Energy Dispersion Spectrometry (EDS)

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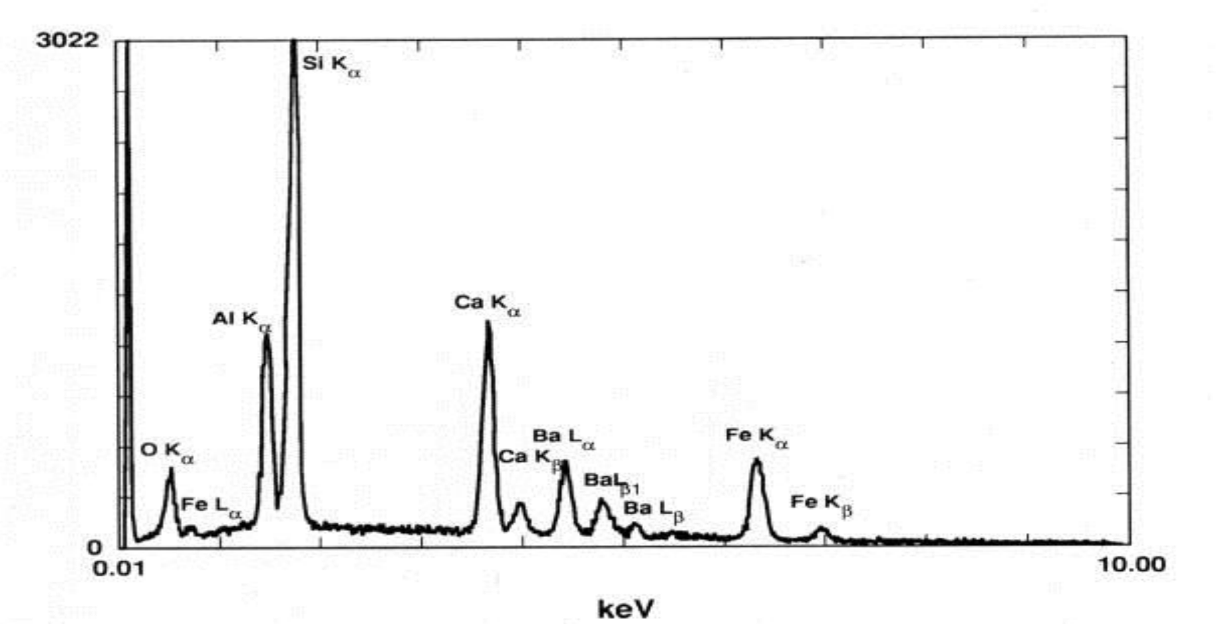
What is EDS?

Using X-rays to produce electron-hole pairs (total summed charge which is proportional to incident x-ray energy), which are amplified and then "digitized" by voltage, displayed as a histogram of the number of x-rays pulses (y axis) versus x-ray energy (x axis).

