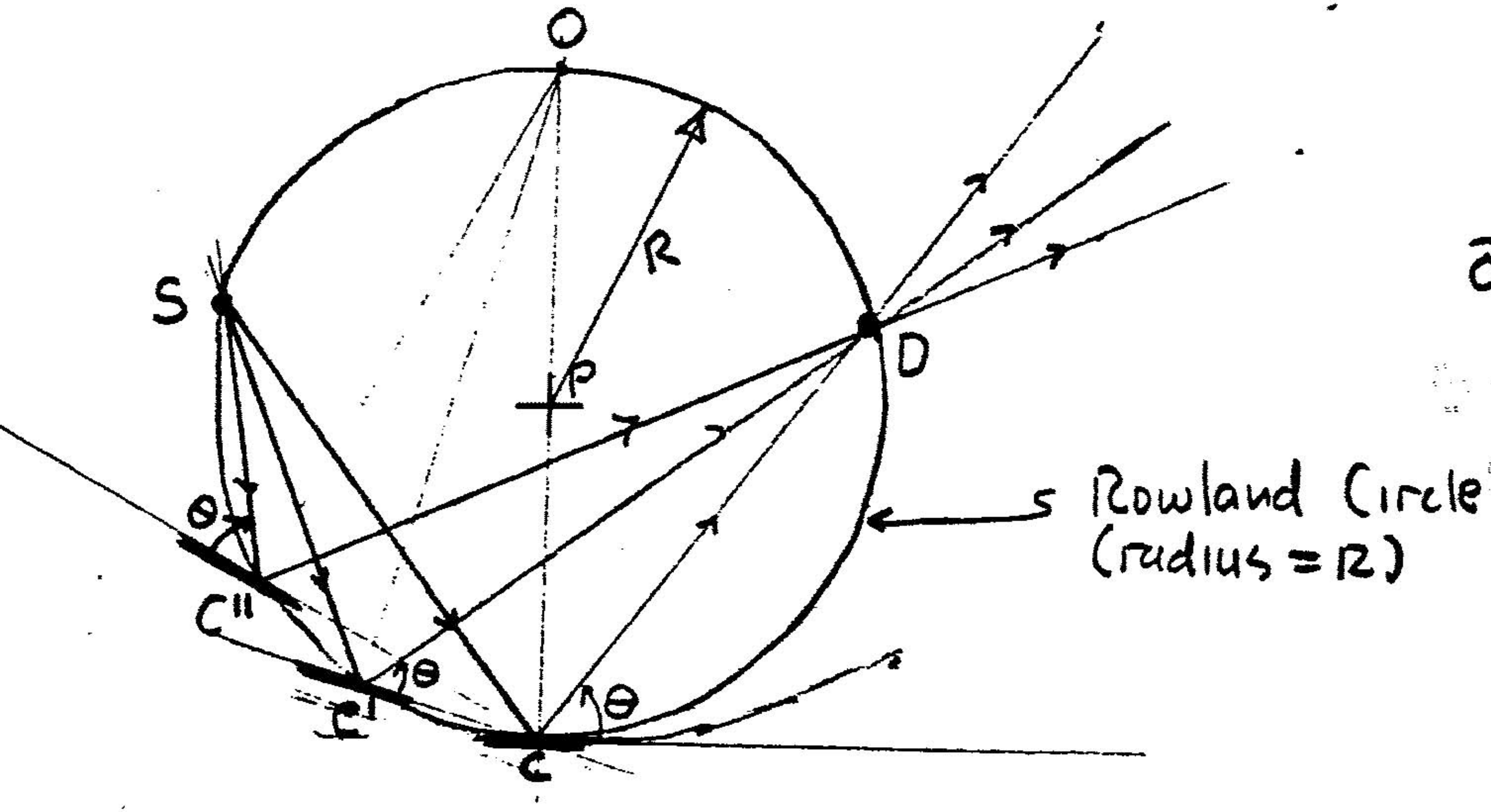
constructive interference between the two beams diffracted (reflected) off the 2 layers is:

path diff = $n\lambda$ , $n=0,1,2.$

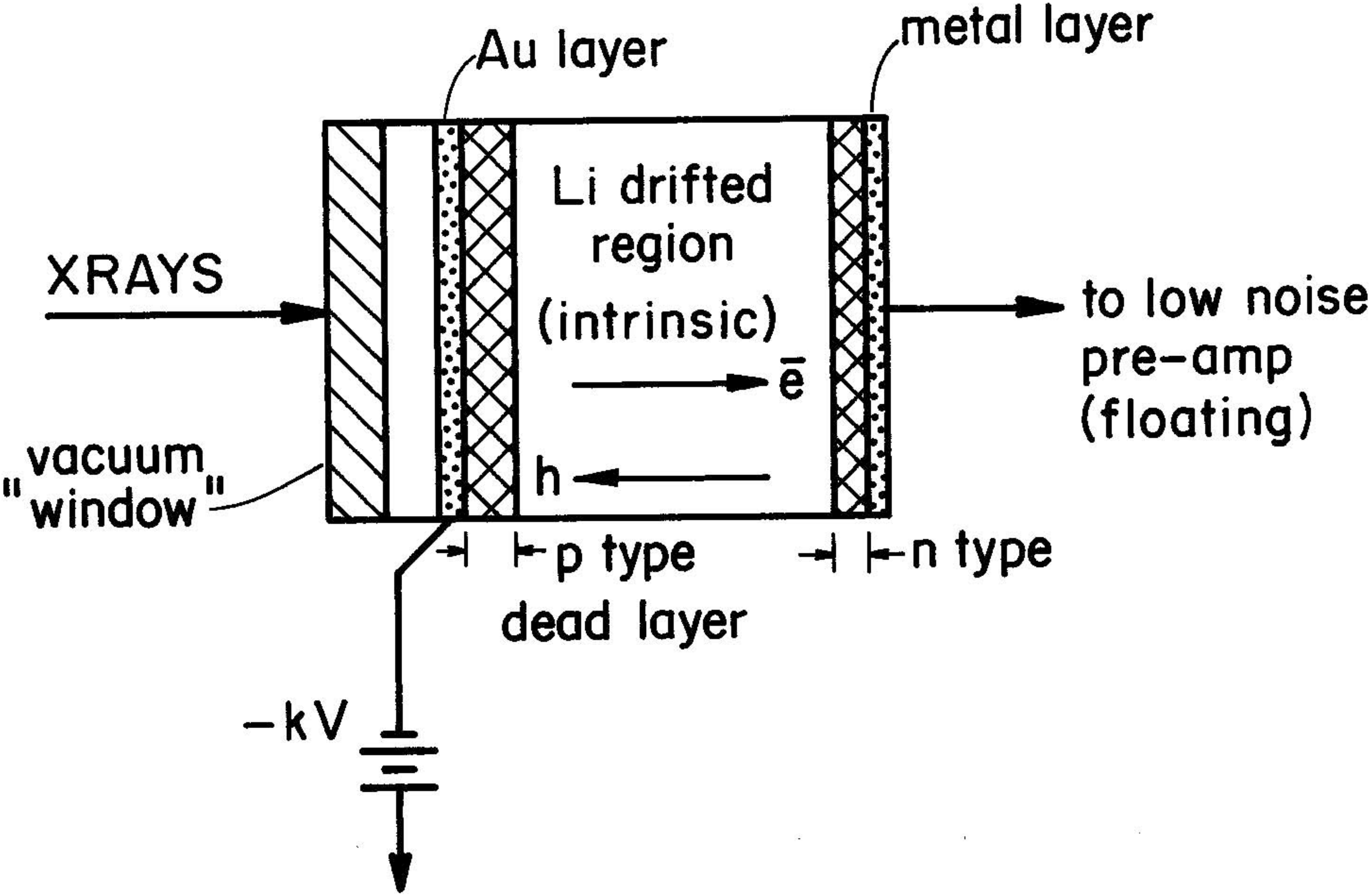
$\lambda$= XRay wavelength

$\therefore \boxed{n\lambda=2d\sin\theta}$ Bragg's law → $E=h\nu=\frac{hc}{\lambda}$