EDS spectrum for NIST glass K309



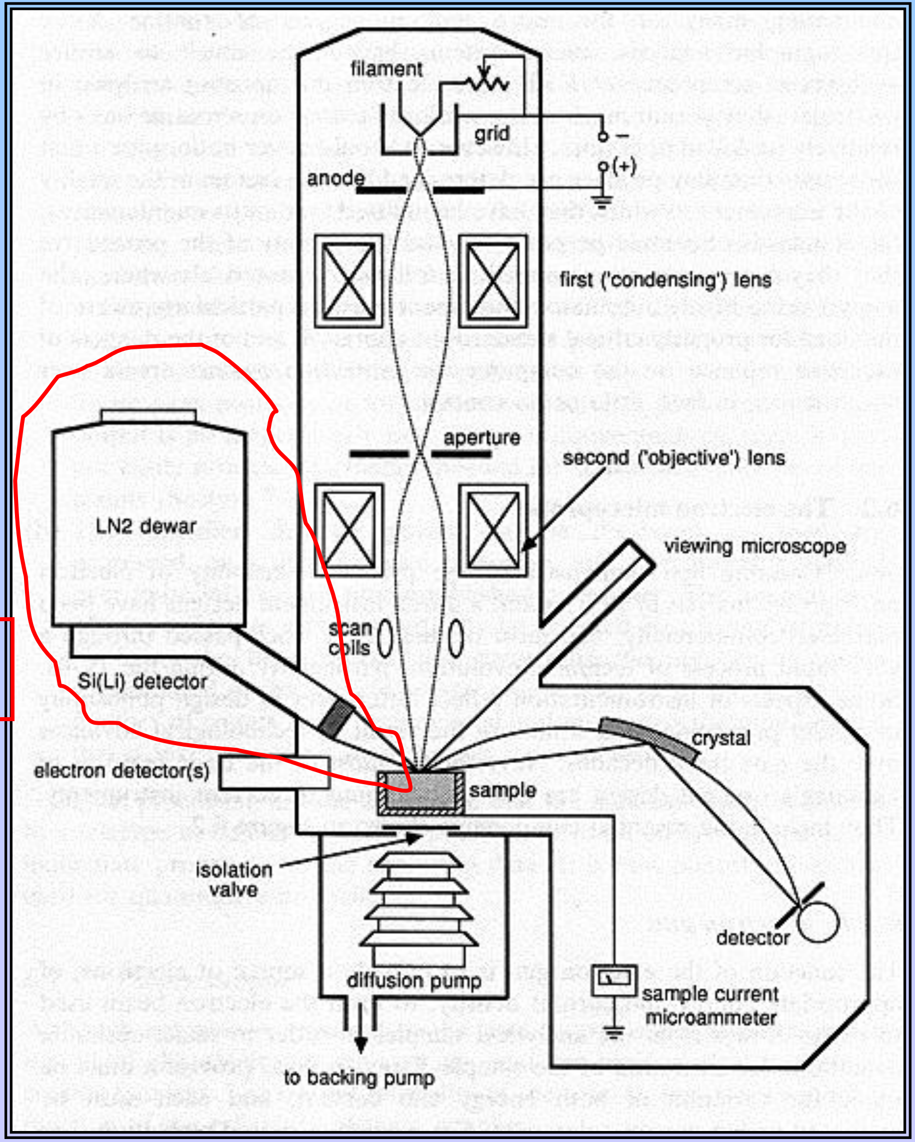
Summary

- X-rays cause small electric pulses in a solid state detector. Associated electronics produce ‘suddenly’ a spectrum, i.e. a histogram of count (number, intensity) v.s the energy of the X-ray

- there are probably 50-100 EDS detectors in the world for every 1 WDS (electron microprobe)

- Operator should be aware of the limitations of EDS, mainly the specific spectral artifacts, and the poor spectral resolution for some pairs of elements

Generic EMP/SEM



EDS detector

SE,BSE detectors

Vacuum
pumps

Electron gun

Column/ Electron
optics

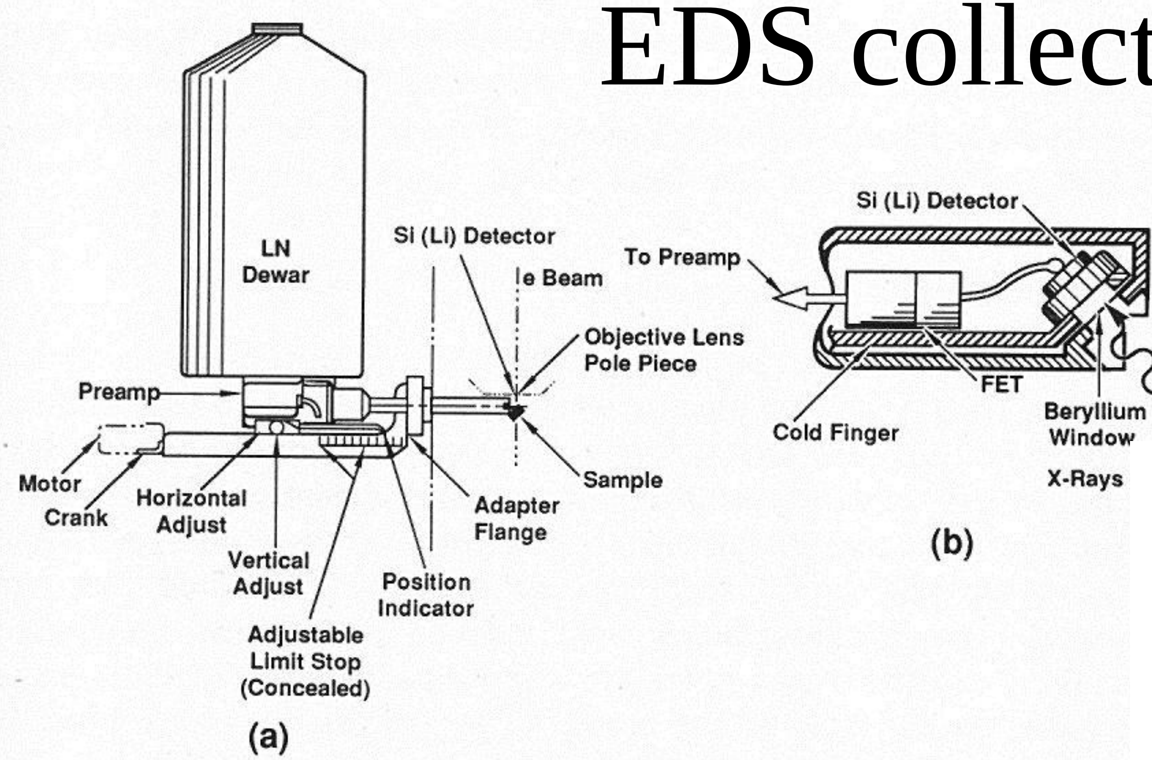
Optical microscope

Scanning coils

WDS
spectrometers

Faraday current
measurement

EDS collection



There are several types of solid state EDS detectors, the most common (cheapest) being the Si-Li detector.

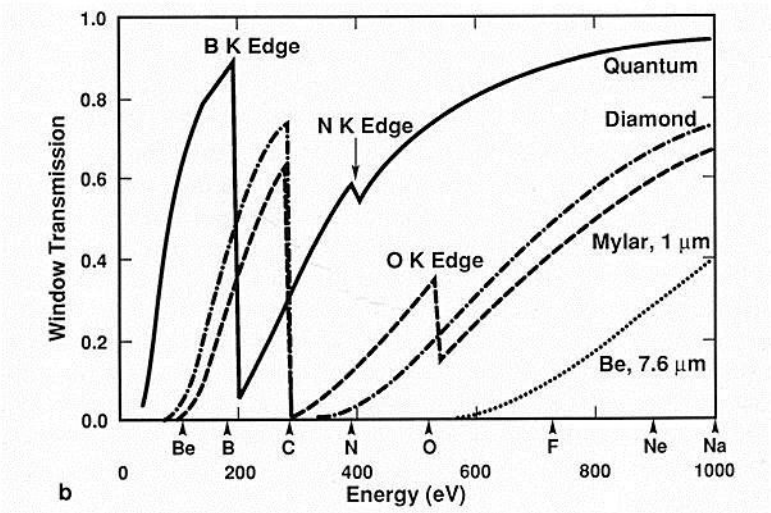
Components: thin window (Be, C, B); SiLi crystal, FET (field effect transistor: initial amp), vacuum, cold finger, preamp, amp and MDA electronics ("multi channel analyzer").

EDS Windows

Windows allow X-rays to pass and protect detector from light and gases.

Be: The most common EDS detector window has been made of Be foil ~7.6 µm (0.3 mil) thick. It allows good transmission of X-rays above ~ 1 keV. It is strong enough to withstand venting to atmospheric pressure, and opaque to optical photons.

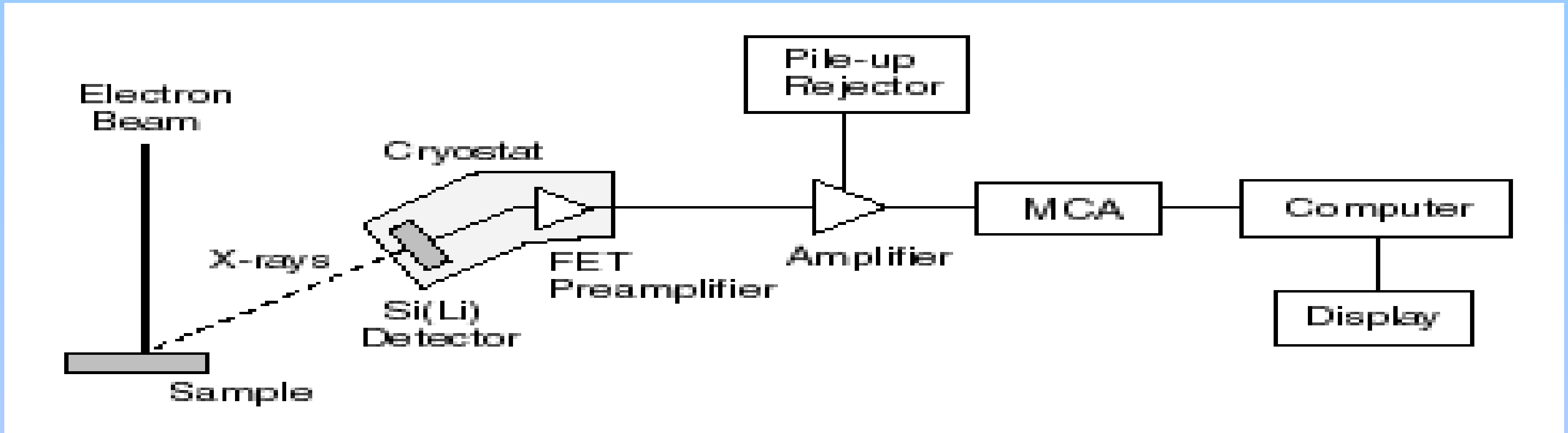
Thin - Ultrathin: For transmission of light element X-rays (<1 keV), windows ~0.25 µm thick of BN, SiN, diamond or polymer are used. They must use supporting grids to withstand pressure differentials; the grid (e.g., Si or Ni) takes up about 15% of the area, but thin enough that low energy X-rays pass through.