# crystal spectrometer geometry

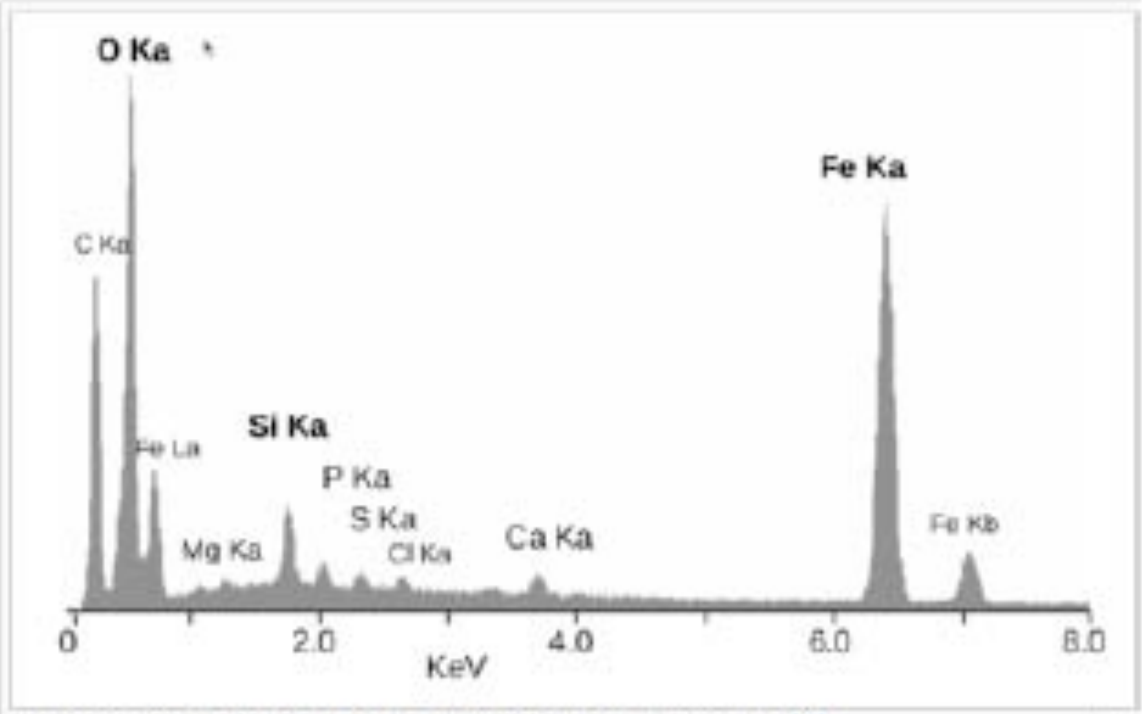


$\overline{OP} = R = \overline{PC}$


metal layer
Au layer
Li drifted region (intrinsic)
XRAYS
to low noise pre-amp (floating)
$\bar{e}$
h
vacuum "window"
p type dead layer
n type
−kV

# Energy Dispersive XRay Analysis
# (real detector/sample)

From: L.Corbari, et.al. Biogeosciences.5.(2008).1295-1310.



EDS spectrum of the mineral crust of *Rimicaris exoculata*[1]

Comparison between EDS and WDS
X Ray Spectra from the same material
(from NBS.)