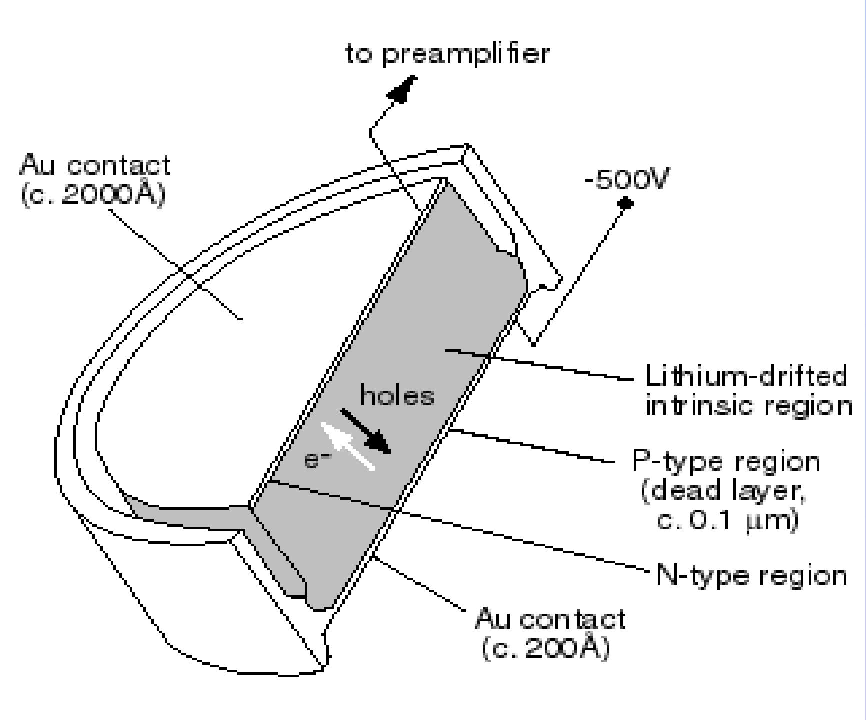


This plots shows the transmittance of X-rays thru difference types of window material. (Quantum [BN] 0.25 um, diamond 0.4 um). The higher the number, the better

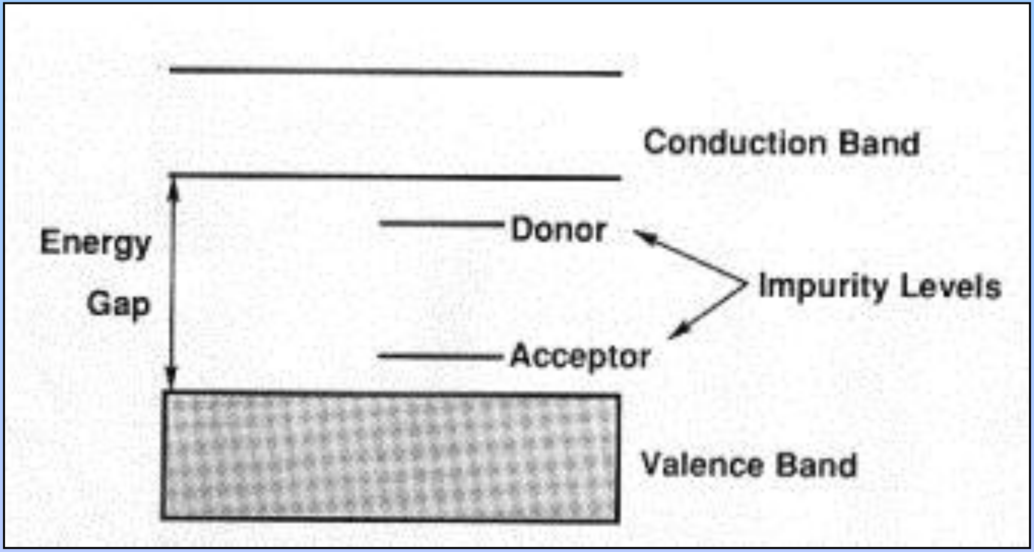
“Windowless”: Here there is no film, and there is a turret that allows swapping with a Be window. Difficult to use as oil or ice can coat the detector surface.

Energy Dispersive System Schematic





How it works: energy gap



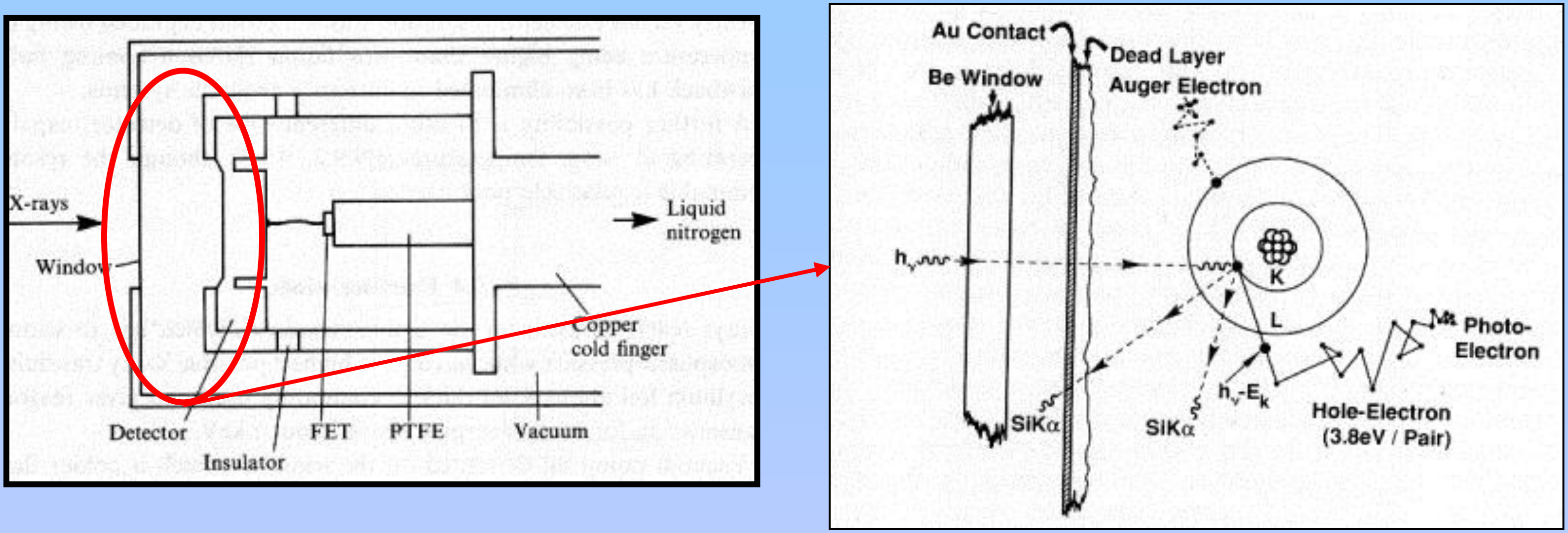
A semi-conductor like Si has a fully occupied valence band and largely unfilled conduction band, separated by an energy gap (1.1 eV). Incident energy can raise electrons from the valence to the conduction band.