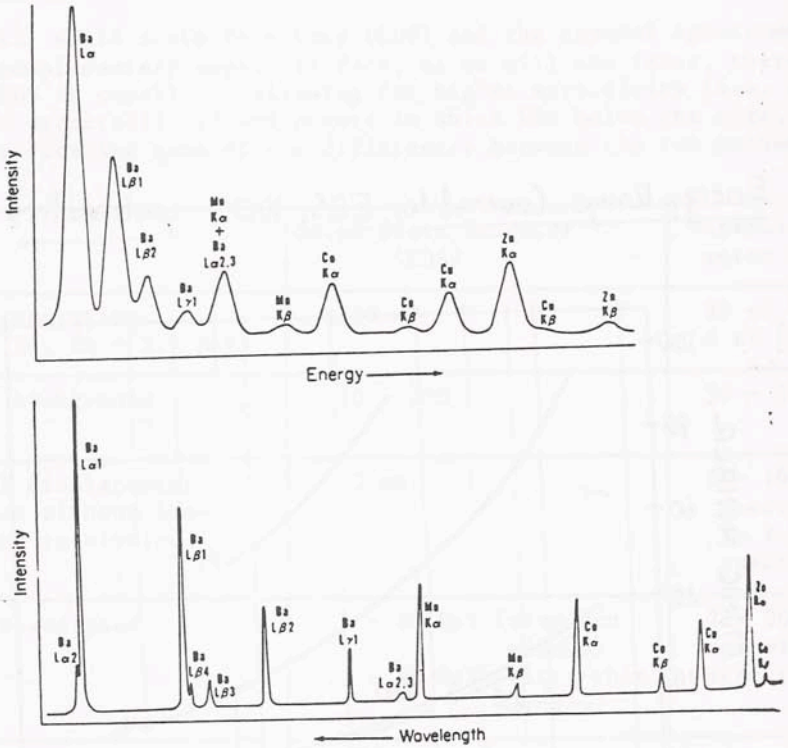
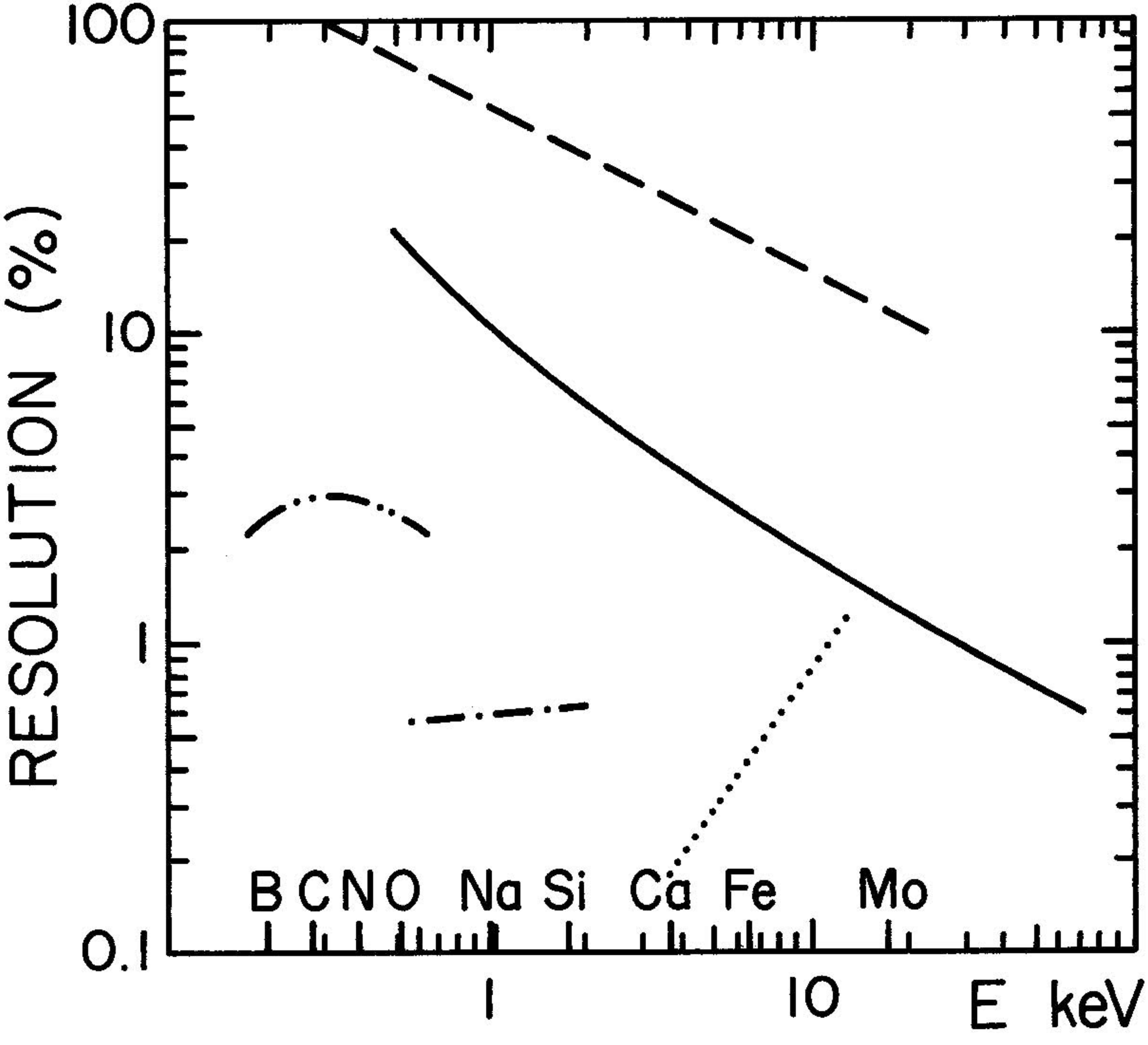
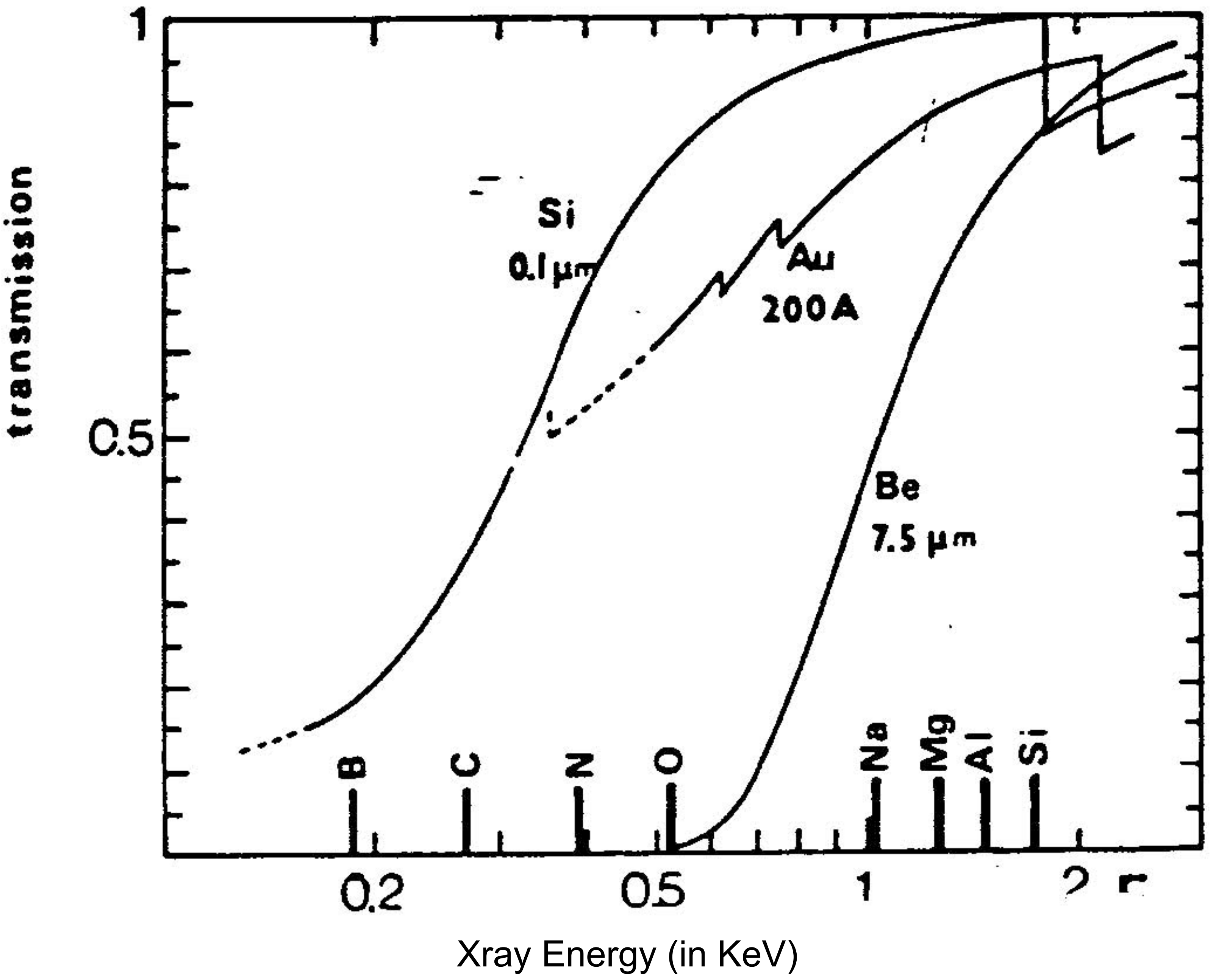


Fig. 5.24. Comparison of spectra of a glass (NBS K252) with a Si(Li) detector (above) and with a 10-cm radius LiF curved-crystal spectrometer (below). Both performed at 20-kV excitation potential. The composition of the glass is as follows: $SiO_2$: 0.40, BaO: 0.35, MnO: 0.10, $MnO_2$: 0.05, CuO: 0.05, CoO: 0.05, all mass fractions.

NOTE energy resolution, range and peak to background differences



RESOLUTION (%)
100
10
1
0.1
B C N O Na Si Ca Fe Mo
1
10
E keV


transmission
1
0.5
Si
0.1 μm
Au
200 A
Be
7.5 μm
B
C
N
O
Na
Mg
Al
Si
0.2
0.5
1
2
Xray Energy (in KeV)

X Ray analysis (cont)

2. Li Drifted Si, energy dispersive

Li acts as donor to compensate for impurity acceptor levels (B)

- results in intrinsic region in which $e^- - h^+$ can only be created by external ionizing radiation.
- bias the detector to drag $e^-$ to 1 side.

xray absorbed in intrinsic layer, $E_x$

$$\#e^- - h^+ \text{ pairs produced} = \frac{E_x}{\epsilon} \text{ — } 3.7\text{eV in Si}$$

$$\therefore \text{charge collected } Q = \left(\frac{E_x}{\epsilon}\right) \cdot e$$

detector has capacitance, so we actually get a voltage pulse $V = \frac{Q}{C} = \frac{e}{C}\left(\frac{E_x}{\epsilon}\right)$

pulse ht ∝ X ray energy → energy dispersive

we count pulses (one at a time)

assuming Poisson statistics for Q then standard dev. of voltage pulse is $\underline{\underline{\Delta V = \frac{e}{C}\sqrt{\frac{E_x}{\epsilon}}}}$ ← $\sqrt{N}$

$$\therefore \frac{\Delta V}{V} = \frac{\frac{e}{C}\sqrt{\frac{E_x}{\epsilon}}}{\frac{e}{C}\left(\frac{E_x}{\epsilon}\right)} = \sqrt{\frac{\epsilon}{E_x}} \qquad \left(\frac{\sqrt{N}}{N}\right)$$

not Poisson exactly $\Delta N < \sqrt{N}$

$$\therefore \frac{\Delta E_x}{E_x} = \sqrt{\frac{\epsilon}{E_x}} \Longrightarrow \boxed{\Delta E_x = \sqrt{\epsilon E_x}}$$

energy res.

for a Gaussian distrib: FWHM = 2.36 $\Delta E_x$ //

# X Ray Anal (cont)

call $\frac{\text{St. dev}}{N} = F$ ⇒ for Poisson, $F = 1$

$F$ = Fano factor — for Si(Li) = .12 ←

$$\therefore \boxed{\Delta E_x^{Si(Li)} = 1.6\sqrt{E_x} \text{ in eV}} \leftarrow$$

intrinsic energy resolution

but pulses are small, need amplification

∴ extra elec. noise

$$\Delta E_{FWHM} = \sqrt{(1.6\sqrt{E_x})^2 + E^2_{elec\,noise}}$$

($E_{elec} \sim 50-100$ eV //

|| need to cool to liq N2 to reduce thermal noise

efficiency of detector:

1) solid ∡
2) window, metalization layer, dead layer etc..
   all of these depend upon the x ray energy

2 effects:

1) x ray absorption in these layers resulting in fewer $e^- - h^+$ produced
2) x rays of high enough energy don't get absorbed in the intrinsic layer

this is similar for all EDX type detectors —

X Ray Analysis (cont)

consider the efficiencies resulting from these various layers

$$f_{\substack{DET\\ABS,trans}} = \Big[\prod_i \underbrace{e^{-(\mu/\rho)_i(\rho t)_i}}_{\text{product of absorption in each layer}}\Big]\times\Big[1-e^{-(\mu/\rho)_A(\rho t)_A}\Big]$$

probability that A xray won't be absorbed in intrinsic region

NOTE: if we calc for a layer of given thickness t, we can do any thickness t' as

$$t' = Nt \implies e^{-Nt} = (e^{-t})^N \quad ///$$

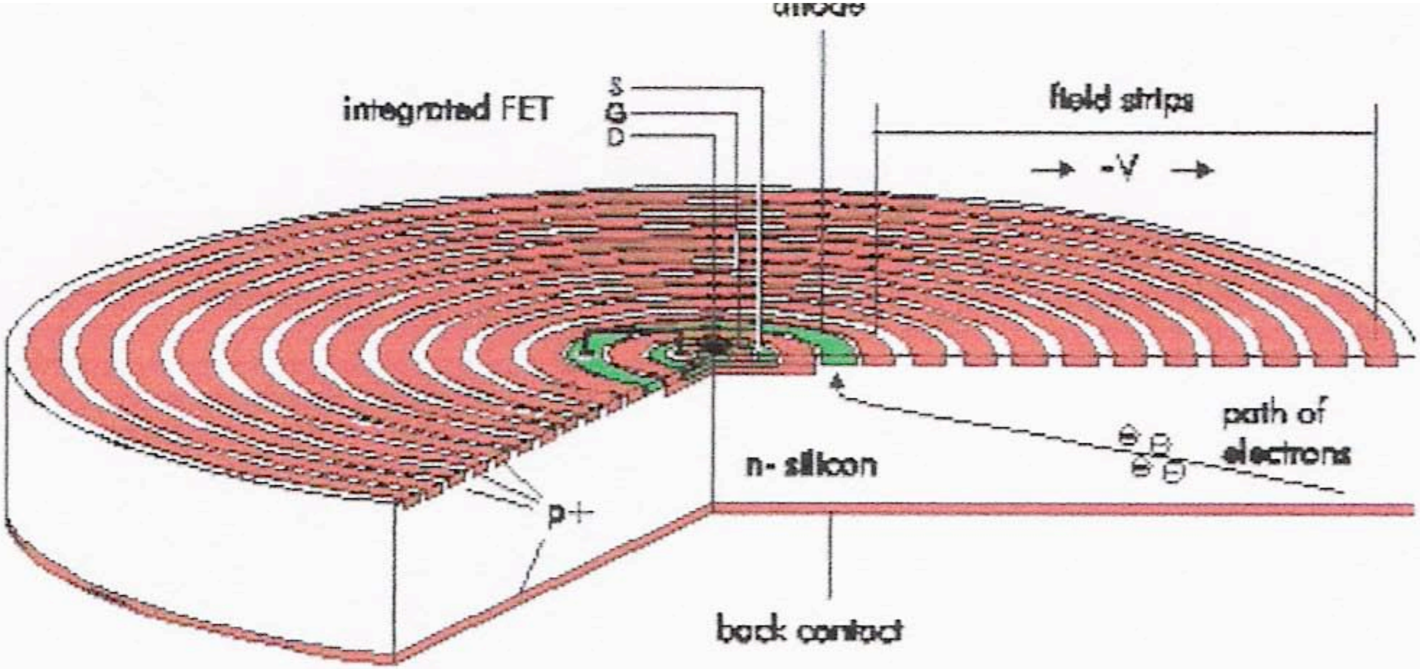
it is clear that to minimize absorption in front layers, want to make them as thin as possible.

best is "windowless" detector — no vacuum window barrier || probs.

doc cam

trans. char of diff. det. materials

pt. to note / — light element detection is poor since xray energies are so low
— any kind of physical window cuts down on transmission.



Silicon Drift Detector for X-ray spectroscopy.

[up]