X-ray hits the SiLi crystal, producing a specific number of electron-hole pairs proportional to X-ray energy; e.g. one pair for every 3.8* eV, so for incident Fe Ka, 6404 eV, 1685 e-hole pairs are produced. With a bias** applied across the crystal, the holes are swept to one side, the electrons to the other, producing a weak charge .Boron is important acceptor impurity in Si and degrades it (permits thermal excitation: bad); Li is drifted in (donor impurity) to counter its effects.

*** 1.1 eV + energy wasted in lattice vibrations, etc**

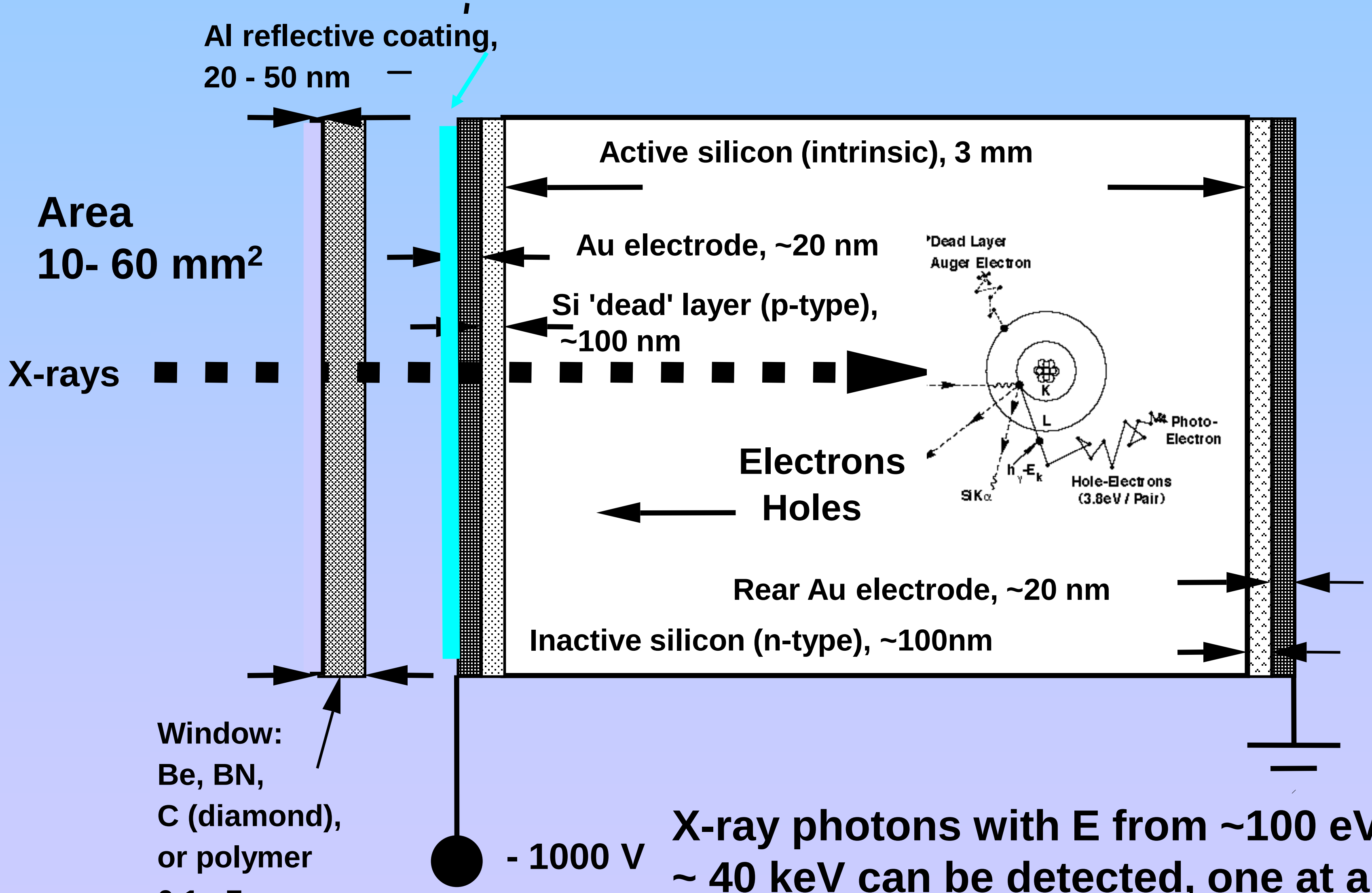
****bias: a voltage is applied between 2 points; e.g. one +1500 v, other -1500 v.**

How it works: inside the detector



X-rays are absorbed by Si, with K shell (photo) electron ejected with h_v - E_c (E_c=1.84 keV) energy. This photoelectron then creates electron-hole pairs as it scatters inelastically. The Si atom is unstable and will either emit a characteristic Auger electron or Si ka X-ray. If Auger, it scatters inelastically and produces electron-hole pairs. If Si Ka X-ray, it can be reabsorbed, in a similar process, or it can be scattered inelastically. In either case, the energy will end up as electron-hole pairs. **The result, in sum, is the conversion of all the X-ray's energy into electron-hole pairs** -- with 2 exceptions.

The Monolithic Semiconductor Energy Dispersive X-ray Spectrometer



Al reflective coating, 20 - 50 nm

Active silicon (intrinsic), 3 mm

Area
10- 60 mm^2

Au electrode, ~20 nm

Si 'dead' layer (p-type), ~100 nm

X-rays

Electrons

Holes

Rear Au electrode, ~20 nm

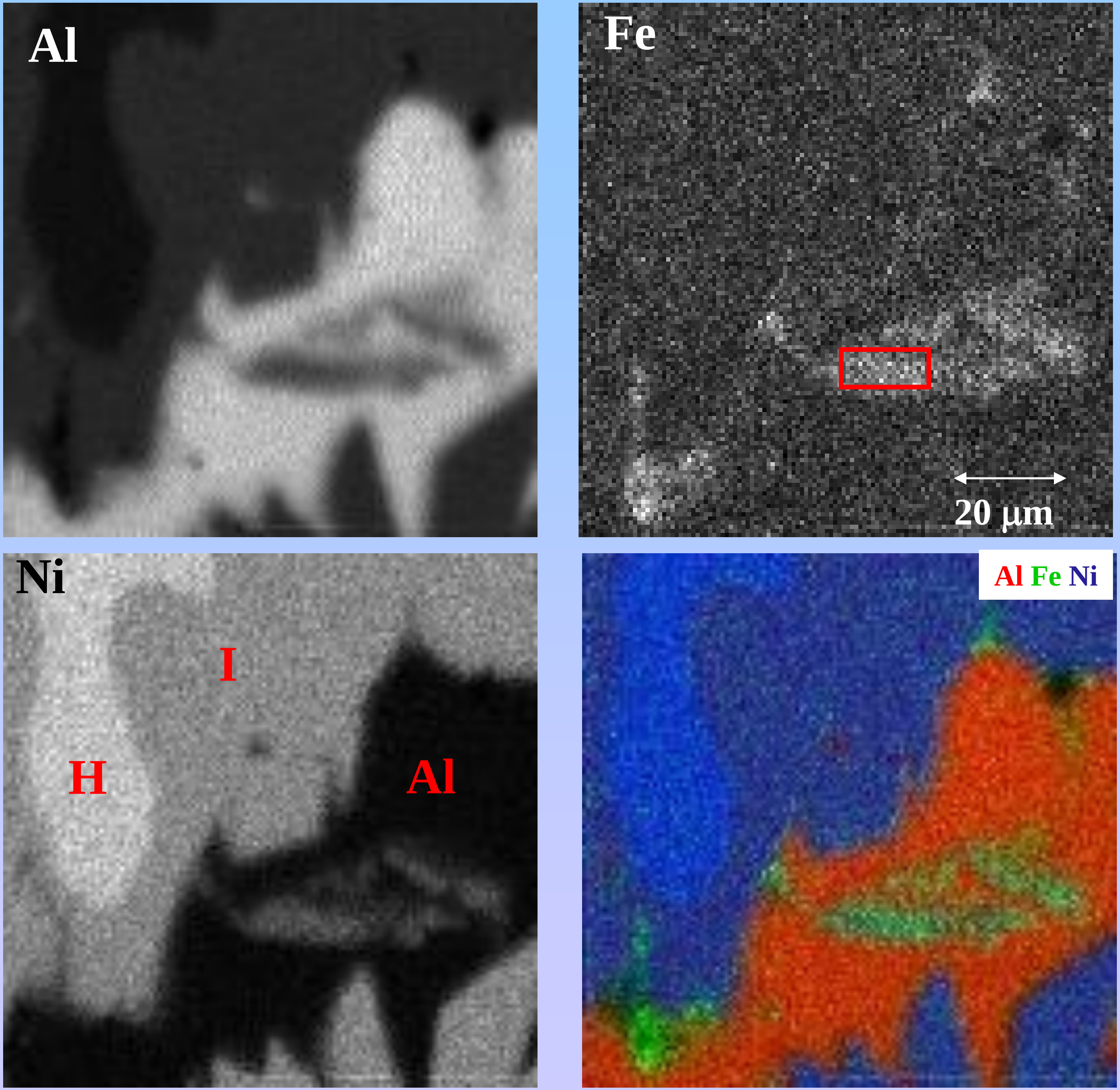
Inactive silicon (n-type), ~100nm

Window:
Be, BN,
C (diamond),
or polymer
0.1 - 7 µm

- 1000 V

X-ray photons with E from ~100 eV to ~ 40 keV can be detected, one at a time!