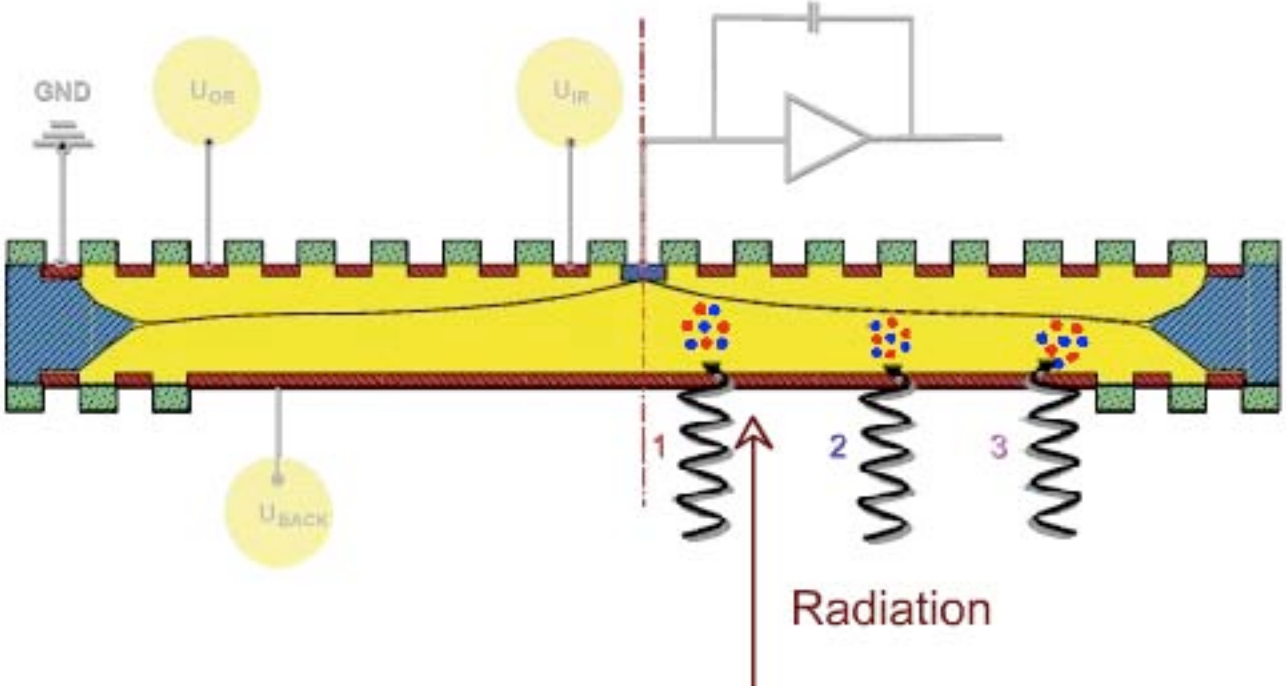
3. silicon drift detector (SDD).

same Si PN junction concept. (1983)

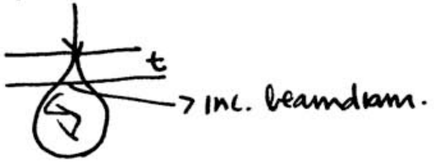
- recently used in EM's for xray detection (last few yrs)
  differences with Si(Li).
  - elec field parallel to the surface (rather than ⊥)
  - drives electrons towards small central anode
    (thus, lower capac.)
    ↳ + capac. indep. of active area
    whereas in Si(Li) depends on active area.

  small capac. means shorter shaping time → <u>fast cts</u> rate
- geometry minimizes pickup (elec or mech)
- virtually no det dead time
- leakage current extremely low so no liq $N_2$ cooling
  - uses Peltier cooling (−20°C vs −170°C Si(Li))
- resolution comparable to Si(Li)

# Function of Silicon Drift Detector



spatial resolution of Xray signal for thin films : beam spreading



rough calculation:

consider Ruth. Scatt

x section $>\theta$ : $\sigma_R(>\theta) = \int_\theta^\pi \frac{d\sigma_R}{d\Omega} d\Omega$

$$= \int_\theta^\pi 2\pi \sin\theta \, d\theta \left[\frac{Z^2}{64\pi^4 a_0^2}\right]\left[\frac{\lambda}{\sin\frac{\theta}{2}}\right]^4$$

$$\text{Å} \leftarrow \lambda = \sqrt{150/E_0^*} \, , \quad E_0^* = E_0\left[1 + \frac{1}{2}\frac{E_0}{mc^2}\right] \quad \text{eV}$$

rel. corr. wavelength

$$\therefore \sigma_R(>\theta) \cong \frac{162}{E_0^{*2}} Z^2 \operatorname{ctn}^2(\theta/2) \quad \text{in Å}^2$$

of $E_0^*$ in eV

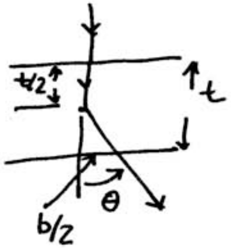
assuming single scatt, then prob. of scatt thru $>\theta$ in dist. t:

$$\underline{P(>\theta) = n\sigma_R(>\theta)t .}$$

$$\therefore \boxed{P(>\theta) \propto \frac{ntZ^2}{E_0^{*2}} \operatorname{ctn}^2\left(\frac{\theta}{2}\right)}$$

XRay anal (cont)

what is scatt ∡
whereby 90% events inside?



ie only 10% scattered outside.
call this $P(>\theta_{.1}) = 10^{-1}$ //

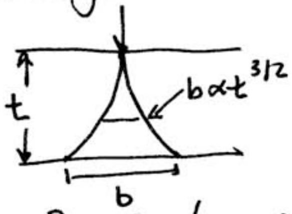
assume (in avg) scatt occurs ~~for mi~~ from middle of film, at t/2

then $\frac{b/2}{t/2} = \tan(\theta_{.1})$ which for small θ (<20°) gives:

$$\boxed{b \propto \frac{1}{E_0^*} \frac{Z}{\sqrt{A}} t^{3/2} \sqrt{\rho}}$$

ρ in gm/cm³
t in cm
A in gm/mole
$E_0^*$ in eV

that is
beam spreading $\propto t^{3/2}$



valid in limit $\tan\frac{\theta}{2} \cong \frac{\theta}{2}$ / can find out what that is.