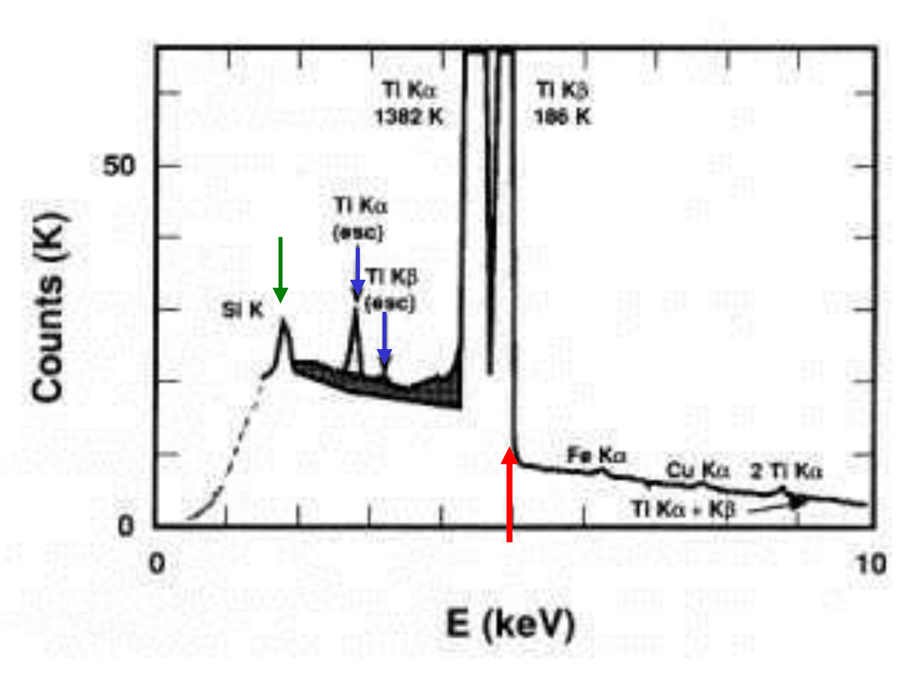
Raney Nickel Alloy
E_0 = 20 keV 10 nA
TC = 500 ns (188 eV at $MnK\alpha$)
128x128
10 ms per pixel
Mapping 185 sec



Phases
Al 99.5 Ni 0.5
Al 71.2 Ni 24.6 Fe 4.2
Al 60 Ni 40 “I”
Al 46.5 Ni 53.5 “H”

Artifacts: Si-escape peaks; Si internal fluorescence peak; extraneous peaks

The figure shows a real spectrum of a sample of pure Ti metal -- but there are 7 peaks besides the Ti Kα and Kβ. At 1.74 keV below each, are the respective escape peaks (blue arrows). Also present is a Si internal fluorescence peak (green arrow). The Fe and Cu peaks are from excitation of metal in chamber or sample holder by BSE or Ti X-rays. Note the sharp drop in the background intensity on the high side of the Ti Kβ peak (= Ti K absorption edge, red arrow). (2 Ti Kα and Ti Kα+Kβ explained shortly.)

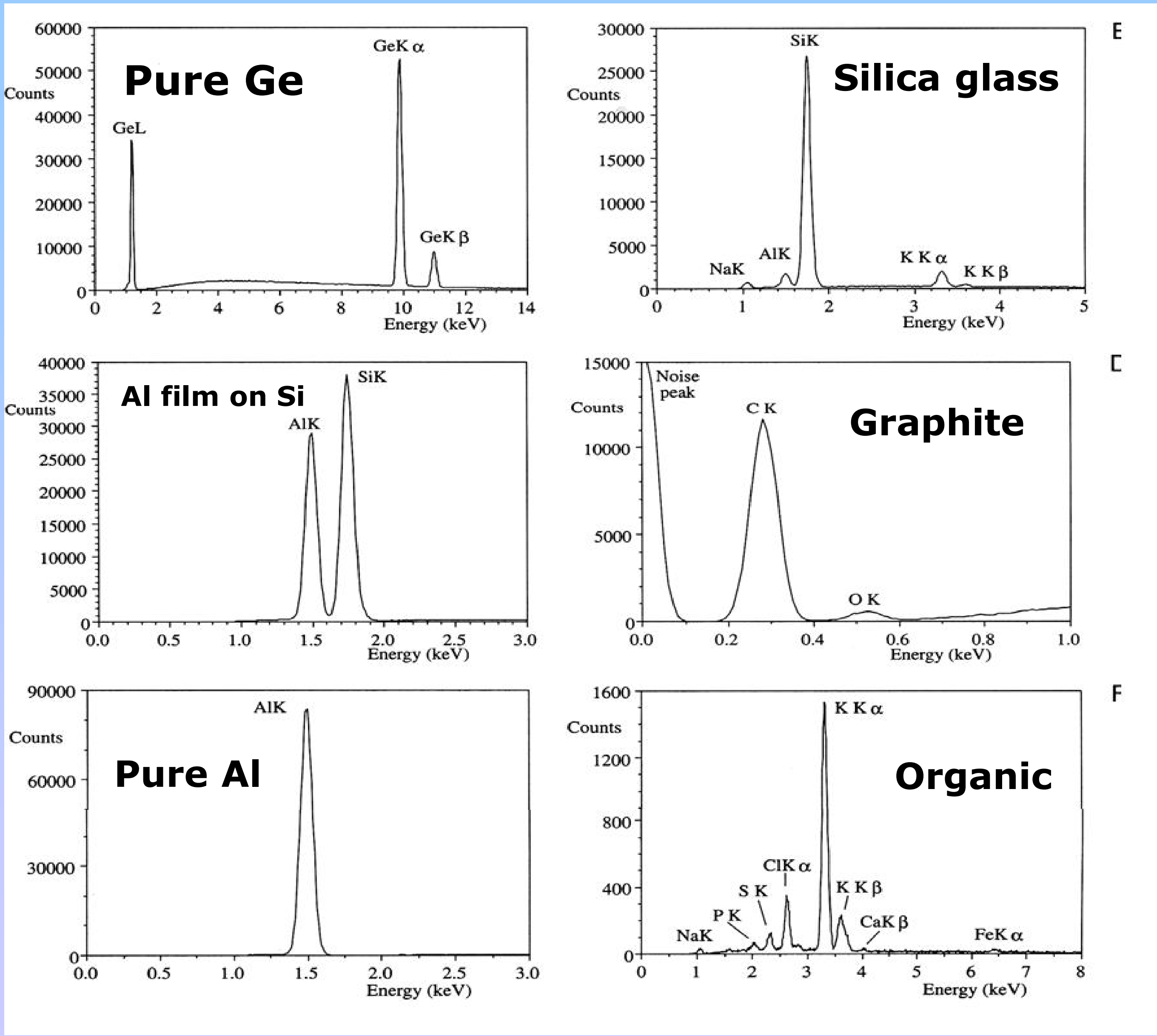


Note the scale of the spectrum: the Ti Kα max is 1.3 million counts. These effects are generally weak, but evident when you are looking for minor elements.

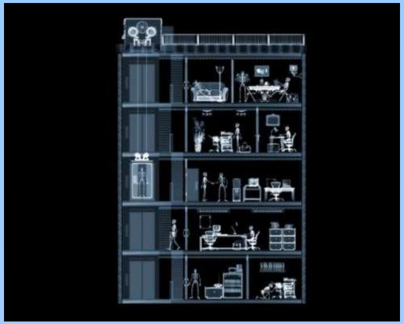
EDS-WDS comparison

Characteristic	EDS	WDS
Geometric collection efficiency (solid angle)	<2%	<0.2%
Spectral resolution (FWHM)	160 eV and less	2-10 eV
Instantaneous X-ray detection	~ 1 keV thru E0 (window dependant)	a few eV
Maximum count rate	10s of thousands cps over whole spectrum	tens of thousands cps (one wavelength)
Artifacts	sum peaks, Si escape peaks, Si fluor. peak	n>1 peaks, Ar escape peaks
Light elements?	With windowless or thin window detector	With synthetic diffractors ("crystals")
Detection Limits	~1000-5000 ppm, 0.1-.5 wt%	<100-500 ppm, <0.01-.05 wt%
Bottom Line	Cheaper, quicker but some elements are too close together to resolve (eg S Ka, Mo La, Pb Ma)	More expensive, but with much better spectral resolution and higher Pk/Bkg, giving lower detection limits.

Several examples of EDS spectra



X-Ray Detection



- Electron - hole pairs created.
- Each electron-hole pair requires a mean energy of 3.8 eV
- Bias voltage sweeps charge carriers to either side
- Charge proportional to Xray energy

- Note: Charge is small! Noise is a potential problem.
- Note: High energy X-rays may not be dissipated in the active region of the crystal! Incomplete charge collection.
- (EDX spectrometers work best in the region 1-20 Kev)

Q//The different between XRD& EDS???

- 1. EDS is used for *chemical identification of elements* and their concentration. XRD is used to identify *spatial arrangements of atoms* in crystalline phases.

  2. In EDS, electrons knock out electrons from atoms, producing X-rays of *characteristic wavelength*. These X-Rays are then detected to identify the element from which they were produced. In XRD, X-rays of *known wavelength* are used to probe the structure of the material, using the lattice as a diffraction grating.

  3. EDS you need an electron source. For XRD you need an X-ray source.

  4. If you are looking for chemical composition, concentration gradients, or solute segregation, use EDS.If you are looking for phase distribution, lattice strain fields or stored defect content, use XRD.


EVO 16


PGT