$$t \lesssim .312 \times \frac{A}{Z^2 \rho} E_0^{*2}$$

in cm, of $E_0^*$ eV, ρ in gm/cm³

verified by MC calcs

do cam/
show
exp meas/
plots

important
for characterizatn
across interfaces

NOTE/ for high Z – long tails outside 10% can be significant

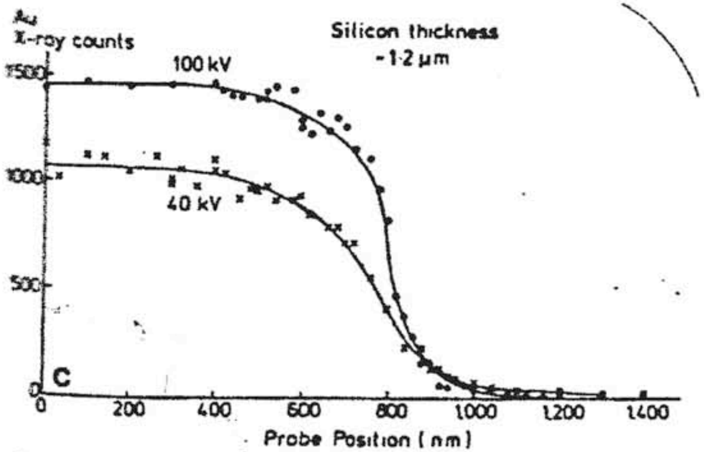
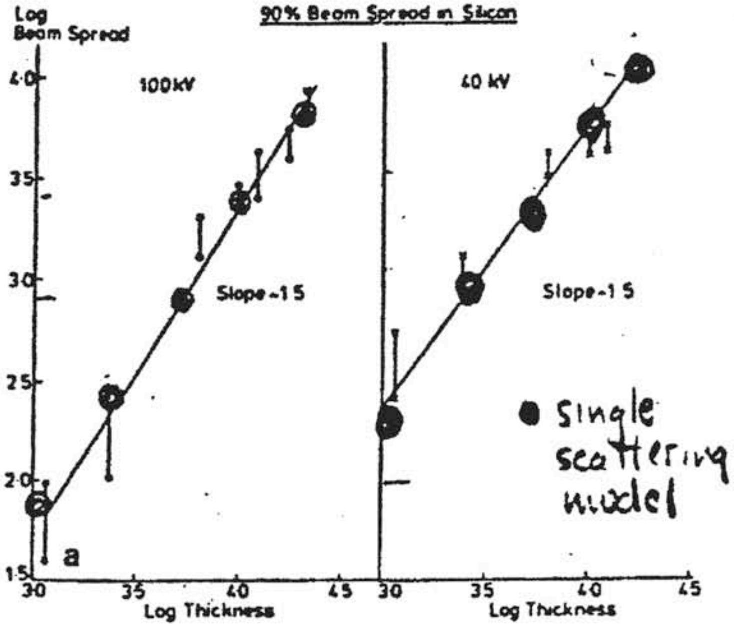
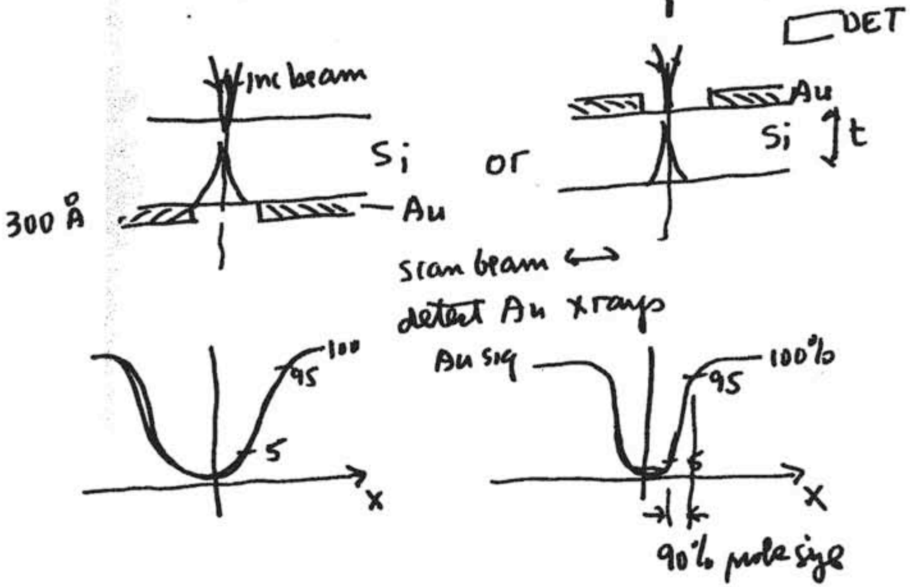


Fig. 3. Au X-ray counts at accelerating voltages of 40 and 100 kV as a function of probe position (a) and (b) from an area approximately 2400 A thick and (c) from an area ~1.2 µm thick.

from Hutchings et.al.





#26

SI

Hutchings, et.al (1978)
Ultramicroscopy.3. 401.